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**DISSERTATION**

**TECHNOLOGY OF PHYSICALLY MODIFICATION OF POTATO  
STARCH AND THEIR APPLICATIONS IN FOOD PRODUCTS**

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## АНОТАЦІЯ

Ден Чунлі. Технологія фізичної модифікації картопляного крохмалю та його використання у виробництві харчових продуктів. – Кваліфікаційна наукова робота на правах рукопису.

Дисертація на здобуття наукового ступеня доктора філософії за спеціальністю 181 – «Харчові технології» – Сумський національний аграрний університет, Суми, 2023.

Дисертаційну роботу присвячено розробці технології модифікації картопляного крохмалю за допомогою фізичних методів, таких як вологотермічна обробка НМТ в поєднанні з мікрохвильовою обробкою MW, та науковому обґрунтуванню застосування фізично-модифікованого крохмалю у технології виробів з пшеничного борошна.

У першому розділі дисертаційної роботи представлені результати аналітичного огляду літературних джерел, а саме встановлено взаємозв'язки між структурою та властивостями крохмалю, проведено аналіз способів оброблення нативного крохмалю за допомогою фізичних, хімічних та ферментативних методів модифікації, визначено перспективи застосування модифікованого крохмалю у виробництві продуктів харчування.

НМТ і MW є найпривабливішими методами фізичної модифікації крохмалю завдяки їхнім перевагам: відсутність побічних продуктів-реагентів, можливість отримати модифікований крохмаль із заданими властивостями при зміні параметрів оброблення, простий процесу контролю якості та безпеки в умовах промислового виробництва, екологічність процесу та відсутність негативного впливу на навколишнє середовище.

У другому розділі викладено наукову проблему дисертаційної роботи, обґрунтовано вибір об'єктів та напрямів дослідження, наведено методи фундаментальних та прикладних досліджень. Відповідно до завдань дослідження було складено робочу програму досліджень, яка передбачає дослідження модифікації технологічних процесів вологотермічної обробки

НМТ нативного картопляного крохмалю для визначення оптимальних параметрів оброблення та дослідження модифікації технологічних процесів вологотермічної обробки НМТ крохмалю в поєднанні з мікрохвильовою MW попередньою та післяобробкою. А також представлено методи визначення фізико-хімічних властивостей крохмального клейстеру, фізико-хімічних властивостей крохмальних гранул та їх багатомасштабної структури для встановлення можливості застосування модифікованого картопляного крохмалю у виробництві печива, свіжої локшина, хлібу на пару.

Досліджено вплив умов НМТ на морфологічні, фізико-хімічні властивості та властивості перетравлення модифікованого картопляного крохмалю *in vitro*, проведено оптимізацію процесу оброблення, що надає комплексне розуміння впливу умов НМТ на функціональні властивості крохмалю та механізм перетворень, який забезпечить корисну теоретичну основу для подальших досліджень щодо покращення застосування модифікації крохмалю методом вологотермічної обробки НМТ.

Прозорість і стійкість до ретроградації крохмального клейстеру після НМТ були знижені, розчинність і здатність до набухання крохмальних зерен змінювалися в залежності від температури оброблення. НМТ може суттєво впливати на текстурні властивості картопляного крохмалю, а твердість, клейкість, жувальні властивості та пружність крохмальних гелів після НМТ спочатку значно збільшуються, а потім зменшуються із подовженням часу обробки. Короткий час нагрівання (<1,5 години), відносно низька температура нагрівання (<100°C) і низький вміст води у сировині (<25%) у процесі НМТ можуть значно покращити текстурні властивості крохмальних гелів. НМТ обробка картопляного крохмалю впливає на його функціональні властивості, тому для використання його у складі різних харчових продуктів (хліба на пару, локшини, печива) необхідно підбирати відповідні умови вологотермічної обробки.

НМТ обробка здійснювала значний вплив на розмір частинок, морфологічні, структурні та перетравлювальні властивості картопляного крохмалю. НМТ обробка сприяла утворенню частинок великого розміру, гранули крохмалю мали шорстку поверхню, що призвело до утворення порожнистої структури, розташованої на гілумі гранул картопляного крохмалю. Результати рентгеноструктурного аналізу XRD показали підвищену відносну кристалічність і трансформовану кристалічну структуру з В-типу на С-тип після тривалої обробки теплом та вологою. В результаті проведення інфрачервоної спектроскопії з перетворенням Фур'є FTIR встановлено, що обробка теплом і вологою може призвести до розриву молекулярного ланцюга крохмальної молекули або розриву асоціативного водневого зв'язку молекул крохмалю. Результати визначення перетравлюваності крохмалю *in vitro* показали, що процес НМТ може значно зменшити кількість швидко засвоюваного крохмалю (RDS), але збільшити кількість резистентного крохмалю (RS), це вказує на те, що крохмалі НМТ потенційно можуть стати сировиною для промислового виробництва резистентного крохмалю та можуть використовуватися в якості низькокалорійних харчових інгредієнтів.

Оптимізовані параметри процесу зниження в'язкості крохмальних гелів модифікованого крохмілю шляхом НМТ були наступними: температура становила 90°C, час обробки складав 1,5 години та вміст води у сировині відповідно був 23,56%. За таких умов максимальне значення теоретичної зниженої в'язкості становило 3871 с·Па. Перевірочний експеримент показав, що фактична середня знижена в'язкість становила 3677 с·Па, тобто похибка між фактичною зниженою в'язкістю та теоретичною зниженою в'язкістю була незначна. Порівняно з нативним картопляним крохмалем (NS), крохмаль, який пройшов вологотермічне оброблення (НМТS), мав нижчу пікову в'язкість (2966 с·Па), нижчу стійку в'язкість (2882 с·Па) і нижчу в'язкість руйнування (84,5 с·Па), але вищу



температуру клейстеру (71,1°C), вищу кінцеву в'язкість (6559 с·Па). Твердість, когезія, клейкість, жувальна здатність гелю NS становили 2706 г; 0,63; 1700 г; 1404 г·мм відповідно, тоді як твердість, когезія, клейкість, жувальна здатність гелю HMTS становили 6082 г; 0,73; 4920 г; 3570 г·мм відповідно. Порівняно з гелем нативного крохмалю NS, твердість, когезія, клейкість і розжовування гелю модифікованого крохмалю HMTS були значно вищими, однак не було істотної різниці в пружності гелю. Ретроградація гелю NS і HMTS зростала з подовженням часу зберігання, і ретроградація гелю HMTS була вищою, ніж NS, що свідчить про більшу схильність модифікованого крохмалю до ретроградації. Результати дослідження процесу перетравлення крохмалю *in vitro* показали, що HMTS мав більший вміст повільно розщепленого крохмалю (SDS) та вміст резистентного крохмалю RS, ніж нативний картопляний крохмаль NS, але нижчий вміст швидко-засвоюваного крохмалю RDS. Порівняно з нативним картопляним крохмалем вміст RDS зменшився на 10,05%, повільно-засвоюваного крохмалю SDS збільшився на 5,06%, а резистентного крохмалю RS збільшився на 2,48% відповідно в оптимізованому зразку модифікованого крохмалю HMTS.

Вплив вологотермічної обробки крохмалю НМТ у поєднанні з мікрохвильовим обробленням MW до і після вологотермічної обробки на морфологічні, фізико-хімічні властивості та властивості перетравлення *in vitro* картопляного крохмалю були оцінені в четвертому розділі роботи. Це дослідження представило всебічне розуміння ефектів двонаправлених модифікацій НМТ і MW на функціональні властивості та властивості засвоюваності крохмалю, а також дозволило визначити механізм, який забезпечить корисну теоретичну основу для подальших досліджень щодо покращення застосування технології мікрохвильового оброблення сировини для модифікації її властивостей.

Усі зразки крохмалю, модифіковані за допомогою однієї НМТ, однієї

MW та подвійної модифікації НМТ у поєднанні з MW, показали нижчу здатність крохмалю до набухання, ніж нативного крохмалю NS, при температурі випробування 65–85°C, тоді як протилежні результати були отримані при 95 °C. Всі зразки крохмалю мали вищу розчинність, ніж у нативного крохмалю, при температурі 75–95°C. Оброблення MW, оброблення НМТ і короткочасне оброблення НМТ у поєднанні з попередньою обробкою MW можуть підвищити стабільність багаторазового заморожування-розморожування гелю модифікованого крохмалю, тоді як тривале оброблення НМТ ( $\geq 4$ h) може послабити стабільність гелів картопляного крохмалю при заморожуванні та розморожуванні. Подвійна модифікація НМТ і MW здійснювала більший вплив на процес ретроградації крохмалю, ніж використання НМТ та MW окремо, крім того, НМТ у поєднанні з попередньою обробкою MW також мала більший вплив на ретроградацію крохмалю, ніж НМТ у поєднанні з постобробкою MW. Час вологотермічного оброблення НМТ мав значний вплив на прозорість крохмального клейстеру, подвійна модифікація НМТ у поєднанні з MW мала більший вплив на прозорість крохмального клейстеру, ніж обробка крохмалю НМТ та MW окремо. Твердість, когезія, клейкість і жувальна здатність усіх гелів картопляного крохмалю, модифікованого НМТ (включаючи оброблення НМТ, оброблення НМТ у поєднанні з MW), зменшилися зі збільшенням часу нагрівання. Картопляний крохмаль НМТ, попередньо оброблений MW, мав вище значення твердості, ніж картопляний крохмаль НМТ, з MW післяобробкою.

Обробка НМТ призвела до незначного збільшення білизни (значення  $L^*$ ), тоді як одноразова обробка MW спричинила незначне зменшення білизни, що вказує на те, що колір усіх оброблених НМТ зразків (НМТ, MW-НМТ, НМТ-MW) став яскравішим, а колір одного зразка, обробленого MW (MWS), став темнішим. Експерименти з розподілом частинок крохмалю за розміром показали, що D50, D (4,3) і D (3,2) всього обробленого крохмалю

були вищими, ніж NS, тоді як значення S.S.A було значно знижено за MW і HMT, тобто MW і HMT обробки можуть спричинити розширення, часткову клейстеризацію та агломерацію крохмальних гранул, що призведе до великого розміру частинок, що узгоджується з результатами скануючої електронної мікроскопії (SEM). Результати розподілу води в картопляному крохмалі показали, що зв'язана вода у всіх зразках крохмалю становила близько 90%, крім того проведені дослідження свідчать, що в результаті оброблення змінюється розподіл вологи  $PT_{21}$ ,  $PT_{22}$  та  $PT_{23}$ , покращується взаємодія між крохмалем і водою.

Подвійна обробка крохмалю за допомогою MW і HMT робила поверхню гранул крохмалю більш шорсткою і спричиняла утворення більших западин або гребінців, ніж окрема обробка за допомогою MW або HMT, особливо у випадку HMT-MW. Отримані результати підтверджено за допомогою скануючої електронної мікроскопії, оскільки на поверхні крохмальних гранул після модифікації з'явилися деякі западини або вибоїни, а центр поляризованої поперечної структури повільно розширювався. Усі види обробки підвищували температуру клейстеризації та знижували в'язкість, але зменшували пікову в'язкість і в'язкість руйнування крохмального клейстеру. Спектри FT-IR та XRD показали, що HMT та MW руйнують подвійні спіралі та кристалічну структуру картопляного крохмалю. Усі види обробки підвищували вміст RS, але знижували вміст RDS картопляного крохмалю. За тієї самої тривалості нагрівання HMT вміст RS у крохмалі, модифікованому HMT у поєднанні з MW післяобробкою, був значно вищим, ніж у крохмалю, модифікованому HMT у поєднанні з попередньою MW обробкою та окремою вологотермічною обробкою HMT. Результати досліджень, отримані в даному розділі, можуть використовуватися для промислового застосування методів вологотермічного та мікрохвильового оброблення для модифікації крохмалю та виробництва нових видів модифікованих крохмалів із

заданими властивостями.

У п'ятому розділі представлено результати досліджень впливу заміни частини пшеничного борошна на модифікований крохмаль, на якісні характеристики продуктів із пшеничного борошна, включаючи печиво, свіжу локшину та хліб на пару. У цьому дослідженні модифікований картопляний крохмаль (HMTS) готували за допомогою НМТ при 90°C протягом 1,5 години з вмістом вологи 23,56%, тоді як модифіковані картопляні крохмалі (MWS) готували шляхом MW обробки при потужності 400 Вт протягом 5 хвилин з вмістом вологи - 25,0%.

Заміна борошна з низьким вмістом білку (вміст білку  $7,0\% \pm 1,5\%$ ) на модифікований крохмаль HMTS або MWS у кількості понад 5% вплинула на органолептичні показники печива, колір став інтенсивнішим, жовтішим і менш червонуватим. Твердість (включаючи середню твердість, твердість поверхні та максимальну твердість) печива з HMTS або MWS була значно нижчою, ніж контрольного зразку ( $P$  значення  $< 0,05$ ), що вказує на меншу роботу, яку необхідно затратити під час жування, на поверхні печива утворювалася хрустка скоринка. Додавання відповідної кількості HMTS або MWS в рецептуру печива може покращити зовнішній вигляд. Печиво з додаванням крохмалю HMTS або MWS у кількості 15% мало кращі органолептичні показники (зовнішній вигляд, аромат, смак) хрустку скоринку і отримало найвищу оцінку під час дегустаційного аналізу.

Заміна частини пшеничного борошна з низьким вмістом білку (вміст білку  $10,0\% \pm 1,0\%$ ) на HMTS або MWS вплинуло на текстуру та властивості тіста (здатність до розтягування) для локшини. За допомогою кореляційного аналізу було зроблено висновок, що властивості тіста на розтягування, а саме стійкість до розтягування, були надзвичайно значущими, позитивно корелюючими з властивостями стійкості до розтягування та еластичності вареної локшини. Внесення HMTS і MWS у рецептуру локшини значно зменшило час приготування продукту ( $P$  значення  $< 0,05$ ), швидкість

водопоглинання сухої речовини та швидкість втрати сухої речовини значно зросла зі збільшенням кількості модифікованого крохмалю HMTS та MWS. Локшина мала хороші органолептичні та варильні властивості при заміні пшеничного борошна на HMTS менше, ніж на 30%, на MWS – менше, ніж на 20%. Додавання понад 30 % HMTS або 20 % MWS відповідно впливає на деформацію локшини та призводить до збільшення кількості лому. Таким чином, максимальне включення HMTS або MWS має становити 30% та 20% відповідно.

В роботі досліджували доцільність заміни пшеничного борошна (вміст білку  $10,0\% \pm 1,0\%$ ) на HMTS або MWS у рецептурі хліба на пару. Питомий об'єм хліба, приготованого на пару, зменшувався зі збільшення кількості модифікованого крохмалю HMTS або MWS. Питомий об'єм хліба на пару із застосуванням MWS, був нижчим, ніж об'єм хліба на пару із застосуванням HMTS, що свідчить про те, що MWS мав більший вплив на питомий об'єм хліба на пару, ніж HMTS. Експериментальний хліб на пару, показав вищі значення  $L^*$  і  $a^*$ , але нижчі значення  $b^*$ , ніж контрольний зразок, приготований на пару, зміни кольору були більш очевидними зі збільшенням рівня заміни HMTS або MWS, тобто хліб, що містить більше HMTS або MWS має світліший колір. Коли рівень заміщення пшеничного борошна на HMTS або MWS був вищим за 30% і 20% відповідно, відмінності в кольорі ( $\Delta E > 3$ ) між контрольним і експериментальним зразками можна було виявити органолептично. Структурно-механічні властивості хліба на пару при заміні пшеничного борошна на модифікований крохмаль HMTS або MWS більше 30% та 20% відповідно змінилися через порушення структури тіста, це призвело до підвищення твердості, клейкості та жувальної здатності, тоді як значення пружності було знижено, що вказує на утворення більш твердої та щільної структури хліба на пару. Загальні сенсорні показники знижувалися зі збільшення кількості модифікованого крохмалю HMTS або MWS. Загальна сенсорна оцінка була вищою за 80, коли рівень

заміни пшеничного борошна на модифікований крохмаль HMTS або MWS становив не більше 30%, це свідчить про те, що розроблений продукт буде прийнятий споживачами. Крім того, результати визначення рівня глюкози в крові учасників експерименту після прийому їжі в різний час після вживання хліба, печива, локшини показали, що продукти з додаванням HMTS або MWS мають нижчий глікемічний індекс та можуть бути рекомендовані для діабетиків та людей похилого віку.

Проведені дослідження можуть надати цінні рекомендації щодо подальшого застосування картопляного крохмалю, модифікованого НМТ та MW, у виробництві харчових продуктів, а також мають велике значення для просування картоплі, як основного продукту харчування.

**Ключові слова:** крохмаль картопляний, вологотермічна обробка, мікрохвильова обробка, фізико-хімічні властивості, клейстеризація, структурні властивості, дегідратація, засвоюваність, борошняні кондитерські вироби, хлібобулочні вироби, структура, органолептичні властивості, тісто, печиво, хлібці, макарони, локшина, глікемічний індекс, харчова цінність.

## СПИСОК ПУБЛІКАЦІЙ ЗДОБУВАЧА ЗА ТЕМОЮ ДИСЕРТАЦІЇ

### Статті в наукових фахових виданнях України

**1. Chunli Deng, Oksana Melnyk, Yanghe Luo.** EFFECT OF DIFFERENT HEAT MOISTURE TREATMENT CONDITIONS ON POTATO STARCH PHYSICOCHEMICAL PROPERTIES. *Journal of Chemistry and Technologies*, 2022, 30(1), pp.139-150. <http://chemistry.dnu.dp.ua/issue/view/15177> (Scopus, Q4) *(The applicant participated in research, analysis of the results and writing the article)*

**2. Chunli Deng, Oksana Melnyk, Yanghe Luo.** OPTIMIZATION OF HEAT-MOISTURE TREATMENT ON POTATO STARCH AND STUDY ON ITS PHYSICOCHEMICAL PROPERTIES. *Technology Audit and Production*

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**3. Chunli Deng,** Oksana Melnyk, Yanghe Luo. Effects of microwave and heat-moisture treatments on color characteristics, particle size and water distribution of potato starch, Ukrainian Journal of Food Science. 2021,9(2), pp.156-166. <https://doi.org/10.24263/2310-1008-2021-9-2-4> (*The applicant participated in research, analysis of the results and writing the article*)

**4. Chunli Deng,** Oksana Melnyk, Yanghe Luo. INFLUENCE OF SUBSTITUTION OF WHEAT FLOUR WITH MODIFIED POTATO STARCH ON THE QUALITY OF CHINESE STEAMED BREAD, Eastern-European journal of enterprise technologies. 2022, 5/11(119), pp. 12-27. <http://journals.uran.ua/eejet/article/view/265234> (Scopus, Q3) (*The applicant participated in research, analysis of the results and writing the article*)

#### Статті у наукових виданнях інших країн

**5. Chunli DENG,** Oksana MELNYK, Yanghe LUO. Substitution of wheat flour with modified potato starch affects texture properties of dough and the quality of fresh noodles. Food Science and Technology (Campinas), 2023, 43, e128222. <https://doi.org/10.1590/fst.128222> (Scopus, Q2) (*The applicant participated in research, analysis of the results and writing the article*)

**6. Chunli Deng,** Oksana Melnyk, Tatyana Marenkova, Yanghe Luo. Modification in Physicochemical, Structural and Digestive Properties of Potato Starch During Heat-Moisture Treatment Combined with Microwave Pre- and Post-Treatment. Polish Journal of Food Nutrition Science, 2022, 72(3), pp. 249-261. <https://doi.org/10.31883/pjfn/151566> (Scopus, Q2) (*The applicant participated in research, analysis of the results and writing the article*)

**7. Chunli Deng,** Oksana Melnyk, Yanghe Luo. EFFECT OF PARTIAL SUBSTITUTION OF LOW GLUTEN FLOUR WITH MODIFIED POTATO

STARCH ON THE QUALITY OF COOKIES. The scientific heritage, 2022, 87(1), pp.42-47. <http://www.scientific-heritage.com/wp-content/uploads/2022/04/The-scientific-heritage-No-87-87-2022-Vol-1.pdf> (*The applicant participated in research, analysis of the results and writing the article*)

**8. Chunli Deng**, Oksana Melnyk, Yanghe Luo. THE EFFECT OF HEAT-MOISTURE TREATMENT CONDITIONS ON THE STRUCTURE PROPERTIES AND FUNCTIONALITIES OF POTATO STARCH. *Potravinarstvo Slovak Journal of Food Sciences*, 2021,15, pp.824-834. <https://doi.org/10.5219/1647> (Scopus, Q3) (*The applicant participated in research, analysis of the results and writing the article*)

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#### Тези доповідей

**10. Chunli Deng**, Yanghe Luo, Melnyk O. EFFECT OF HEAT-MOISTURE TREATMENT ON TEXTURAL PROPERTIES OF POTATO STARCH. II International Scientific and Practical Conference «The world of science and innovation», London, United Kingdom, September 16-18, 2020, pp.40. (*The applicant participated in research, analysis of the results and writing the article*)

**11. Chunli Deng**, Yanghe Luo, Melnyk O. EFFECT OF HEAT-MOISTURE TREATMENT REACTION CONDITIONS ON THE PASTING PROPERTIES OF POTATO STARCH. II International Scientific and Practical Conference «Topical issues of modern science, society and education», Kharkiv, Ukraine, September 5-7, 2021, pp.120. (*The applicant participated in research, analysis of the results and writing the article*)



**12. Chunli Deng, Yanghe Luo, Melnyk O.** EFFECT OF HEAT-MOISTURE TREATMENT REACTION CONDITIONS ON PARTICLE SIZE DETERMINATION OF POTATO STARCH. III International Scientific and Practical Conference «Modern scientific research: achievements, innovations and development prospects», Berlin, Germany, August 29-31, 2021, pp.53. *(The applicant participated in research, analysis of the results and writing the article)*

**13. Deng Chunli, Luo Yanghe, Melnyk O.** The effect of heat-moisture treatment on digestive properties of potato starch. II International Scientific and Practical Internet Conference «Informational and innovative technologies in hotel and restaurant business, tourism and design», Dnipro – Opole, December 1-2, 2021. pp.33. *(The applicant participated in research, analysis of the results and writing the article)*

**14. Chunli Deng, Melnyk Oksana, Yanghe Luo.** EFFECT OF PARTIAL SUBSTITUTION OF LOW GLUTEN FLOUR WITH MODIFIED POTATO STARCH ON THE COLOR AND TEXTURE PROPERTIES OF COOKIES. VI International Scientific and Practical Conference «MODERN RESEARCH IN WORLD SCIENCE», Lviv, Ukraine, 4-6 September 2022, pp.141-147. *(The applicant participated in research, analysis of the results and writing the article)*

## ANNOTATION

*Deng Chunli* Technology of physically modification of potato starch and their applications in food products. Qualified scientific work as manuscript.

Dissertation for the degree of Doctor of Philosophy in specialty 181 – «Food technology» – Sumy National Agrarian University, Sumy, 2023.

The thesis is dedicated to the development of the technology of modified potato starch by using physically modification of heat-moisture treatment (HMT) combined with microwave treatment (MW), and thesis is dedicated to the scientific substantiation development of the technology of food products with incorporation of modified potato starch.

This first section of this thesis presents the results of an analytical review of literary sources, namely, the relationships between the properties and structure of starch, the analysis of physical, chemical and enzymatic modification of starch, and the application of modified starch in food. HMT and MW are the most appealing physical modification method of starch due to their advantages of environmental protection, no reagent by-product, easy control process and safe for industrial production. Therefore, the literature review especially focuses on the effects of HMT and MW on the properties of starch.

On the basis of analyzing the literature, the scientific problem of this thesis is expounded in the second section, the choice of objects and the direction of researches is substantiated, methods of fundamental and applied researches are resulted. In accordance with the objectives of the study, a work program was formulated, which not only provides the studies of modification technological processes of single heat-moisture treatment of native potato starch to determine the optimal parameters and the studies of modification technological processes of heat-moisture treatment combined with microwave pre- and post-treatment, but also provides the determination methods of physicochemical properties of starch paste, physicochemical properties of starch granules, multiscale structure of starch granules, and provides the potential application of heat-moisture treatment

and microwave treatment modified potato starch in food products (cookies, fresh noodles and steamed bread). In order to achieve the established tasks, the research methods and techniques are demonstrated and their descriptions are given.

The third section of this thesis systematicall studies the effects of HMT conditions the morphological, physicochemical and in vitro digestion properties of potato starch and its process optimization presented a comprehensive understanding of the effects of HMT conditions on functional and digestibility properties of starch, as well as the related mechanism, which would provide a useful theoretical basis for further studies on improving the application of thermal treatment technology in starch modification.

The transparency and retrogradation stability of potato starch after HMT were reduced, solubility and swelling power varied with the gelatinization temperature. HMT can significantly affect the textural properties of potato starch and the hardness, gumminess, chewiness and resilience of HMT starch gels first increased significantly and then decreased with the extension of treatment time. Short heating time (<1.5 h), relatively low heating temperature (<100°C) and low moisture content (<25%) of HMT process can significantly enhance the texture properties of HMT starch gels. The HMT potato starch pasting properties results indicated that it is necessary to select appropriate heat-moisture treatment conditions for the preparation of vermicelli food by using the HMT potato starch in order to obtain better edible quality.

HMT had great effect on the particle size, morphological, structural, and digestive properties of potato starch. HMT led to large particle size, rough surface of starch granules and resulted in hollow structure located at the hilum of potato starch granules. XRD results showed an increased relative crystallinity and transformed crystalline structure from B-type to C-type with the extension heat moisture treatment. FTIR spectroscopy results indicated that the heat moisture treatment may result in the breaking of starch molecular chain or the breaking of the associative hydrogen bond of starch molecule. *In vitro* digestion results

showed that HMT process could significantly decrease rapidly digested starch (RDS) content, but increase resistant starch (RS) content, which indicating HMT starches could potentially become sources of industrial-resistant starch and as low-calorie food ingredients.

The optimized process parameters of setback viscosity of HMT were as follow: the temperature was 90°C, the time was 1.5 h and the moisture content was 23.56%. Under such conditions the maximum theoretical setback viscosity value was 3871 cP. The verification experiment showed the actual mean setback viscosity was 3677 cP and there was little error between the actual setback viscosity and theoretical setback viscosity. Compared with native potato starch (NS), optimized heat-moisture treatment modified starch (HMTS) had lower peak viscosity (2966 cP), lower hold viscosity (2882 cP) and lower breakdown viscosity (84.5 cP), but higher paste temperature (71.1°C), higher final viscosity (6559 cP) and setback viscosity (3677 cP). The hardness, cohesiveness, gumminess, chewiness of NS gel were 2706 g, 0.63, 1700 g, 1404 g·mm, respectively, while the hardness, cohesiveness, gumminess, chewiness of HMTS gel were 6082 g, 0.73, 4920 g, 3570 g·mm, respectively. Compared with the NS gel, the hardness, cohesiveness, gumminess and chewiness of HMTS gel were increased significantly, and there was no significant difference in springiness and resilience. The retrogradation of NS and HMTS increased with the extension of storage time, and the HMTS had higher retrogradation than that of NS, indicating that potato starch modified by heat-moisture treatment was more prone to retrogradation. *In vitro* digestion results showed that HMTS had higher slowly digested starch (SDS) content and RS content than that of NS, but lower RDS content. Compared to the native potato starch, the RDS decreased by 10.05%, the SDS increased by 5.06% and the RS increased by 2.48%, respectively in the optimized HMT potato starch.

The effects of HMT assisted by MW pre- and post-treatment on the morphological, physicochemical and *in vitro* digestion properties of potato starch

were also evaluated in the fourth section of this thesis. This research presented a comprehensive understanding of the effects of HMT and MW bi-directional modifications on functional and digestibility properties of starch, as well as the related mechanism, which would provide a useful theoretical basis for further studies on improving the application of microwave technology in starch modification.

All the starch samples modified by single HMT, single MW, and dual modification of HMT combined with MW showed lower swelling power than that of native starch when the test temperature was 65–85°C, while opposite results were obtained at 95°C. All the modified starch samples showed higher solubility than that of native starch when test temperature was 75–95°C. Single MW, short-time single HMT and short-time HMT combined with MW pretreatment can enhance the repeated freeze-thaw stability of potato starch pastes, while long-time HMT ( $\geq 4$ h) could weaken the freeze-thaw stability of potato starch. Dual modification of HMT and MW had greater effects on starch retrogradation than that of single HMT or single MW, moreover, HMT combined with MW pretreatment also had greater effects on starch retrogradation than that HMT combined with MW post-treatment. HMT heating time had great significant effect on starch paste transparency, dual modification of HMT combined with MW had greater effect on the transparency of starch paste than that of single HMT and MW. The hardness, cohesiveness, gumminess and chewiness of all the HMT modified potato starch gel (including single HMT, HMT combined with MW) decreased with the extension of heating time. The HMT potato starch pretreated by MW had higher hardness value than that of HMT potato starch post-treated by MW.

HMT treatment caused a slight increase of lightness ( $L^*$  values), while single MW treatment caused a slight decrease of lightness, indicating that the color of all the HMT treated samples (HMT, MW-HMT, HMT-MW) became brighter and the color of the single MW treated sample (MWS) became darker. The starch particle size distribution experiments showed that D50, D (4,3) and D (3,2) of all

treated starch were higher than NS, while the value of S.S.A. was significantly decreased by MW and HMT, indicating that MW and HMT treatments can caused expansion, partial gelatinization and agglomeration of starch granules, resulting in large particle size of starch granules, which were consistent with the results of scanning electron microscopy (SEM). The results of water distribution in potato starch showed that the bound water in all starch samples was the main water which at least accounted for 90%, the MW treated starch had three different state water, while NS and single HMT treated starch only had two different state water. There were significant differences of  $PT_{21}$  and  $PT_{22}$  between NS and all treated starch, NS had the lowest  $PT_{21}$  but highest  $PT_{22}$ , indicating that MW and HMT treatments could change the water distribution and improve the interaction between starch and water.

Dual starch modification *via* MW and HMT made the surface of starch granules rougher and caused more serious depressions or scallops than single modification with MW or HMT, especially in the case of HMT-MW, which can be verified from the scanning electron microscopy, normal light and polarized light microscopy that some depressions or potholes appeared on the surface of starch granules after modification, and the center of polarized cross structure slowly expanded. All the treatments increased the pasting temperature and setback viscosity but decreased peak viscosity and breakdown viscosity of starch. The FT-IR and XRD spectra implied that HMT and MW destroyed the double helices and crystalline structure of potato starch. All treatments increased the content of RS but reduced the content RDS of potato starch. Under the same HMT heating duration, the RS content of starch modified by HMT combined by with MW post-treatment was significantly higher than that of starch modified by HMT combined by MW pre-treatment and single HMT. The information obtained in this research might be beneficial to the industrial applications of microwave and heat-moisture techniques deployed to modify starch and eventually produce new starch materials satisfying the potential consumer requirements.

The fifth section of this thesis also presents the results of the effects of substitution of wheat flour with potato starch modified by heat-moisture treatment and microwave treatment on the quality characteristics of three typical food products including cookies, fresh noodles and steamed bread, which would provide a beneficial theoretical basis for further research on the application of HMT and MW modified starch in food. In this research the HMT modified potato starches (HMTS) were prepared by HMT at 90°C for 1.5 h with 23.56% moisture content of starch (the optimized process parameters of HMT), whereas the MW modified potato starches (MWS) were prepared by MW at 400 W power for 5 min with 25% moisture content of starch.

The substitution of low protein flour (the protein content was  $7.0\% \pm 1.5\%$ ) with HMTS or MWS in quantity above 5% made cookies brighter, yellower, and less reddish. The differences in color between the control and experimental cookies were detectable by the human eye when the substitution amount of low protein flour with HMTS or MWS reached 5%. The hardness (included average hardness, surface hardness and max hardness) of cookies with HMTS or MWS was significantly lower than of control ( $P_{\text{value}} < 0.05$ ), but higher crispy value, indicating less work to be consumed when chewing. The addition of appropriate amount of HMTS or MWS to cookies could improve the appearance. Cookies with addition of HMTS or MWS powder in the amount of 15% not only had crispy taste, but also had the highest acceptability score and yellowest color, therefore, the appropriate incorporation of HMTS or MWS was 15%. The present research might help to enlarge the application of modified potato starch in confectionery products.

Substitution of with HMTS or MWS altered the texture and tensile properties of dough. Through correlation analysis, it has been concluded that the dough tensile properties of resistance to extension and extensibility were extremely significant positive correlated with the cooked fresh noodles tensile properties of tensile strength and elasticity. Substitution wheat flour (the protein content was

10.0%±1.0%) with HMT and MW modified potato starch (HMTS and MWS) significantly decreased the optimal cooking time of fresh noodles ( $P_{\text{value}} < 0.05$ ), the dry matter water absorption rate and loss rate of dry matter significantly increased with the increase of substitution amount of HMTS and MWS. When the incorporation amount of HMTS was less than 30% and the incorporation amount of MWS was less than 20%, the noodles could maintain good organoleptic and cooking quality attributes. More than 30% of HMTS or 20% MWS will deform the noodles and cause breakage. Therefore, the maximum incorporation of HMTS or MWS should be 30%, 20%, respectively. The present research results can be applied to the noodle industry, and it might also help to enlarge the application of modified potato starch in cooking noodle-like food.

The appropriated amount of substitution of wheat flour (the protein content was 10.0%±1.0%) with HMTS or MWS not only can maintain the quality of steamed bread, but also increase the nutrition of steamed bread. The specific volume of steamed bread decreased with more incorporation of HMTS or MWS. The specific volume of steamed bread buns made by incorporating MWS was lower than that of steamed bread made by incorporating HMTS, which indicated that MWS had greater impact on the specific volume of steamed bread than HMTS. The experimental steamed bread showed higher  $L^*$  and  $a^*$ , but lower  $b^*$  values than those of the control steamed bread, and the color changes was more obvious with the increase of substitution level of HMTS or MWS, indicating that steamed bread with more incorporation of HMTS or MWS displays lighter transparent color. When the substitution level of HMTS or MWS was higher than 30%, 20%, respectively, the differences in color ( $\Delta E > 3$ ) between the control and experimental steamed bread can be detectable by the human eye. Texture properties of steamed bread were affected with substitution due to the disruption of dough structure, and the incorporation of HMTS or MWS led to higher value of hardness, gumminess and chewiness, whereas the value of springiness,



cohesiveness and resilience were reduced, which indicated that the incorporation of HMTS or MWS led to firmer and denser structure of steamed bread. The sensory total scores decreased with more incorporation of HMTS or MWS. The total sensory score of was higher than 80 when the substitution level of wheat flour with HMTS or MWS was less than 30%, indicating the produced CSB can be accepted by consumers. Based on the above research results of steamed bread quality, the optimal substitution of wheat flour with HMTS or MWS was 30%. Moreover, the results of postprandial blood glucose levels of participants at different times after eating steamed breads indicated that products prepared with incorporation of HMTS or MWS were more suitable for diabetics or the elderly.

This research can provide valuable guidance for further application of HMT and MW modified potato starch in wheat-based products, and it is also of great significance for promoting potato as staple food.

**Keywords:** potato starch, moisture-thermal treatment, microwave treatment, physical and chemical properties, pasteurization, structural properties, dehydration, digestibility, flour confectionery, bakery products, structure, organoleptic properties, dough, cookies, bread, makarony, noodles, glycemic index, nutritional value.

## **LIST OF THE APPLICANT'S PUBLICATIONS ON THE TOPIC OF THE DISSERTATION**

### **Articles in scientific professional publications of Ukraine**

1. **Chunli Deng**, Oksana Melnyk, Yanghe Luo. EFFECT OF DIFFERENT HEAT MOISTURE TREATMENT CONDITIONS ON POTATO STARCH PHYSICOCHEMICAL PROPERTIES. Journal of Chemistry and Technologies, 2022, 30(1), pp.139-150. <http://chemistry.dnu.dp.ua/issue/view/15177> (Scopus, Q4)  
(*The applicant participated in research, analysis of the results and writing the article*)

**2. Chunli Deng, Oksana Melnyk, Yanghe Luo.** OPTIMIZATION OF HEAT-MOISTURE TREATMENT ON POTATO STARCH AND STUDY ON ITS PHYSICOCHEMICAL PROPERTIES. Technology Audit and Production Reserves, 2022, 3(3(65)), pp. ,43-49.

<http://journals.uran.ua/tarp/issue/view/15640> (*The applicant participated in research, analysis of the results and writing the article*)

**3. Chunli Deng, Oksana Melnyk, Yanghe Luo.** Effects of microwave and heat-moisture treatments on color characteristics, particle size and water distribution of potato starch, Ukrainian Journal of Food Science. 2021,9(2), pp.156-166. <https://doi.org/10.24263/2310-1008-2021-9-2-4> (*The applicant participated in research, analysis of the results and writing the article*)

**4. Chunli Deng, Oksana Melnyk, Yanghe Luo.** INFLUENCE OF SUBSTITUTION OF WHEAT FLOUR WITH MODIFIED POTATO STARCH ON THE QUALITY OF CHINESE STEAMED BREAD, Eastern-European journal of enterprise technologies. 2022, 5/11(119), pp. 12-27. <http://journals.uran.ua/eejet/article/view/265234> (Scopus, Q3) (*The applicant participated in research, analysis of the results and writing the article*)

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**5. Chunli DENG, Oksana MELNYK, Yanghe LUO.** Substitution of wheat flour with modified potato starch affects texture properties of dough and the quality of fresh noodles. Food Science and Technology (Campinas), 2023, 43, e128222. <https://doi.org/10.1590/fst.128222> (Scopus, Q2) (*The applicant participated in research, analysis of the results and writing the article*)

**6. Chunli Deng, Oksana Melnyk, Tatyana Marenkova, Yanghe Luo.** Modification in Physicochemical, Structural and Digestive Properties of Potato Starch During Heat-Moisture Treatment Combined with Microwave Pre- and Post-Treatment. Polish Journal of Food Nutrition Science, 2022, 72(3), pp. 249-261. <https://doi.org/10.31883/pjfn/151566> (Scopus, Q2) (*The applicant*

*participated in research, analysis of the results and writing the article)*

**7. Chunli Deng**, Oksana Melnyk, Yanghe Luo. EFFECT OF PARTIAL SUBSTITUTION OF LOW GLUTEN FLOUR WITH MODIFIED POTATO STARCH ON THE QUALITY OF COOKIES. The scientific heritage, 2022, 87(1), pp.42-47. <http://www.scientific-heritage.com/wp-content/uploads/2022/04/The-scientific-heritage-No-87-87-2022-Vol-1.pdf> *(The applicant participated in research, analysis of the results and writing the article)*

**8. Chunli Deng**, Oksana Melnyk, Yanghe Luo. THE EFFECT OF HEAT-MOISTURE TREATMENT CONDITIONS ON THE STRUCTURE PROPERTIES AND FUNCTIONALITIES OF POTATO STARCH. *Potravinarstvo Slovak Journal of Food Sciences*, 2021, 15, pp.824-834. <https://doi.org/10.5219/1647> (Scopus, Q3) *(The applicant participated in research, analysis of the results and writing the article)*

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**10. Chunli Deng**, Yanghe Luo, Melnyk O. EFFECT OF HEAT MOISTURE TREATMENT ON TEXTURAL PROPERTIES OF POTATO STARCH. II International Scientific and Practical Conference «The world of science and innovation», London, United Kingdom, September 16-18, 2020, pp.40. *(The applicant participated in research, analysis of the results and writing the article)*

**11. Chunli Deng**, Yanghe Luo, Melnyk O. EFFECT OF HEAT-MOISTURE TREATMENT REACTION CONDITIONS ON THE PASTING PROPERTIES OF POTATO STARCH. II International Scientific and Practical

Conference «Topical issues of modern science, society and education», Kharkiv, Ukraine, September 5-7, 2021, pp.120. (*The applicant participated in research, analysis of the results and writing the article*)

**12. Chunli Deng, Yanghe Luo, Melnyk O.** EFFECT OF HEAT-MOISTURE TREATMENT REACTION CONDITIONS ON PARTICLE SIZE DETERMINATION OF POTATO STARCH. III International Scientific and Practical Conference «Modern scientific research: achievements, innovations and development prospects», Berlin, Germany, August 29-31, 2021, pp.53. (*The applicant participated in research, analysis of the results and writing the article*)

**13. Deng Chunli, Luo Yanghe, Melnyk O.** The effect of heat-moisture treatment on digestive properties of potato starch. II International Scientific and Practical Internet Conference «Informational and innovative technologies in hotel and restaurant business, tourism and design», Dnipro – Opole, December 1-2, 2021. pp.33. (*The applicant participated in research, analysis of the results and writing the article*)

**14. Chunli Deng, Melnyk Oksana, Yanghe Luo.** EFFECT OF PARTIAL SUBSTITUTION OF LOW GLUTEN FLOUR WITH MODIFIED POTATO STARCH ON THE COLOR AND TEXTURE PROPERTIES OF COOKIES. VI International Scientific and Practical Conference «MODERN RESEARCH IN WORLD SCIENCE», Lviv, Ukraine, 4-6 September 2022, pp.141-147. (*The applicant participated in research, analysis of the results and writing the article*)

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## **LIST OF CONDITIONAL DESIGNATIONS**

CHMT: starch modified by heat-moisture treatment with different moisture content

HMT: heat-moisture treatment

HMT-MW: heat-moisture treatment in combination with microwave post-treatment

HMTS: potato starch modified by single heat-moisture treatment under optimized conditions

MW: microwave treatment

MW-HMT: heat-moisture treatment in combination with microwave pre-treatment

MWS: potato starch modified by single microwave treatment

NS: native potato starch

THMT: starch modified by heat-moisture treatment with different temperature

tHMT: starch modified by heat-moisture treatment with different heating time

RDS: rapidly digested starch

RS: resistant starch

SDS: slowly digested starch

SEM: scanning electron microscopy

XRD: X-ray diffraction

FT-IR: Fourier transform infrared spectroscopy

## INTRODUCTION

**Actuality of the Topic.** Potato (*Solanum tuberosum* L.) as the fourth largest food crop after wheat, rice and maize, plays an important role in the world food security [1]. According to the Food and Agriculture Organization of the United Nations (FAO) data, potatoes were cultivated in 157 countries and regions with a total planting area of 16.49 million hectares and a total output of 359 million tons in 2020. In 2020, the potato production in Asia accounted for 49.7% of the global potato production, while the potato production in Europe accounted for 30%, which indicated that Asia and Europe were the main potato producing areas in 2020 [2]. Starch is the main component of potato tubers and the starch content varies with cultivar and plant growth stage, generally accounting for 66-80% of the dry weight basis [3]. Potato starch is extensively utilized as food ingredients, thickeners, excipients or pharmaceutical fillers in variety food systems for its unique properties of large granule size, neutral flavor, high pasting viscosity and swelling power, high clarity of starch paste and ability to form thick viscoelastic gels upon heating and subsequent cooling [4-5].

However, native potato starch exhibits poor shear and heat resistance, poor water solubility and thermal stability, which limits the application of starch [6]. Various chemical, enzymatic and physical methods or their combinations have been used to overcome the inherent shortcomings of native starch in order to acquire suitable properties for special applications [7-9]. Chemical method is the most widely used modification method for its low cost of production and high control over final product customization [10], but partly due to the formation of chemical residues, it is usually considered to environmentally unfriendly [11]. Enzymatic method is effective with fewer by-products, but the cost is high and the reaction process is difficult to control, while the physical method is simple, economical and easy for commercialization [5]. Considerable interests in physical



modification methods to have been generated due to absence of chemical contamination, food safety, environmental friendliness and low cost of production [12].

Heat-moisture treatment (HMT) is one of the most important physical modification methods of starch, which refers to the utilization of high moisture content (10-35%) and temperature (90-120°C) for a period of treatment time (15 min- 16 h) [13]. Many previous researches have confirmed that HMT directly affects starch digestibility through the formation of slowly digestible starch (SDS) and resistant starch (RS) and the reduction of rapidly digestible starch (RDS), which is very important for realizing consumers' health benefits [14-17]. Microwave treatment (MW) is another appealing physical modification method of starch for its effectively heating, high yield and potentially good quality of products [18]. MW was used in modification of sago (*Metroxylon sagu*) [19], potato [20], millet starches [21] and other starches [22]. Many researches have studied the influence of MW on the digestibility of starch [16, 19, 23], and the results of these researches indicating that MW could increase the content of RS and SDS.

Obesity represents a growing global public health concern in the 21<sup>st</sup> century [24]. It is predicted that 20% of the world's adult population will be obese by 2030, and in develop countries such as Australia, Canada, the UK and the USA, the figure is expected to reach upwards of 50% [25]. Obesity is associated with heightened all-cause mortality and significantly increases the risk of including type 2 diabetes, cardiovascular disease, respiratory dysfunction and several other cardiometabolic comorbidities [26]. During the past ten years, the prevalence of diabetes in Chinese adults has been maintaining at 10% [27]. An analysis of a China nationally representative survey shows that China has more than 130 million adults with type 2 diabetes, and 350 million individuals with prediabetes (3) [28]. Therefore, it is a feasible strategy to develop functional foods products

with reduced glycemic index (GI) to solve these health problems, and adding modified starch rich in RS, SDS in food is one of the effective methods.

Nowadays, with the improvement of people's living standards and the emphasis on healthy diet, special nutritional food products have attracted more and more attention around the world [29]. Since most of the wheat products belong to high glycemic index (GI) foods, enhancing food products with functional components has the potential to be beneficial [30]. Strategies for developing food products with low GI remain to be developed to help people with diabetes and other diseases [31]. Partial substitution of wheat flour with whole flour or other functional ingredients can enhance the nutritional quality of food products. However, the dough rheological properties and the product quality may be altered by substitution of wheat flour with other types of low-gluten flour [32]. Therefore, it is necessary to investigate the effects of substitution of wheat flour with other types of low-gluten flour (i.e. modified starch) on the quality characteristics of dough and the final products.

**Connection of work with scientific programs, plans, themes.** Scientific research was carried out within the framework of the thematic plan of research works according to topic 0119U103484 "Scientific grounding and development of technologies of food and culinary products using innovative types of raw materials", Department of Food Technology, Sumy National Agrarian University, Ukraine, and the College of Food and Biological Engineering, Hezhou University, China.

**The purpose and objectives of this research. The aim of the research is** to develop technology of physically modified starches with heat-moisture treatment (HMT) and microwave treatment (MW) and investigate the effects of their application in the production of food products.

**To achieve this aim, the following objective should be accomplished:**

- investigate the effects of heat-moisture treatment conditions on potato starch physicochemical properties, mainly include swelling power, solubility,

freeze-thaw stability, retrogradation, transparency and textural properties.

- investigate the effects of heat-moisture treatment reaction conditions on the structural and digestion characteristics of potato starch, mainly include paste viscosity properties, particle size, morphological properties, crystal structure and *in vitro* digestibility.

- optimize the modification process of potato starch by heat moisture treatment using Box-Behnken response surface methodology.

- investigate the effects of heat-moisture treatment combined with microwave pre- and post-treatment on potato starch physicochemical properties, mainly include color characteristics, particle size, water distribution, swelling power, solubility, freeze-thaw stability, retrogradation, transparency and textural properties.

- investigate modification in physicochemical, structural and digestive properties of potato starch during heat-moisture treatment combined with microwave pre- and post-treatment, mainly include paste viscosity properties, morphological properties, crystal structure and *in vitro* digestibility.

- investigate the effects of partial substitution of wheat flour with modified potato starch on the quality of cookies, Chinese steamed bread, noodles, mainly include the texture properties of dough, the textural properties, color characteristics and the sensory acceptance of products.

**The object of this research** -the potato starch modification technology of heat-moisture treatment combined with microwave treatment and its application in food products.

**Research methods-** experimental determination methods of on the physicochemical properties, morphological characteristics, crystal structure and *in vitro* digestion properties of potato starch modified by heat-moisture treatment and microwave treatment; experimental determination methods of the effects of partial substitution of wheat flour with modified potato starch on the quality of final products, mainly include the texture properties of dough, the textural

properties, color characteristics and the sensory acceptance of products; mathematical methods for experiment planning and experimental data processing.

**The scientific novelty** of the obtained results is as follows:

- the starch modification methods used in this research are single HMT and HMT combined with MW pre- and post-treatment, which only involve water and heat, and will not cause any pollution to the environment. The modification processes are simple, the operations are convenient, and no subsequent treatments are required. Therefore, the modification technologies of the research have board application prospects in the improvement of starchy food quality.

- there are few systematic researches on the effects of single HMT conditions on the structure properties and functionalities of potato starch, and there are also few researches on the effects of HMT combined with MW pre- and post-treatment on physicochemical, structural and digestive properties of potato starch. The results of this research provide a theoretical basis for the application of single HMT and dual modification of HMT combined with MW pre- and post-treatment in potato industrialization.

- for the first time, the viscosity properties, gel textural properties and *in vitro* digestion characteristics of the HMT modified starch and HMT and MW bi-directional modified starch were systematically analyzed to provide reliable evidence for their application in food industry.

- the regularities of the effects of wheat flour substitution with modified potato starch on properties of mixed dough and quality of cookies, steamed bread and noodles are substantiated.

- experiments on cookies, steamed bread and noodles products confirmed that adding a suitable dose of modified potato starch can improve the quality of the products, making the products have good texture properties and sensory acceptability, which is of positive significance for promoting the process of potato staple food.

**The practical significance of the obtained results.** The research presented

a comprehensive understanding of the effects of HMT, HMT and MW bi-directional modifications on functional and digestibility of starch, as well as the related mechanism, which would provide a useful theoretical basis for further studies on improving the application of hydrothermal or microwave technology in starch modification. Meanwhile, the modified starch obtained in the research has high content of RS and SDS, and had good cold paste gel texture, which is suitable for the development of low GI food, as well as suitable for use in vermicelli and noodle products.

The recipes of cookies, steamed breads and noodles with the addition of the obtained modified starch were investigated, and the optimal substitution amounts of wheat flour with HMT modified potato starch and MW modified potato starch were given. A technological scheme of cookies, steamed bread and noodles production and a project of technological documentation were developed (ТУ У 00383403.001:2023 Модифікований картопляний крохмаль (фізична модифікація), ТІ печиво «МоКа», ТІ хлібці «Парові», ТІ локшина «Легка»).

The results of the dissertation can be used in the educational process when studying the disciplines "Theoretical foundations of food production", "General technologies of food production", "General technologies of starchy food products", as well as during the as well as during the conduct of fundamental and applied research in the direction of the development of technologies of flour products or processing starch raw materials.

**The personal contribution of the applicant** is to plan and conduct experimental research in laboratory, perform mathematical processing and scientific analysis of the experimental results, formulate conclusions and recommendations, prepare of materials for publication, test and implement new technologies of this research.

**Approbation of dissertation results.** Approbation of the scientific and practical results presented in the dissertation was carried out by the applicant personally with the methodical and scientific support of the scientific supervisor.

The main results of the work were reported at the II International Scientific and Practical Conference "The world of science and innovation", London, United Kingdom, September 16-18, 2020; II International Scientific and Practical Conference "Topical issues of modern science, society and education", Kharkiv, Ukraine, September 5-7, 2021; III International Scientific and Practical Conference "Modern scientific research: achievements, innovations and development prospects", Berlin, Germany, August 29-31, 2021; II International Scientific and Practical Internet Conference "Informational and innovative technologies in hotel and restaurant business, tourism and design", Dnipro – Opole, December 1-2, 2021; VI International Scientific and Practical Conference "MODERN RESEARCH IN WORLD SCIENCE", Lviv, Ukraine, 4-6 September 2022.

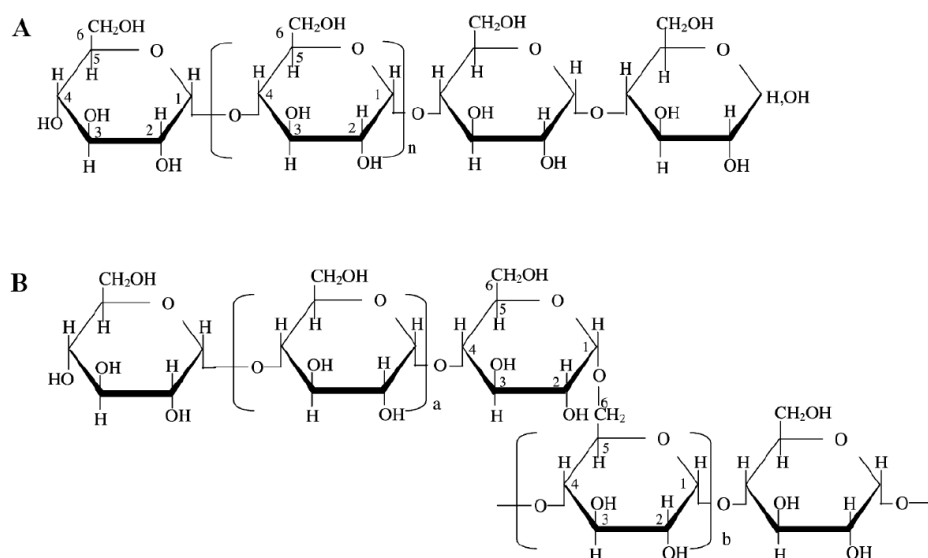
**Publications.** According to the results of the research, the applicant published 14 scientific papers, including 9 journal articles, 2 of which are published in a category B scientific professional publication approved in Ukraine and 2 in a scientific professional publication approved in Ukraine indexed by Scopus, 1 publication in a Polish scientific journal indexed by Web of Science, 1 publication in Slovakia scientific journals indexed by Scopus, 2 in Hungarian scientific journals, 1 publication in a Brazil scientific journal indexed by Scopus; 5 abstracts of scientific conference reports.

**The structure of the dissertation.** The dissertation consists of an introduction, 5 sections, conclusions, a list of references of 236 names, appendices. Main content dissertation is laid out 186 pages of printed text, contains 48 tables and 48 figures.

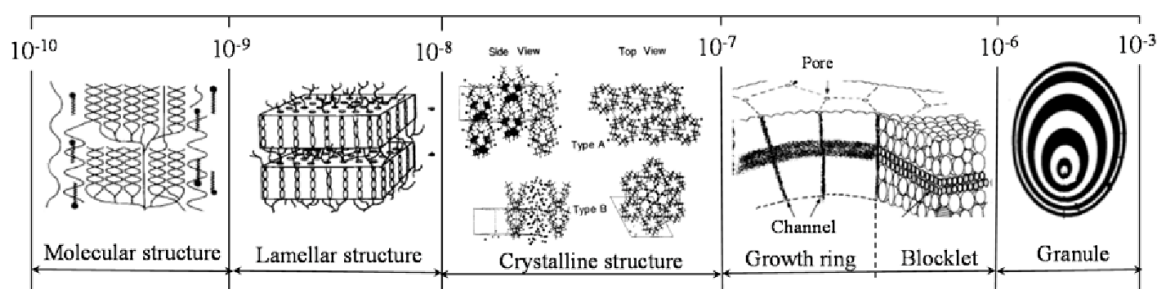
## SECTION 1 STARCH MODIFICATION AND ITS APPLICATION PROSPECT IN FOOD PRODUCTS

### 1.1 The relationships between the properties and structure of starch

Starch is a renewable carbohydrate in nature and plays an important role in various food industries [33]. Starch is a useful biopolymer constituting two primary types of polymeric components of amylose and amylopectin [34]. Amylose molecules are chain-like molecules connected by  $\alpha$ -1,4 glycosidic bonds, and amylopectin is the main chain formed in the same connection method as amylose molecules, and then connected with the main chain by  $\alpha$ -1, 6-glycosidic bonds to form branched starch molecules [35]. The molecular structures of amylose and amylopectin were showed in Fig.1.1. The the ratio of amylose and amylopectin as well as the entanglement between amylose/amylopectin and amylose/amylose have great impact on starch properties, such as water binding capacity, thermal properties, and enzyme susceptibility [36-38]. In general, amylopectin is the main component in most starch granules by weight, whereas amylose accounting for 15-30% [39]. Lots of amylose molecules and amylopectin molecules cluster together to form starch granules, of which are the form starch exists. The two starch polymers of amylose and amylopectin are organized on different scales in the starch granules to form a multi-scale supramolecular, which mainly includes the whole granule ( $<1\ \mu\text{m}$ - $100\ \mu\text{m}$ ), the growth rings ( $100$ - $400\ \text{nm}$ ), blocklet structure ( $20$ - $500\ \text{nm}$ ) the semicrystalline lamellae ( $9$ - $10\ \text{nm}$ ) and the molecular structure ( $0.1\ \text{nm}$ ) [40-42]. The diagram for multi-scale structure of starch was showed in Fig.1.2.



**Fig. 1.1** Structure of amylose and amylopectin (A: Amylose:  $\alpha$ -(1 $\rightarrow$ 4)-glucan; B: Amylopectin:  $\alpha$ -(1 $\rightarrow$ 6) branching points)



**Fig. 1.2** Diagram for multi-scale structure of starch

Structural changes in starch during gelatinization and retrogradation process can greatly affect the rheological behavior, thermal, pasting, and textural properties of starch [43-45], of which determine the main functional properties of its application in food. When the starch suspension is heated to gelatinization temperature, the starch granules begins to absorb water, expand and melt, the crystal structure begins to be destroyed and then melted completely, the amylopectin double helix is dissociated, amylose is leached out, leading to the increase of viscosity of the system, ultimately resulting in the formation of starch paste or starch gel [46-47]. After gelatinization, the completely unwound starch molecular chains gradually recrystallize (rearrange) to form an ordered structure



during the cooling process, resulting in gradually increase in the viscoelasticity and hardness of the starch gel [48].

## **1.2 The methods of characterizing the physicochemical properties of starch**

The properties of starch largely determine the quality and shelf life of starch-based foods. Therefore, more and more scholars pay attention to the inherent properties of native starch, and various modification methods are used to modify starch to obtain special functional characteristics, and compliant with the requirements of specific food products [34, 42]. There are many methods have been used to determine or characterize the rheological properties, thermal properties, pasting, and textural properties of starch.

The rheological properties of starch paste or gel are generally determined by dynamic rheometer [49-50], which are closely related to the gelatinization and retrogradation of starch, including the rheological behavior of starch during gelatinization, the viscoelasticity of starch gel during and after retrogradation, and the rheology of starch paste [51]. The main parameters describing the rheological behaviors of starch are elastic or storage modulus ( $G'$ ), viscous or loss modulus ( $G''$ ), and loss tangent ( $\tan \delta = G''/G'$ ). When  $\tan \delta$  is higher than 1, the viscoelasticity of the material tends to be liquid-like behavior, while  $\tan \delta$  is lower than 1, the viscoelasticity of the material tends to be solid-like behavior; moreover, when  $\tan \delta$  is lower than 0.1, the material has a “gel property”, while  $\tan \delta$  is higher than 0.1, the material is weak gel, and the storage modulus shows a frequency dependence [52].

The thermal properties of starch are very important for further research, development and manufacture of starchy food. Differential scanning calorimetry (DSC), differential thermal analysis (DTA), and thermogravimetric analysis (TGA) are the most common and most important methods for determining the thermal properties of macromolecular substances. Almost all the thermal properties data are obtained by these instruments or methods [53-55]. The

parameters of thermal properties determined by DSC commonly include onset temperature ( $T_o$ ), peak temperature ( $T_p$ ), conclusion temperature ( $T_c$ ) and gelatinization enthalpy ( $\Delta H$ ) [56]. The starch transition temperature ( $T_o$ ,  $T_p$ ,  $T_c$ ) and the  $\Delta H$  determined by DSC are related to the degree of crystallinity, a high degree of crystallinity indicates high transition temperatures [57].

Pasting refers to the process in which starch granules absorb water, swell and disrupt crystalline structure under high temperature [58]. The pasting properties of starch are key properties for the selecting industrial starch resources, which reflect the gelatinization ability, disintegration ability, swelling ability and gel forming ability of starch [59]. The Brabender Viscograph and Rapid Visco Analyser (RVA) have been widely used for the determination of pasting properties of starch [45, 60-61]. The pasting profiles of starch determined by these two instruments reflect structural changes of starch, i.e. granular swelling after water absorption, melting of crystalline structure, leaching of molecular components, granule breakdown, and finally retrogradation [62]. Texture profile analysis (TPA) determined by texture analyzer is the most widely used method to investigate the textural properties of starch gels and starch-based food systems. The texture properties, including hardness, chewiness, springiness, gumminess, and resilience, are the most direct and vital indicators for consumers to evaluate the palatability, mouthfeel, swallowing properties, sensory properties, storage stability and overall acceptability of starch-based food [63-66].

### **1.3 Analysis of existing starch modification technologies**

Starch, as major source of carbohydrate in human nutrition, have been exploited for thousands of years from various plant resources, and its application in modern industrial products has also been developed for decades [67]. However, the inherent shortcomings of starch such as low shear resistance, low thermal resistance, low water solubility and high tendency towards retrogradation limit its

applications in food and non-food industries. Therefore, it is necessary to modify starch by physical, chemical, and enzymatic modification to overcome these deficiencies and widen the application field of starch [7-9]. The following chapters will summarize the application of existing physical, chemical, and enzyme modification techniques in starch modification.

Physical modification of starch can be categorized into thermal physical modification and non-thermal physical modification, among which thermal physical modification mainly includes pre-gelatinization, heat-moisture treatment (HMT) and annealing, whereas non-thermal physical modification mainly includes ultrasonication, ultrahigh pressure treatment, microwave treatment (MW), and gamma irradiation and milling process of starch [68], which are summarized in Table 1.1. Comparing with starches which are modified chemically and enzymatically, physical modified starches are applied more popular in food products because they are considered healthy and green.

**Table 1.1**

The summary of physical modification

Type of physical modification	Definition	Application
Pre-gelatinization	Pre-gelatinization modification of starch is accomplished by heating and by mechanical shearing.	Pre-gelatinized starch has been widely used feed industry, food industry and pharmaceutical industry.
Heat-moisture treatment (HMT)	HMT involves incubation of starch granules at low moisture level (<35% water w/w) during a certain time (15 min-72 h) at a temperature above the glass transition temperature but below the gelatinization temperature (usually 80–140°C).	HMT starch is widely used in various food, such as dressings, noodles, baked foods, batter products, confections, dairy products, creams, fat mimetics, and resistant starches.
Annealing	Annealing involves incubation of starch granules in an excess of water (generally >40% w/w) at a temperature that is above the starch's glass transition temperature and below its gelatinization temperature for certain time.	Annealed starch can be used in frozen food, rice noodles, bread, cakes, and noodles, starch-based functional foods products.

Table 1.1 is continued

Type of physical modification	Definition	Application
Ultrasonication	Ultrasound defines the mechanical waves at a frequency above the upper value of normal human hearing range (>16 kHz). Most of the ultrasonic application in starch is in a starch-water system.	Application of ultrasonication in starch-related industries is improvement of starch extraction, facilitation of acid- and enzyme- catalyzed hydrolysis of starch, production of starch nanocrystals/ nanoparticles
Ultrahigh pressure treatment	Ultrahigh pressure (UHP) treatment consists of subjecting an aqueous slurry of starch granules to a pressure exceeding 400 MPa, which could partial or complete gelatinize the starch with maintenance of the granular form.	Ultrahigh pressure (UHP) treatment can be used as a method for the preparation of pregelatinized starch, cold water-swelling starch, resistant starch.
Microwave treatment (MW)	MW of starch involves the application of electromagnetic waves within the frequency ranging from 300 MHz to 300 GHz, and the corresponding wavelengths ranging from 1 m to 1 mm	Starches modified with microwave heating alone or in combination with other methods have enormous potentials for various food and pharmaceutical applications.
Gamma irradiation	Gamma irradiation is a nonthermal and ionizing physical method and includes the use of radioactive isotopes such as $^{60}\text{Co}$ or $^{137}\text{Cs}$ forms. The radiation dose depends on the radiation source and exposure time.	Radiation processing can promote the efficiency of starch modification by chemical methods.
Milling process	Milling is a gradual mechanical process to produce flour from grain.	Milling process can largely affect the functional properties of final product, either flour or starch.

Chemical modification of starch is to endow starch with desirable physical and chemical properties while maintaining chain integrity by blocking or introducing functional groups, thereby extending its application [69]. Generally, chemical modification of starch is accomplished through derivatization such as acetylation, cationization, acid hydrolysis, oxidation, esterification, etherification, grafting, cross linking and composite modification[70], which are summarized in Table 1.2. However, the application of these techniques is limited due to issues

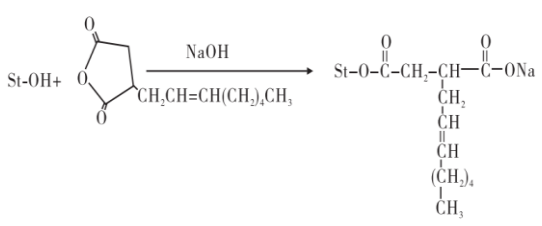
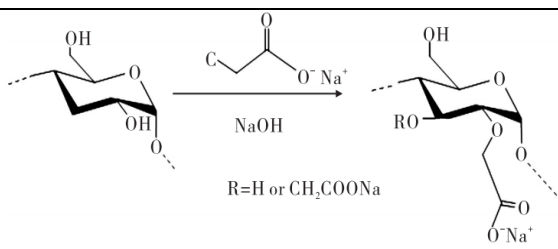
such as consumers' concern for food safety and environmental pollution.

**Table 1.2**

The summary of chemical modification

Type of chemical modification	Reaction mechanism	Application
Oxidation	<p>The semi-crystal structure of starch was destroyed by oxidation modification, and the uniform amorphous polymer matrix was obtained.</p>	Oxidized starch is widely used in papermaking, textile, construction and food industry.
Cross-linking	<p>Step(1) <math>\text{St-OH} + \text{NaOH} \longrightarrow \text{St-O}^-\text{Na}^+ + \text{H}_2\text{O}</math></p> <p>Step(2) <math>\text{St-O}^-\text{Na}^+ + \text{CH}_2\text{-CH-CH}_2\text{-Cl} \longrightarrow \text{St-O-CH}_2\text{-CH-CH}_2 + \text{NaCl}</math></p> <p>Step(3) <math>\text{St-O}^-\text{Na}^+ + \text{CH}_2\text{-CH-CH}_2\text{-O-St} \longrightarrow \text{St-O-CH}_2\text{-CH-CH}_2\text{-O-St}</math></p> <p>The use of a cross-linking agent to form a diester bond or a diether bond between an alcoholic hydroxyl group on a starch molecule, introducing a new chemical bond, and staggering and connecting molecules in starch particles to make two or more starch molecules.</p>	Cross-linked starch is widely used in adhesive production.
Grafting	<p>(a) </p> <p>(b) </p> <p>(c) </p> <p>The initiation sites of graft copolymerization in starch molecules are mainly C<sub>1</sub>-C<sub>2</sub> terminal group and C<sub>2</sub>-C<sub>3</sub> ethylene glycol group.</p>	Grafted starch is widely used in the fields of superabsorbent materials, degradable plastics and films, food and packaging.

Table 1.2 is continued

Type of chemical modification	Reaction mechanism	Application
Esterification	 <p>Esterification is a modification method that improves the properties of starch by introducing new functional groups through the esterification reaction of hydroxyl groups in starch molecules with other substances.</p>	Esterified starch is widely used in food, medicine, textile, papermaking and other fields.
Etherification	 <p>Etherified starch is produced by replacing the alcoholic hydroxyl groups in starch with etherified compounds.</p>	Etherified starch can be used in food, textile and packaging production fields.

In recent decades, enzymatic technology has become an promising modification method of starch for its high yield, few by-products, easy process control and desired functional characteristics of final products [71]. During enzymatic modification of starch, the most common used enzymes include  $\alpha$ -amylase (AM),  $\beta$ -amylase, glucoamylase, debranching enzyme, pullulanase, transferase, cyclodextrin glycosyltransferase, and glucose isomerase [72], which are summarized in Table 1.3.

**Table 1.3**

### The summary of enzymatic modification

Reaction method	Reaction mechanism	Common enzyme agent
Liquefaction	The $\alpha$ -1, 4-glycosidic bonds within the starch molecules are hydrolyzed in a random manner to produce linear and branched oligosaccharides with varying lengths.	Medium-temperature $\alpha$ -amylase High-temperature $\alpha$ -amylase

Table 1.3 is continued

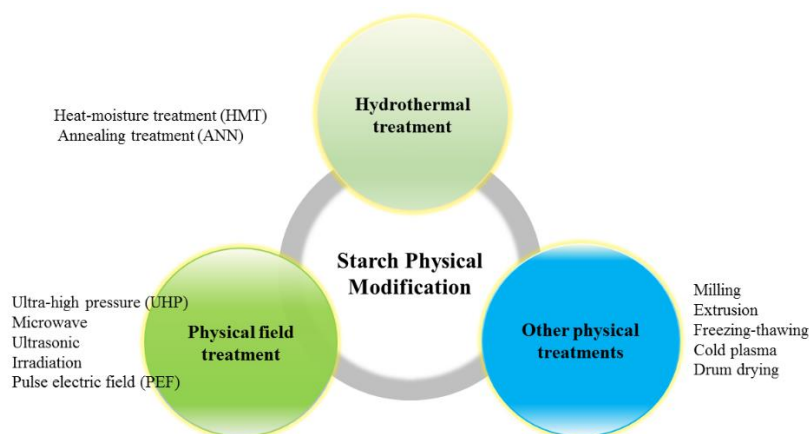
Reaction method	Reaction mechanism	Common enzyme agent
Saccharification	The non-reductive ends of $\alpha$ -1,4 glucan molecules such as starch and dextrin are continuously hydrolyzed by glucose or maltose.	Glucoamylase $\beta$ -amylase
Debranch	The $\alpha$ -1,6 glycosidic bonds within the $\alpha$ -1,4 glucan molecule can be specifically hydrolyzed.	Pullulanase Isoamylase Amylopulanase
Transglycosidation	The process of transferring sugar groups from one glycoside to another.	Cyclodextrin glucosyltransferase $\alpha$ -glucosidase
Isomerization	The reaction of substrate molecules to form isomers under the action of catalyst	Glucose isomerase Starch branching enzyme Trehalose synthase

All these modification methods are aimed at changing the structure of starch, affecting the functional characteristics and digestibility of starch, so that the modified starch acquires the desired properties to meet the requirement of food industry applications. Due to the risk of reagent residue and the environmental pollution caused by chemical reagents in chemical modification, the application of chemical modified starch in food has certain potential safety hazards, while enzymatic modification is relatively expensive and difficult to control the reaction process, enzymatic modified starch is not often used in the food industry. In contrast, physical modified starch is widely applied in food industry for its safety, non-toxicity, cost efficiency and easy commercialization.

#### **1.4 Theoretical basis of processing native starch using physical modification methods**

Physical modifications of starches are generally considered to be those modifications that destroy or produce changes in starch properties affected by physical treatments alone without introducing any chemical modifications to the starch polysaccharide molecule (except for limited glycosidic bond cleavage (depolymerization), leading to only some reduction in average molecular weight) [73]. These structural changes can alter the properties and functionalities of the starch, including the properties of its hot pastes and gels and their digestibility.

Physical modification of starch can be categorized into thermal mechanical force treatments, physical field treatments and other physical treatments, as shown in Fig.1.3. The physicochemical properties, functional properties and digestive properties of starch are altered differently depending on the methods and the extent of modification, which determine its application in food industry.



**Fig.1.3** Common physical modification of starch

The effects of single HMT and MW on functionalities and structural properties of starch have been investigated by many researchers as reviewed by Schafranski et al.[74] and Oyeyinka et al.[22]. However, Due to the diversity of reaction conditions, including the botanical source, moisture content, temperature, treatment time, heat source, and cooling progress, it is difficult to define the properties of HMT modified starch and the mechanism of dual modification combined with HMT and MW is far from being fully understood. Therefore, this research selected the physical modification methods of single HMT and heat–moisture treatment combined with microwave pre – and post – treatment to modify potato starch. The objective of this research was to evaluate the effects single HMT, HMT and MW bi-directional modifications on functional and digestibility properties of starch, as well as the related mechanism. The purpose of this research was to provide new knowledge on HMT, HMT and MW bi-directional modifications affecting potato starch and provide a useful theoretical basis for further studies on improving the application of hydrothermal and microwave technology in starch modification, we were also expecting to broaden



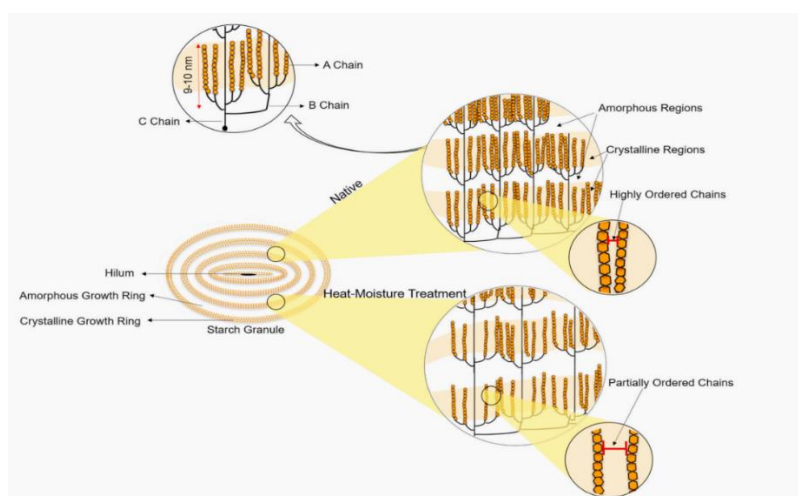
the application of HMT starch, HMT and MW bi-directional modified starch in food field. Therefore, the following section focuses on these two physical modification methods (HMT and MW) and their effects on the properties and structure of starch.

#### **1.4.1 Heat-moisture treatment**

Heat-moisture treatment (HMT), the currently most studied method of physical modification of starch, refers to the process in which starch is performed at high temperature (80–140°C) for a period of time ranging from 15 min to 24 h with a low moisture content (10–35%) [75-77]. Not only the HMT process parameters of heating temperature, heating time and moisture content of starch could affect the effect of HMT on starch modification, but also the characteristics of starch such as botanical source, structure, amylose/amylopectin ratio and organizational form could affect the effect of HMT on starch modification [78]. Among all parameters, the moisture content of starch and the heating temperature of HMT play a vital role in the effect of HMT on starch [73].

HMT enhances the interactions between the starch chains, leading to the disruption of crystalline structure and separation of the double helical structure; the broken crystals subsequently rearrange themselves (Fig. 1.4) [74]. HMT facilitates polymer chain interactions by disrupting the crystal structure and dissociating the double helix in the amorphous region, and then rearranging the disrupted crystals. The disruption of crystalline near the starch granule surface caused by HMT conducive to the attack of  $\alpha$ -amylase within it. When the crystals are not destroyed by HMT, the sensitivity of enzyme is increased due to the destruction of the double helices in amorphous regions. The disruption facilitates the access of enzyme into the interaction sites between amylose chains during polymer chains rearrangement [79]. Several properties of starch are altered or improved by HMT, including changes on granule morphology, swelling power, solubility, pasting properties, crystallinity, gelatinization characteristics, digestibility, enzymatic hydrolysis susceptibility, acid hydrolysis sensitivity,

thermal stability, and gel textural properties of starch [73], thus broadening the application of HMT starch in food industry. The main advantages of HMT compared to other starch modification techniques (chemical and biological) include high product safety, flexibility in relation to heat sources, low waste generation, non-generation of chemical residues and non-restriction by food legislation [80-81]. These advantages make it an extremely attractive methodology for industries, especially food.



**Fig. 1.4** Structure of native starch granules and after HMT-modified

HMT is by far the most studied method of physical modification of starch. The properties of HMT modified starch largely depends on processing conditions (i.e., moisture content, temperature, heating time, etc.) and properties starch (i.e., source, composition, morphology, granular size, as well as its amylose content, etc.) [82-83]. Therefore, the effects of these treatments are different for different starch sources and different processing conditions and should be investigated individually. According to previous studies, enhancing HMT modification by increasing the treatment temperature [84], prolonging the heating time [85] or increasing the cycle time [86] can promote the further rearrangement between starch chains, consequently affecting the structure and physicochemical properties of HMT-modified starch. In addition to technological conditions, starches with a higher ratio of amylopectin to amylose are more sensitive to HMT because the steric hindrance near the  $\alpha$ -(1,6)-glycosidic bond is weaker than that near the  $\alpha$ -

(1,4) bond [87]. Besides, the roles of the other factors such as heating source of HMT, operating pressure and cooling process in modification need to be further studied [88].

In HMT-modified starch, the extent of morphological changes is correlated with the HMT process conditions and species of starch [89-90]. Increasing moisture content promote the expansion of starch granules and favors morphological changes [91], while starch granules with higher amylopectin content is more sensitive to HMT, which can be demonstrated by the example that HMT-modified rice starch with different amylose showed different extent morphological changes that higher degree of agglomeration and surface fusion in rice starch containing lower amylose content [92]. The HMT-modified starch granules may undergo morphological changes, and the extent of this changes is closely related to processing conditions (including moisture content, temperature, heating time) and the botanical source of the starch. Increasing moisture content enables starch granules to absorb more water during processing, promotes their expansion and facilitates thermally driven morphological changes [91]. Under polarized light, HMT-treated starch exhibits weaker and fuzzier birefringence cross than that of native starch, and it may even completely disappear when intensity of HMT process conditions is high, which indicates the destruction of ordered structure in the starch granules [93] and changes in the radial orientation of double helices [94]. According to H. Li et al.[95], the observation of birefringence cross is also consistent with the crystallinity results of X-ray diffraction (XRD) measurements, indicating that low- moisture treatment can induce starch chain reassembly to increase the starch order, while high-moisture treatment can lead to disorder of starch crystallization. HMT destroys the regularity and compactness of starch semi- crystalline system, enhances the thickness of crystalline and amorphous lamellae, results in the reduction of crystallinity [91], implying that semi-crystalline lamellae damage may be also a contributor to the reduction of crystallinity [80]. By contrast, the sufficient

mobilization of starch branches during the HMT processing improves the orientation of less stable helical chains, induces the recombination of smaller crystals or facilitates the transformation of amorphous regions into new crystalline states, thus resulting in an increase in crystallinity [88].

In general, HMT-modified starch has higher  $T_o$ ,  $T_p$  and  $T_c$  but lower  $\Delta H$  than that of native counterpart [96-97]. The pasting temperature is the minimum temperature of starch viscosity development, reflecting the structural stability of starch in the early stage of paste formation [98]. As reviewed by Q. Wang [88], the pasting temperature of cereal starch increased more or less after HMT modification, which is consistent with the above-mentioned increase in gelatinization temperature. The elevation of gelatinization temperatures and pasting temperature implies the enhancement of thermal stability. HMT may facilitate the diffusion of starch molecules through intra-/intermolecular bonds and promote the entanglement of amylose and amylopectin, which not only limits the flexibility of the amorphous domain, delays its expansion, but also makes the crystal structure more robust [91]. Moreover, swelling power and solubility of cereal starch such as maize [99], buckwheat [100], oat [101], sorghum [102] and rice starch [103] exhibit a reduction after HMT modification, indicating that HMT can disrupt amylose/amylopectin array within granules. Furthermore, HMT-induced complexes of amylose-lipid can inhibit the expansion of starch, thus reducing amylose leaching and solubility [88]. HMT has been widely reported to alter starch digestibility and enhance the nutritional value of starchy food by increasing slowly digestible starch (SDS) and resistant starch (RS) content [104]. According to Chung et al [105], HMT increased the SDS and RS content in corn, pea, and lentil starches at 100 and 120°C with 30% moisture content. Several studies have also reported that HMT can elevate the content of SDS and/ or RS [13, 91-92, 99].

#### **1.4.2 Microwave treatment**

Microwave treatment (MW) of starch involves the application of electromagnetic waves within the frequency ranging from 300 MHz to 300 GHz, and the corresponding wavelengths ranging from 1 m to 1 mm [106]. MW converts electromagnetic energy into heat energy by triggering high-frequency movement of molecules, and it also has a non-thermal effect that directly affects the reaction molecules [107]. Due to its' uniformly heating, high heating rates and environmental protection, microwave treatment become another appealing physical modification method of starch [108]. Majority of the studies in starch modification using microwaves and other methods to modify starch take advantage of the rapid heating involved during the microwave process, as well as the ability of microwave to promote starch interactions with added chemicals or additives. The mechanism of starch structure changes during MW can be classified into four stages. The first stage involves the phenomenon of dielectric relaxation of water molecules, which is responsible for the initial heating of starch. This is followed by a stage of rapid temperature rise, during which moisture is lost from the starch granule interior. The loss of moisture presumably creates high pressure within the granules and further cause the granule to expand from the center. The last stage involves the obvious degradation of starch granules [22].

The frequency, power, radiation time and geometry of heating system of microwave, and the botanical source, density and dielectric properties of starch affect the effect of microwave on starch [77]. Generally, the higher microwave frequency and moisture content of starch, the greater degree of damage of starch structure caused by microwave treatment. Researches have shown that microwave irradiation of starch leads to changes in granule morphology, molecular structure, structural order and crystallinity, which consequently affects its functional properties, such as solubility, swelling powder, gelatinization, retrogradation and digestion ability [20, 109-111], thus boarding the potential applications of microwaved modified starch in various food and pharmaceutical industry.

The application of single MW in starch modification has been widely studied [18, 20, 65, 112], and in order to obtain better functionality and expand the application of starch in different fields, the combination of MW with other methods has also been widely studied recently [113-116]. The high penetrating power of microwave energy involved in the heating process and the rapid and uniform heating of starch samples make MW seems to be a convenient method to modify starch, although the physicochemical changes observed with MW-modified starches are similar to those modified starches treated by traditional physical modification methods such as HMT and annealing [117]. The heat generated during MW could cause starch to produce free radicals, which can depolymerize large starch molecules into small ones through the cleavage of glycosidic bonds, resulting in structural changes and physicochemical properties changes of starch granules [22]. The MW-modified starch granules may undergo different morphological changes depending on MW processing conditions (including starch moisture content, microwave power, exposure time and the subsequence of MW performed before or after other modification methods) and the botanical source of the starch. Double treatment of MW combined with other heating methods resulted in higher degree of starch fragmentation and agglomeration/fusion of taro starch granules than single MW [113]. The analogous result was reported by L. Wang et al. [116] that double treatment of MW with ultrasound promoted drastic changes with rougher surface and more pits in potato and maize starch granules surface morphology than that of single treatment modified starch granules, and the surface damage of ultrasound-MW- modified samples was more severe than that of the other counterparts. High moisture content of starch seems to be conducive to greater penetration of microwave energy, resulting in severe surface damage of starch granules, collapse in granule structure, aggregation to form larger starch clusters or fusion with the adjacent granules, which can be exemplified by the research of Y. Li et al. [116].

The different influences in crystalline pattern and crystallinity of starch modified by MW can occur depending on the botanical source, moisture content and MW processing conditions. The degree of crystallinity of wheat and normal starches decreased and the waxy corn starch remained almost unchanged after microwave, the crystalline pattern of all starch samples retained the A-type [117], whereas the crystalline pattern of potato starch modified with microwave heating and esterification changed from the B-type polymorph to the C-type [118]. It is reported that MW disrupted the amylopectin,  $\alpha$ -1,6-glycosidic bonds, resulting in a decrease in the number of the outermost unbranched chains [112], and the fracture of intra and intermolecular hydrogen bonds in starch caused a decrease of in the intensity of diffraction peaks and the relative crystallinity [119]. In contrast, starch also can transform to more dense crystalline type after MW under certain conditions. Maize flour (30% moisture content) under microwave irradiation (400 W) with different exposure time (0.5, 1, 2, 4 min) exhibited higher V-type crystalline structure and diffraction intensity, indicating a crystalline growth fostered by MW [120]. The analogous result was reported by M. Villanueva et al [121] that rice flour (20%,30% moisture) under microwave irradiation (900 W) for 4 min and 9 min also displayed higher V-type crystalline structure and diffraction intensity. The occurrence of these results may be attributed to the generation of double helical chain caused by MW through rearrangement of starch crystals, forming more ordered crystal arrays than that in native starch [122].

Similar to HMT-modified starch, MW-modified starch also has higher  $T_o$ ,  $T_p$  and  $T_c$  except  $\Delta H$  than that of native counterpart [112, 117, 122-124], which may be attributed to better hydration of starch amorphous regions after microwave heating [124]. It was reported that MW-modified starch had lower breakdown, trough, final and setback viscosity than that of native starch, and the reduction of these pasting properties was related to microwave heating time, as longer microwave heating time resulted in greater decrease in the pasting properties of

starch [112, 125]. However, the effect of MW on the pasting temperature of starch is not always the same. According to the research result of Colman et al. [125] that MW-modified cassava starch exhibited a slight but significant decrease in the pasting temperature than that of native cassava starch, while other researchers reported that waxy maize starch showed higher pasting temperature than that of native waxy maize starch [112], wheat and corn starches [117]. Most starch exhibit decreased swelling properties and solubility after microwave irradiation. The impacts of MW on starch swelling and solubility are similar to HMT at the condition of relatively low moisture content (<50%) of starch [126]. The solubility and swelling power of wheat and corn starches (30% moisture content) could be reduced after MW (0.5 W/g, 60 min) [117]. The swelling power of MW-modified starch reportedly decreased continuously with the increase of starch moisture content [21]. The reduction of swelling power might be attributed to the enhancement of intra-/intermolecular forces through hydrogen bonding caused by MW, and the inhibition of water molecules binding to free hydroxyl groups of amylose and amylopectin [127]. Moreover, MW-modified starch samples exhibited slow digestion properties, resulting in increased SDS components and decreased starch digestibility [128]. The increase amounts of SDS and RS components in MW-modified starch was not only starch moisture content, microwave power, microwave heating time, but also related to the difference of amylose content and the starch crystal structure [22].

### **1.5 Applications of physically modified starch in food production**

Physical modification methods of starch mainly involve heat-moisture treatment (HMT), annealing, microwave treatment (MW), ultra-high pressure (UHP), ultrasonic, ultraviolet light, radiation and ohmic heating, of which HMT and MW are the most studied physical modification methods of starch from different botanical source. Starch modified by these methods can be used in various field, such as textile, paper, biomedicine, chemicals and food industries,



among others. The structure of starch (arrangement of starch components) can be altered after being modified by physical modification methods to obtain appropriate functional properties for production of specific products. Physically modified starch is preferred in processed food for its appropriate functionalities, safety, non-toxicity, cost efficiency and easy commercialization [129]. According to the forecast report released by Fior Markets company that the global modified starch market is expected to grow from USD 10.13 billion in 2017 to USD 15.53 billion by 2025 at a compound annual growth rate of 5.5% during the forecast period 2018-2025 and the physical modification modified starch had a 45.60% market share in 2017 [130]. Physically modified starches have been widely used in the preparation of flour products, bakery products, beverages, meat products and other food products. Germinated brown rice treated with HMT can partially or completely replace wheat starch to improve nutritional content and higher consumer acceptability of cookies [131]. HMT-modified potato starch with sodium chloride can enhance the hardness of starch dough and the starch noodles exhibited less solid loss and broken noodles, firmer texture, and better elasticity [132]. According to L. Wang [133] annealed rice starch can increase the sensory evaluation scores, cooking qualities and texture properties of rice starch noodles. Waxy maize starch pregelatinized by physical modification methods (extrusion or drum-dryer) can be used in the preparation of jelly confectionery, e.g. soft candy or chewing gum [134]. In addition, pregelatinized waxy maize starch can be used as alternative thickener in low-fat emulsions, replacing non-starch hydrocolloids [135]. MW-modified starch has great potential application in food and pharmaceutical [22]. MW-modified starch exhibited superior properties as hydrophilic matrix excipients for sustained release tablets compared to their unmodified counterpart [136]. The cooking time of noodles can be reduced when MW-modified starch was incorporated into wheat dough or other flour composites [137].

### **Conclusions to section 1**

1. Native starch is widely found in the form of granules in many tissues of most plant species. Starch granules are composed of various structural elements, such as single chains, helices, superhelices, clusters, lamellae, blocklets, growth rings, and granules. As an important raw material used in food industry, the rheological, pasting, and textural properties of starch are the major functional properties to determine its applications.

2. The inherent deficiencies of native starch can be overcome by physical, chemical, and enzyme modification techniques. All these modification methods have the potential to change the structure of starch, thus affecting the functional properties, digestible properties of native starch or delivering new functional properties to modified starches, which ultimately determine the application in food and nonfood industries.

3. Physical modification of starch refers to the use of thermal mechanical force or physical field to destroy or produce changes in the packing arrangements of the starch polysaccharide molecules within granules, thereby ultimately changing the properties and functionality of starch. Heat-moisture treatment and microwave treatment are the most appealing physical modification method of starch due to their advantages of environmental protection, no reagent by-product, easy control process and safe for industrial production.

4. Various modified starches have been used widely in many food and nonfood industries. In the future, novel modification methods or techniques are required to produce starches with more diversified and promising properties for wider industrial applications.

## **SECTION 2 OBJECTS, SUBJECTS AND METHODS OF RESEARCH**

### **2.1 Objects and materials of research**

The object of research in the dissertation was the technologies of modified potato starch and their applications in flour products include cookies, fresh noodles and steamed bread.

The subjects of research were: the properties of modified potato starch obtained by the method of heat-moisture treatment (HMT) processing and the method of microwave treatment (MW) processing, the properties of dough made by incorporation of modified potato starch and the properties of cookies, steamed bread and noodles made by incorporation of modified potato starch. Therefore, the physicochemical properties as well as color characteristics, particle size and water distribution, morphological, the pasting behavior, structural characteristics, and in vitro digestibility properties of modified potato starch were evaluated. Moreover, the potential effects of wheat flour substitution with HMT and MW modified potato starch on the quality characteristics of cookies, fresh noodles and steamed breads were assessed.

The following materials were used as research materials:

- modified potato starches were obtained from the native potato starched modified by heat-moisture treatment and microwave treatment. The native potato starches were obtained from potato tubers of Favorita variety sold locally in Hezhou city (Guangxi, China). Favorita variety potato is widely cultivated in China and characterized by high yield and good edible quality.

- wheat flour (Chen Keming Food Co., LTD, Yiyang city, Hunan province, China).

- low protein flour (Guangzhou Da Fan Zhuo Food Co. Ltd., China).

- white granulated sugar (Nanjing Ganzhiyuan Sugar Co. Ltd., China).

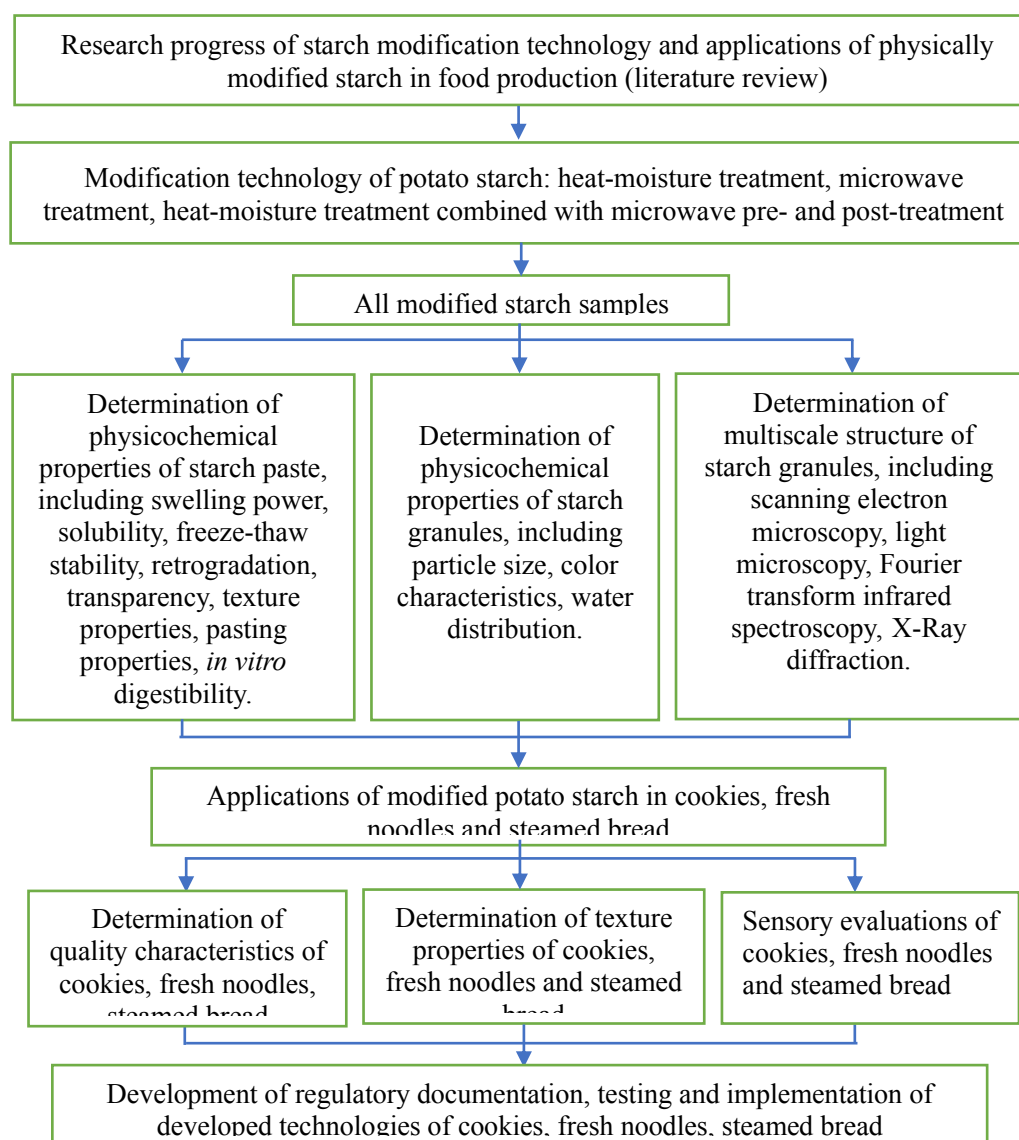
- unsalted butter (Anchor, New Zealand Milk Brand Co. Ltd.).

- pure milk (Yili industrial group Co. Ltd., China).
- active dry yeast (Angel Yeast Co., LTD, Yichang, Hubei province, China).

Raw materials and materials used during experimental research and production tests met the corresponding requirements of applicable regulations.

## 2.2 Staging experiments

Experimental studies were conducted in the laboratories of Hezhou University (China) and the laboratories of Guangxi Key Laboratory of Health Care Food Science and Technology (China). The plan of theoretical and experimental works is given in Fig. 2.1.



**Fig. 2.1** The plan of theoretical and experimental works

## 2.3 Research methods for starch modification

During the research, standard and special organoleptic, physicochemical, structural and mechanism methods were used to determine the properties of modified potato starch, dough and final products.

## 2.4 Laboratory determination methods of starch

### 2.4.1 Determination of swelling power and solubility of starch

Swelling power and solubility of starch samples were determined in triplicate according to the method of [138]. A 0.60 g portion (dry basis) of starch sample was placed into a pre-weighed 50-mL centrifuge tube with 30 mL of distilled water. The starch was completely dispersed in the distilled water by oscillating with a turbine mixer (XW-80A, Haimen Kylin-Bell Lab Instruments Co., Ltd., Haimen, Jiangsu, China) for 5 s. After that, all the centrifuge tubes with starch samples were placed in a water bath oscillator with the speed of 200 rpm for 30 min at 55, 65, 75, 85, and 95°C. The samples were cooled to room temperature before being centrifuged at 2150×g for 20 min with an L550 centrifuge (Hunan Xiangyi Laboratory Instrument Development Co., Ltd., Changsha, Hunan, China). The supernatants were poured into pre-weighed aluminum specimen boxes and dried to a constant weight in a DH411C hot-air oven at 105°C, while sediments were immediately weighed. The swelling power (SP, g/g, on dry weight basis) and solubility (S, %) were calculated as follows:

$$\text{Solubility } (S) = \frac{A}{W} \times 100 \quad (2.1),$$

$$\text{Swelling power } (SP) = \frac{P}{W(1-S/100)} \quad (2.2),$$

where  $A$  is the weight of dried supernatant,  $W$  is the weight of dried sample, and  $P$  is the weight of sediment.

### 2.4.2 Determination of starch paste freeze-thaw stability

The freeze-thaw stability of potato starch gels was investigated by putting samples through alternative freezing and thawing cycles (freezing for 24 h at  $-18^{\circ}\text{C}$  and thawing for 2 h at  $30^{\circ}\text{C}$ ) according to the method of [139] with a slight modification. Potato starch suspension (5%, w/w potato starch on a dry basis) were prepared by blending starch in ultrapure water and then the suspensions were gelatinized by placing in a water bath at  $95^{\circ}\text{C}$  for 30 min with continuously stirring. The samples were held for 5 min before being poured in were pre-weight centrifuge tubes (25 mL), respectively. The weight of centrifuge tubes was recorded, and then the gel samples were frozen in refrigerator at  $-18^{\circ}\text{C}$  for 24h and then thawed in  $30^{\circ}\text{C}$  for 2 h. This was one FT cycle and the FT cycle was repeated for five cycles. After being centrifuged at 3000 rpm for 20 min, the resulting supernatant in the tubes were weighted and recorded. The syneresis rate was calculated as the percentage of supernatant weight on gelatinized gel weight.

#### **2.4.3 Determination of starch paste retrogradation**

Potato starch suspensions (1%, w/w potato starch on a dry basis) were prepared by blending starch in ultrapure water and then the suspensions were gelatinized by placing in a boiling water bath for 40 min with continuously stirring. After the gelatinization was completed, took out the suspensions and cooled to room temperature, poured it into 25 mL tube and kept still in  $25^{\circ}\text{C}$  incubator. The volumes of the starch paste supernatant were recorded every 2 h (total 16h). The percentage of starch paste supernatant liquid volume in the total volume of starch paste changed over time to characterize its retrogradation property.

#### **2.4.4 Determination of starch paste transparency**

The transparency of potato starch was measured using the method described by [140] with modifications. Briefly, potato starch suspensions (1%, w/w potato starch on a dry basis) were prepared by blending starch in ultrapure water and then the suspensions were gelatinized by placing in a water bath at  $90^{\circ}\text{C}$  for 1 h. After gelatinization was completed, took out the suspensions and cooled to room temperature. The transparency of the starch was measured at a wavelength of 640

nm and ultrapure water was used as a blank. The experiment was performed in parallel three times. After the first measurement, the starch-water suspension was stored at 4°C for 120 h, and then measured every 24 h.

#### **2.4.5 Determination of texture profile analysis (TPA) of starch paste**

The potato starch gels were investigated according to the previous study with slight modification [141]. Potato starch suspensions (12%, w/w potato starch on a dry basis) were prepared by blending starch in ultrapure water and then the suspensions were gelatinized by placing in a boiling water bath for 20 min with continuously stirring.

After the gelatinization was completed, potato starch gels were taken out and cooled to room temperature, the samples were then placed in the refrigerator (4°C) and frozen for 24 h. And then the selected starch gels were removed from the container, the size of the container was 55 mm diameter and 20 mm height. Before the measurement, the samples were equilibrated at room temperature for 1 h, and the tests were performed by using texture analyzer (TA.XT PLUS, Stable Micro Systems, UK) with a plate probe with a diameter of 100 mm. The pre-test speed and post-test speed were set at 1.0 mm/s, the test speed was set at 2.00 mm/s, while the strain was fixed at 50% with a trigger force of 5 g. All the textural parameters were measured and calculated by the instrument software from the resulting force-deformation curves, including hardness (g), adhesiveness, springiness, cohesiveness, gumminess, chewiness and resilience. The TPA measurement was carried out parallel three times.

#### **2.4.6 Determination of starch pasting properties**

A rapid visco-analyzer (RVA Starch Master2, Perten Instruments, Stockholm, Sweden) was used to evaluate the pasting and paste properties of the starch according to the methods described by [142] with slight modifications. A 2.5-g portion of each starch (corrected to moisture content of 14 g/100 g) was mixed with 25 mL of distilled water and kept in the test slot of the equipment. The temperature gradient was as follows: equalization at 50°C for 1 min, increased

from 50°C to 95°C within 3.75 min and maintaining at 95°C for 2.5 min, and then decreased to 50°C in 3.75 min and maintaining at 50°C for 2 min.

#### **2.4.7 Particle size determination of starch granules**

The particle size parameters of starch samples were detected by a laser diffraction particle size analyzer (BT-2001, Baxter Instruments Co. LTD, China) with dry method. Air was used as medium and the optical mode was Mie. Starch sample (3 g) was added to the storage hopper with a shading ratio ranging from 5%–12%. The starch particle size parameters included particle diameter of volume (D(4,3)), particle diameter of surface (D(3,2)), specific surface area (S.S.A.), and D50 (median particle diameter) represents the corresponding particle size which is smaller than 50% of the sample particles.

#### **2.4.8 Determination of color characteristics**

Color parameters ( $L^*$ ,  $a^*$ ,  $b^*$ ) of starch granules were determined at least three times by using a colorimeter (CR-400, Konica Minolta Inc., Japan.) after the calibration of the equipment with a standard-white reflection plate.  $L^*$  indicates lightness, which varies from black ( $L^* = 0$ ) to white ( $L^* = 100$ ),  $a^*$  is greenness / redness value, which varies from green ( $-60$ ) to red ( $+60$ ) and  $b^*$  is blueness / yellowness value, which varies from blue ( $-60$ ) to yellow ( $+60$ ). Color difference ( $\Delta E$ ) between experimental samples and the control samples was calculated with the equation:

$$\Delta E = \sqrt{(L^* - L_0^*)^2 + (a^* - a_0^*)^2 + (b^* - b_0^*)^2} \quad (2.3),$$

where  $L^*$ ,  $L_0^*$  is the lightness value of experimental sample and control sample;  $a^*$ ,  $a_0^*$  is the greenness/ redness value of experimental sample and control sample;  $b^*$ ,  $b_0^*$  is the blueness/yellowness value of experimental sample and control sample. The smaller value of  $\Delta E$  indicates the smaller color difference between experimental sample and control sample. A  $\Delta E$  value  $>3$  was used to indicate whether the color differences between two different samples could be visibly differentiated[143-144] .



#### **2.4.9 LF-NMR spin– spin relaxation (T2) measurements of starch granules**

Low-field nuclear magnetic resonance (LF-NMR) spin-spin relaxation measurements were carried out using a Niumag Benchtop Pulsed NMR Analyzer (NMI120X, Niumag Electric Corp., Shanghai, China) to determine the water distribution of native and treated starch. A saturated NaCl<sub>2</sub> solution was used to equilibrate the water content of the samples for two weeks at 25°C until the water activity of the samples ( $a_w = 0.724$ ) was constant and consistent. According to the method of [145], approximately 1 g of equilibrated starch was placed into the NMR tube with diameter of 15 mm to measure T2 using the Carr-Purcell-Meiboom-Gill (CPMG) sequence. The testing temperature was  $32 \pm 0.1^\circ\text{C}$  and the proton resonance frequency was 18.0 MHz. Typical pulse parameters were as follows: the time of 90° pulse (P1) was 9.5  $\mu\text{s}$  and the time of 180° pulse (P2) was 19.04  $\mu\text{s}$ , the waiting time (TW) between subsequent scans was 3500 ms, data from 5000 echoes were acquired as 4 scan repetitions, and each measurement was performed at least 3 times.

#### **2.4.10 Methods of scanning electron microscopy**

The morphology of starch granule was observed by scanning electron microscope (SU8100, Hitachi Ltd., Tokyo, Japan or JSM-7610F, JEOL, Japan) as [146] with slight modification. The starch samples were sprinkled on a double-sided adhesive tape mounted on an aluminum stub and coated with gold with for 30 s by using an EDT-2000 ion sputter ( $2 \times 10^{-4}$  MPa, 25 mA). Subsequently, all the coated samples were examined at an acceleration voltage of 2.0 kV and the images were captured at  $\times 1000$  and  $\times 1500$  magnification.

#### **2.4.11 Methods of light microscopy**

A small amount of each starch sample was placed on a microscope slide with 1–2 drops of glycerol with a distilled water mixture (1:1, v/v) to disperse the starch evenly, then the starch was covered with coverslip and placed on the objective table of the microscope (BX53, Olympus Corporation, Tokyo, Japan). The normal light microscopic images and polarized light microscopic images of starch

samples were observed and captured under normal light mode and polarized light mode, respectively, with the magnification of  $\times 400$ .

#### **2.4.12 Fourier transform infrared spectroscopy (FT-IR) analysis**

The starch samples were placed directly on to sampling unit of an FT-IR spectrophotometer (Spectrum, Perkin Elmer, Waltham, MA, USA) to determine the FT-IR spectra with a scanning spectral range from  $4000\text{ cm}^{-1}$  to  $400\text{ cm}^{-1}$  at  $25^{\circ}\text{C}$ .

#### **2.4.13 X-Ray diffraction analysis**

X-Ray diffraction (XRD) analysis of starch samples was carried out by an X-ray diffractometer (Rigaku Ultima IV, Ultima IVTM, Tokyo, Japan) equipped with a goniometer at 40 kV (target voltage) and 40 mA (tube current). The measurement diffraction angle ( $2\theta$ ) ranged from  $4^{\circ}$  to  $40^{\circ}$  at a scanning rate of  $4^{\circ}/\text{min}$  with a step size of  $0.02^{\circ}$ . MDI Jade 6 software was used to calculate the relative crystallinity (%) of each starch sample according to the following equation:

$$\text{Relative crystallinity} = \frac{Ac}{Aa} \times 100 \quad (2.4),$$

where  $Ac$  is area of crystalline peaks,  $Aa$  is total area of crystalline and amorphous peaks.

#### **2.4.14 *In vitro* digestibility analysis of starch**

The contents of rapidly digested starch (RDS, starch which was digested within the first 20 min), slowly digested starch (SDS, starch which was digested between 20 and 120 min) and resistant starch (RS, the residual starch which was digested after 120 min) in native potato starch and treated starch were determined according to the method previously described by Han et al [7] with some modifications. In brief, 200 mg of starch samples with 10 mL of a sodium acetate buffer solution (0.1 M, pH 5.2) were added to a flask and heated in boiling water for 30 min to completely gelatinize starch. Afterwards, tubes were cooled in a water bath at  $37^{\circ}\text{C}$  and incubated for 30 min with 160 rpm shaking. Then, the

enzyme solution of 5 mL of  $\alpha$ -amylase from porcine pancreas (300 U/mL, Shanghai Yuanye Bio-Technology Co., Ltd, Shanghai, China) and 2 mL of amyloglucosidase from *Aspergillus niger* (225 U/mL, Shanghai Yuanye Bio-Technology Co., Ltd) were added to each sample tube, and incubation was continued in a water bath at 37°C with 190 rpm shaking. Then, 1 mL of the digestion solution was pipetted into a test tube with 20 mL of anhydrous alcohol to stop the enzyme reaction at intervals of 20 and 120 min, and then centrifuged at 2810×g for 10 min in an L550 centrifuge. The released glucose concentration in the supernatant was measured with the glucose oxidase/peroxidase (GOPOD) assay kit (Megazyme, International Ltd. Co., Wicklow, Ireland). The glucose content multiplied by a factor of 0.9 was used to calculate the percentage of hydrolyzed starch and the following formulas were used to calculate the contents of RDS (%), SDS (%), and RS (%):

$$RDS = G20 \times \frac{0.9}{TS} \quad (2.5),$$

$$SDS = (G120 - G20) \times \frac{0.9}{TS} \quad (2.6),$$

$$RS = 100 - RDS - SDS \quad (2.7),$$

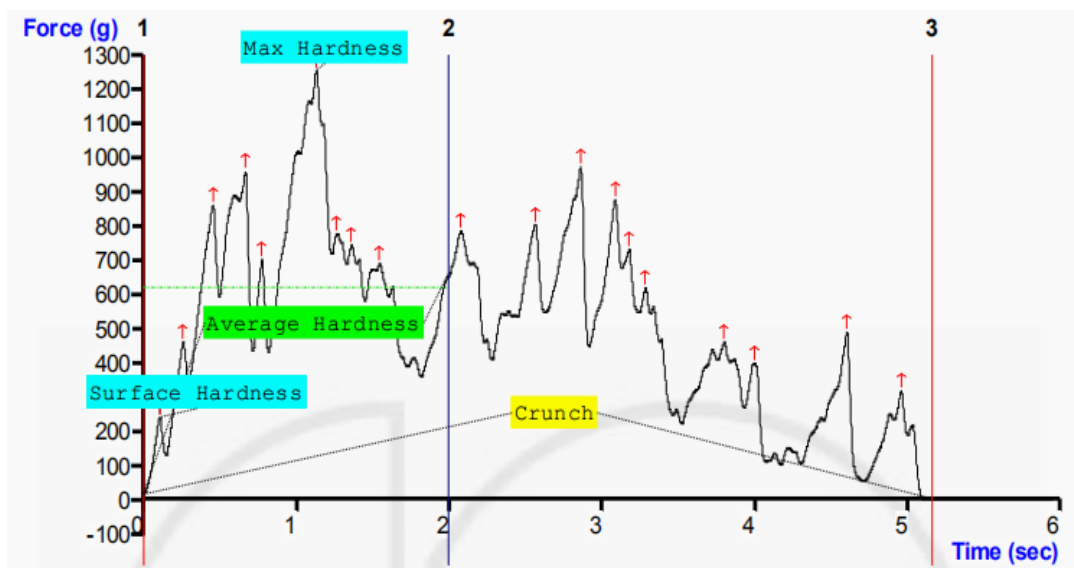
where:  $G20$  and  $G120$  are the glucose contents after 20 min and 120 min of hydrolysis, respectively;  $TS$  is the total starch content of each sample.

## 2.5 Quality determination of cookies, fresh noodles and steamed bread

### 2.5.1 Determination of texture properties of cookies

Cookies texture was determined by texture analyzer (TA.XT PLUS, Stable Micro Systems, UK) with P2 probe on the center of every cookie. The pre-test speed and the test speed were set at 1.00 mm/s, post-test speed was set at 2.0 mm/s, while the distance was fixed at 5 mm with a trigger force of 20 g. All the textural parameters were measured and calculated by the instrument software from the resulting force-deformation curves. As presented in Fig. 2.2, the average force

value between 0 and 2 seconds on the curve reflected the average hardness of samples, the first peak force value reflected the surface hardness of samples, the maximum force value on the curve reflected the max hardness of samples, the number of peaks  $> 100$  g on the curve reflected the crunch value of samples and the number of peaks greater than 10 g and less than 100 g in the curve reflected the crispy value of samples. The TPA measurement was carried out parallel at least three times.



**Fig. 2.2** Graphical analysis of cookie texture curve

### **2.5.2 Determination of texture and tensile properties of noodle dough**

The dough was uniformly molded into cylindrical shape with diameter of 25 mm and height of 34 mm by a mold, and then was wrapped in with plastic wrap and rested at room temperature for 15 min before the determination of texture properties. The texture properties were determined by using a TA-XT plus texture analyzer (Exponent stable microsystem, version 6.1.2.0, Stable Microsystems Ltd., UK) equipped with P/50 probe, and the parameters were set as follows: the pre-test speed and post-test speed were set at 1.00 mm/s, the test speed was set at 1.00 mm/s, while the deformation level was 75% with a trigger force of 5 g. Hardness, adhesiveness, springiness, cohesiveness, chewiness and resilience were measured to evaluate dough texture properties.

The dough was covered with plastic wrap and rested at room temperature for

15 min, and then was put into the special dough strip preparation tank of the texture analyzer A/KIE probe to prepare uniformly size dough strips for tensile test. The parameters were set as follows: the pre-test speed and post-test speed were set at 2.00 mm/s, the test speed was set at 5.00 mm/s with a trigger force of 5 g. The tensile properties of the dough were evaluated by the resistance to extension and extensibility.

### **2.5.3 Determination of texture and tensile properties of fresh noodles**

The texture and tensile properties of fresh noodles were determined as the methods reported by Zhang et al. [147] with slightly modifications. The TPA compressive texture properties and TPA tensile properties were determined within 15 min after cooking using texture analyzer (TA.XT PLUS, Stable Micro Systems, UK) equipped with P/36R probe, A/SPR, respectively. The fresh noodles with same length of 20 cm and same diameter of 2.5 mm were cooked in boiling water until the optimal cooking time was reached, then quickly removed and cooled to room temperature in cold water and drained for 5 min before the measurement. the noodles were sheared same length with 4 cm. 3 sticks noodles were placed on the test bench side by side for determination for each time. As for TPA compressive test, the determination parameters of texture properties were set as follows: the pre-test speed and post-test speed were set at 2.00 mm/s, the test speed was set at 1.00 mm/s, while the deformation level was 75% with a trigger force of 5 g. Hardness, springiness, cohesiveness, chewiness and resilience were measured to evaluate noodle texture quality.

As for the TPA tensile test, 1 stick of cooked noodle was tied at one end of the lower arm groove of the probe and tightened. The other end of the noodle was tied to the upper arm groove with the same procedure.

The determination parameters of tensile properties were set as follows: the pre-test speed and test speed were set at 2.0 mm/s, the pre-test speed was set at 10.0 mm/s, while the distance between the two arms was set at 30 mm. The tensile properties of the noodles were evaluated by the maximum breaking force obtained.

#### **2.5.4 Determination of texture analysis of steamed bread**

Steamed bread texture was determined according to the methods of Hsieh *et al.* [148] with slight modifications. The crumb of steamed bread was cut into cubes with the size of 2×2×2 cm and assayed using texture analyzer (TA.XT PLUS, Stable Micro Systems, UK) equipped with P/36R probe. The pre-test speed and post-test speed were set at 2.0 mm/s, the test speed was set at 1.00 mm/s, while the deformation level was 50% with a trigger force of 5 g. Hardness, springiness, cohesiveness, gumminess, chewiness and resilience were calculated by the instrument software.

#### **2.5.5 Determination of quality characteristics of cookies, fresh noodles, steamed bread**

##### **(1) Determination of color**

Color parameters ( $L^*$ ,  $a^*$ ,  $b^*$ ) of cookies, steamed bread were determined at least three times by using a colorimeter (CR-400, Konica Minolta Inc., Japan.) after the calibration of the equipment with a standard-white reflection plate according to the method of 2.4.8 determination of color characteristics. The center rather than the edge of every cookie was used as the test point for color determination. Both color steamed bread crumb and steamed bread crust were used as the test point for color determination respectively.

##### **(2) Determination of specific volume of steamed bread**

The specific volume of steamed bread was determined by millet displacement method according to [149]. Cooled steamed bread was weighed, and the volume was determined through millet replacement method, and the specific volume (mL/g) of steamed bread was calculated by the ratio of volume to the weight of steamed bread.

##### **(3) Determination of cooking properties of fresh noodles**

The optimal cooking time was determined by the methods of Niu *et al.* [150] with some modifications. 20 sticks fresh noodles with the length of 20 cm were cooked in 500 mL of boiling water and cooked till the central opaque core in

noodles disappeared, as judged by slightly squeezing the noodles between two transparent glass slides.

The dry matter water absorption rate of noodles was determined according to the methods of Mu *et al.* [151] with slightly modifications. The moisture content of fresh noodles was measured firstly, and the total quality ( $M_2$ , g) of 20 sticks fresh noodles with the length of 20 cm was recorded and then cooked in 500 mL of boiling water until the optimal cooking time was reached. The noodles were quickly removed from the cooking water and cooled in distill water, then placed on filter papers to absorb the surface water of the noodles. The total mass of cooked noodles was recorded ( $M_1$ , g). The dry matter water absorption rate was calculated as follows:

$$\text{The dry matter water absorption rate(\%)} = \frac{M_1 - M_2 \times (1 - W)}{M_2 \times (1 - W)} \times 100 \quad (2.8),$$

where  $M_1$  was the mass of cooked noodles (g);  $M_2$  was the mass of fresh noodles (g);  $W$  was the moisture content of fresh noodles (%).

The loss rate of dry matter was determined according to the method of Lin *et al.*[152] with slightly modifications. The noodles soup obtained from the determination of the dry matter water absorption rate was reserved for the determination of the loss rate of dry matter. It was transferred into weighing dish and dried in an oven at 105 °C to constant weight. The mass weight ( $M_3$ ) after constant weight was recorded. The loss rate of dry matter was calculated as follows:

$$\text{The loss rate of dry matter} = \frac{M_3}{M_1(1 - W)} \times 100 \quad (2.9),$$

The cooking breakage rate of noodles was determined by the method of Zhang [153]. 20 sticks fresh noodles with the length of 20 cm was cooked in 500 mL of boiling water until the optimal cooking time was reached. Calculated the percentage of broken noodles, which was the cooking breakage rate of noodles.

#### **2.5.6 Sensory evaluations of cookies, fresh noodles and steamed bread**

The panelists (7 males and 8 females, ages of 20-35) of sensory evaluation

of cookies, fresh noodles and steamed breads were postgraduate students and the staff of the Department of Food Science and Technology. The sensory evaluation methods were as follows:

### **(1) Sensory evaluation of cookies**

The sensory evaluation was done according to method of Cervini et al.[143]. The test was carried out by 9-point hedonic scale for color, texture and over all acceptability of these cookies. For all sensory attributes, a score of 5 was considered as the limit of acceptability.

### **(2) Sensory evaluations of fresh noodles**

The experimental method was based on the scoring method for sensory evaluation of Chinese white salted noodles reported by Liu et al [154], with some modifications as indicated in Table 2.1. For all sensory attributes, total scores of 80 was considered as the limit of acceptability.

**Table 2.1**

Scoring method for sensory evaluation of fresh noodles

Parameters	Score	Evaluation rules
Color	10	Creamy white/pale yellow (8.0-10); white (6.0-7.9); gray or dark (0-6.9)
Appearance shape	10	Smooth (8.0-10); less smooth but good shape (6.0-7.9); little distorted (4.0-5.9); very coarse or misshapen (0-3.9)
Oral chewiness	20	Good chewiness, medium hard (17.0-20); slightly hard or slightly soft (12.0-16.9); poor chewability, very hard or very soft (0-11.9)
Elasticity	25	Very elastic (21.0-25); good elastic (18.0-20.9); medium elastic (12.0-17.9); low elastic (6.0-11.9); no elastic (0-5.9)
Stickiness	25	No stickiness (21.0-25); very slightly sticky (18.0-20.9); medium sticky (12.0-17.9); sticky (6.0-11.9); very sticky (0-5.9)
Smoothness	5	Good smooth (3.0-5.0) ; medium smoothness (2.0-2.9) ; no smoothness or coarse (0-1.9)
Flavor	5	Good flavor (4.0-5.0) ; medium flavor (2.0-2.9) unpleasant abnormal smell (0-1.9)

### **(3) Sensory evaluations of steamed bread**

The experimental method was based on the scoring method for sensory



evaluation of Chinese northern style steamed bread reported by Cheng *et al* [155], with some modifications as indicated in Table 2.2. For all sensory attributes, total scores of 80 was considered as the limit of acceptability.

**Table 2.2**

Scoring method for sensory evaluation of steamed bread

Parameters	Score	Evaluation rules
Specific volume	20	The highest score is 20 points for 2.3 mL/g, the score decreased by 1 point for each decrease of 0.1 mL/g.
Color	10	Creamy white/pale yellow (7.1-10); dark yellow (4.1-7.0), gray or dark (0-4.0)
Appearance shape	20	Good volume and symmetry, good upright, very smooth, bright, no specks (16.1-20.0); basic symmetry and upright, fewer rough surfaces, fewer specks or bubbles on the surface (10.1-16.0); no symmetry, low height, and bad shape, rough surface, specks or bubbles on surface (0-10)
Internal texture	10	Good crumb structure dense homogenous and spongy (no big holes) (7.1-10); acceptable crumb, homogenous with few big holes (4.1-4.0), poor, uneven large holes, and not homogenous (0-4.0)
Flavor	10	Good fragrance with fermented aroma (7.1-10); moderate aroma (4.1-7.0); unpleasant abnormal smell (0-4.0)
Oral chewiness	10	Good chewiness, soft and easy to swallow (7.1-10); moderate hardness and chewiness (4.1-7.0); poor chewability, hard and difficult to swallow (0-4.0)
Elasticity	20	Good bounce back, and can be pressed to 1/2 volume (16.1-20.0); bounce back slowly, and can be pressed to 1/4 volume (10.1-16.0); poor no bounce and crumbly (0-10)

### 2.5.6 Postprandial blood glucose measurement of participants.

The postprandial blood glucose level (mmol/L) in participants was measured using a rapid blood glucose meter according to Health Industry Standard of the People's Republic of China— standard for determination of food glycemic index (WS/T 652—2019). All the participants (15 males and 15 females, ages of 20-45) of postprandial blood glucose measurement of cookies, fresh noodles and steamed breads were healthy. At 8:00 a.m. on the day of experiment, all the participants were instructed to eat 50 g the test food (cookies, fresh noodles and steamed breads) after measuring the fasting blood glucose levels (fasting for more than 10

h), and then their blood glucose levels were measured at 15th, 30th, 45th, 60th, 90th, and 120th min after eating. Each measurement was performed 3 times.

## **2.6 Statistical analysis**

All the measurements were performed three times unless otherwise stated, and the data were recorded as means  $\pm$  standard deviation (SD). One-way analysis of variance (one-way ANOVA) with post-hoc Duncan's test was performed by Data Processing System (7.05 for Windows, Hangzhou Ruifeng Info-technology Co., Hangzhou city, Zhejiang province, China). Differences were considered significant at  $P_{\text{value}} < 0.05$ . Microsoft Office Excel 365 (Microsoft Corp., Redmond, WA, USA) and Origin Pro 8 (OriginLab Corp., Northampton, MA, USA ) were used to report the data.

### **Conclusions to section 2**

1. The subject, materials and additives in research are given. The plan of theoretical and experimental works is made.

2. Research methods for starch modification, laboratory determination methods of starch and applications and influence of modified potato starch in cookies, fresh noodles and steamed bread are introduced in detail.

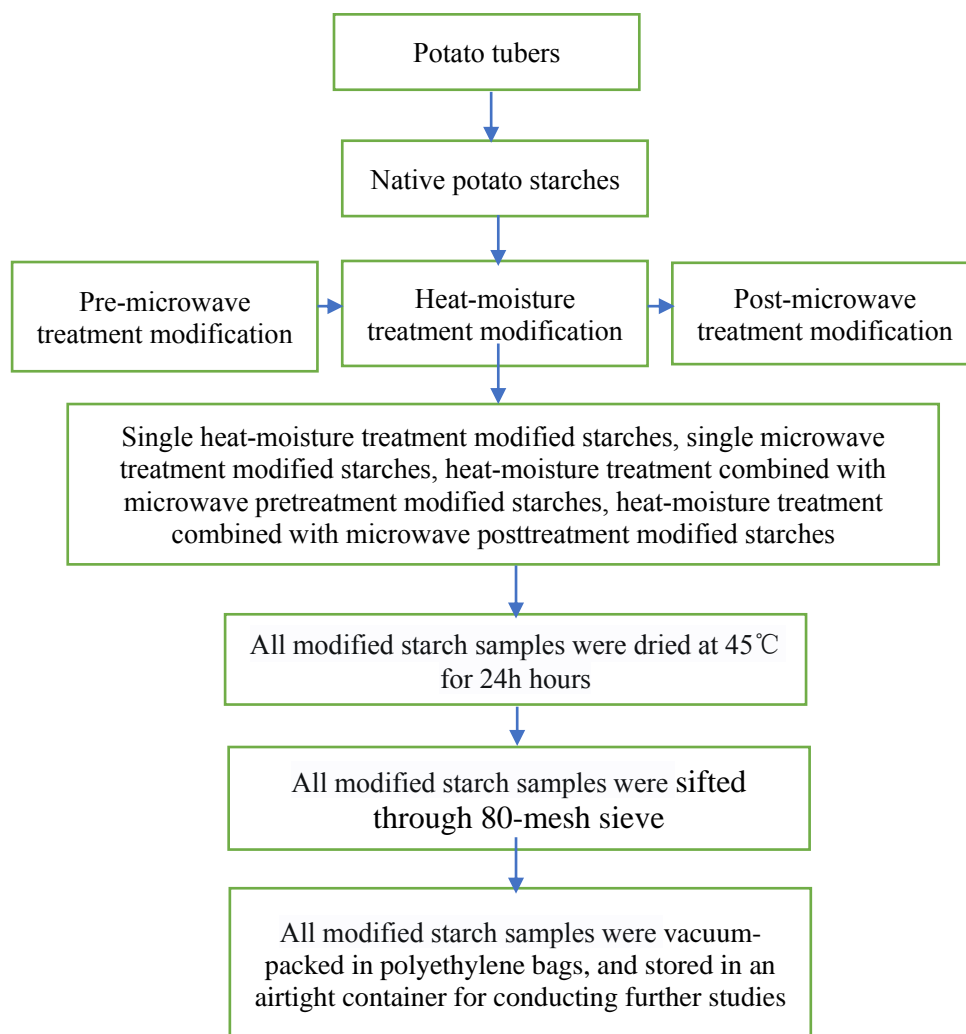
### **SECTION 3 EFFECTS OF HEAT-MOISTURE TREATMENT CONDITIONS ON PHYSICOCHEMICAL PROPERTIES, STRUCTURAL PROPERTIES OF POTATO STARCH AND ITS PROCESS OPTIMIZATION**

Potato is not only the fourth largest crop in world production after rice, wheat and corn, among the most important crops for feeding the global, but also is among the most important crops for its feeding the global population [156]. Potatoes are widely planted in China, and in 2015, China harvested nearly 95 million tons potatoes [157]. Majority of potatoes are processed into starch, flour, flakes and other products, while minority are consumed as fresh eating. Potato starch is mainly used as thickener, colloidal stabilizer, gelling agent, adhesive in food industry, and also used as bulking agent, water-retention agent [158]. With the advantages of being renewable, nontoxic, biodegradable and of relatively low cost, starch-based derivatives are nowadays used for many applications in food processing in order to achieve particular technological properties [159]. Heat-moisture treatment (HMT) is by far the most studied method of physical modification of starch. Due to the reaction conditions included the starch source, moisture content, heating temperature, heating time and other process parameters are diverse, it is difficult to define the properties of HMT starches in definitive way. It is also difficult to define the application of HMT modified starch on the effects of improving the quality of final food products. Previous research had confirmed that setback viscosity of starch had significantly correlation with the quality of food products such as vermicelli and noodles [160].

Therefore, this research was carried out to evaluate the effects of starch moisture levels, heating time and heating temperature of HMT on physicochemical, morphological, the pasting behavior, structural characteristics, and *in vitro* digestibility properties of potato starch. Moreover, in order to obtain HMT modified starch with specified properties, the response surface

methodology (RSM) was used to optimize the HMT processing parameters with setback viscosity as an index. Different HMT modified starches were prepared and named according to the methods of 2.3.1 heat moisture treatment modified potato starch preparation, and the results of these studies are presented in this section.

The mechanism of dual modification combined with HMT and MW is far from being fully understood. Therefore, we also prepared different HMT and MW bi-directional modified starches and investigated evaluate the effects of HMT assisted by MW pre- and post-treatment on the morphological, physicochemical and *in vitro* digestion properties of potato starch. Therefore, the technology of potato starch modification for research refers to the use of heat-moisture treatment and microwave treatment as showed in Fig. 3.1.



**Fig. 3.1** Technological scheme for obtaining modified potato starch

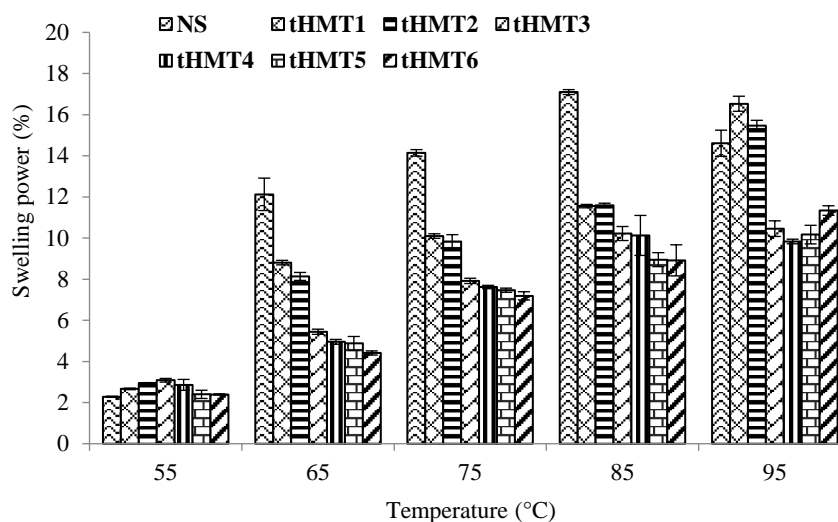
### 3.1 Preparation of heat-moisture treatment modified starch

Potato starch was subjected to HMT in accordance with the method described by Huan Li et al. with some modifications [95]. (1) Different heating time groups (tHMT): Starches were weighted in different spiral blue-moth bottles, and ultrapure water were added to adjust moisture content to 25% and equilibrated for 24 hours at 25°C. Samples were then heated in electric thermostatic drying oven at 110°C for 1 h, 2 h, 3 h, 4 h, 5 h and 6 h ( tHMT1, tHMT2, tHMT3, tHMT4, tHMT5, tHMT6, respectively ); (2) Different heating temperature groups (THMT) : Starches were weighted in different spiral blue-moth bottles, and ultrapure water were added to adjust moisture content to 25% and equilibrated for 24 h at 25°C. Samples were then heated in electric thermostatic drying oven at 90°C, 100°C, 110°C, 120°C, 130°C (THMT90, THMT100, THMT110, THMT120, THMT130, respectively ) for 2 h. (3) Different moisture content of starch system groups (CHMT): Potato starch samples were weighted in different spiral blue-moth bottles, and different volumes of ultrapure water were added to adjust moisture content levels to 15%, 20%, 25%, 30%, 35% (CHMT15, CHMT20, CHMT25, CHMT30, CHMT35, respectively) and equilibrated for 24 h at 25°C. Samples were then heated in electric thermostatic drying oven (DH411C, Yamato, Tokyo, Japan) at 110°C for 2 h. After modification, all these HMT modified potato starch samples were removed from the oven and cooled naturally to room temperature, and then all the treated starch samples were dried at 45°C for 24 h using the same hot-air oven, so that the starch moisture was less than 12 g/100 g. The dried modified potato starches were intermittently pulverized for 50 s (pulverized for 5 s, stopped for 5 s to avoid too high temperature of starch caused by pulverization) using an FW100 pulverizer (Tianjin City Taisite Instrument Co., Ltd.). The pulverized starches were sieved through an 80-mesh sieve, then hermetically packaged in polyethylene bags, and stored in a glass desiccator for conducting further studies.

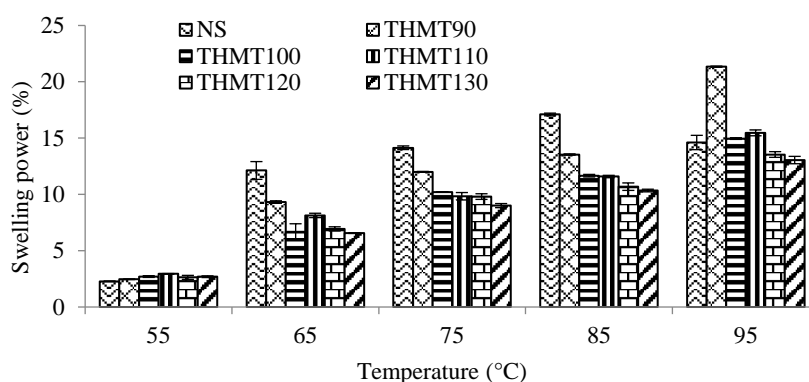
### **3.2 Effects of heat-moisture treatment conditions on the physicochemical properties of potato starch**

#### **3.2.1 Effects of heat moisture treatment conditions on swelling power and solubility of potato starch**

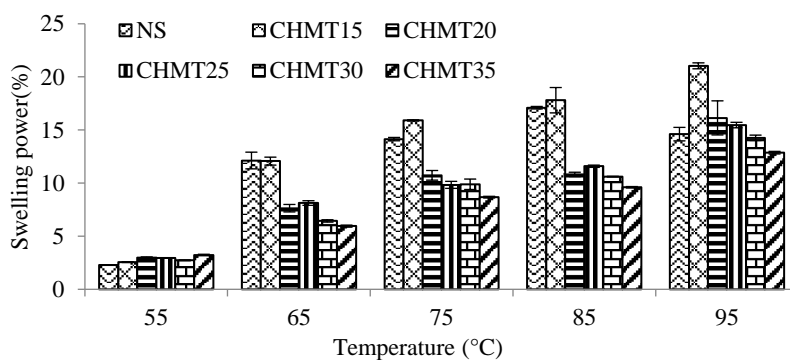
The swelling power of HMT starches and native potato starch (NS) at 55°C - 95°C were shown in Fig. 3.2, Fig. 3.3 and Fig. 3.4. The swelling power of NS and HMT starches showed an increasing trend with increase of test temperature. The swelling power of tHMT and THMT were significantly reduced ( $P_{\text{value}} < 0.05$ ) in comparison with that of NS in the test temperature of 65°C - 85°C. The swelling power of HMT starches were increased in comparison with that of NS in the test temperature of 55°C, which indicated that HMT increased the swelling power of potato starch in low temperature. At the same test temperature (65°C - 95°C), the swelling power of HMT starches decreased with the increasing heating time, heating temperature and moisture content, and these results were consistent with the result of a pervious stay focusing on mung bean starch [161] and corn starch [99]. The reduction in swelling power of HMT starches could account for the rearrangement of starch molecule or / and re-associations of starch chains caused by HMT [162]. With high moisture content, HMT destroyed the crystal structure because of water molecules could enter the crystallization zone of starch granules and the water molecules were likely to interact with starch molecules in the destroyed crystals, resulting in a relatively high swelling power.



**Fig. 3.2** Effect of heat-moisture treatment on swelling power of potato starches under different heating time



**Fig. 3.3** Effect of heat-moisture treatment on swelling power of potato starches under different heating temperature

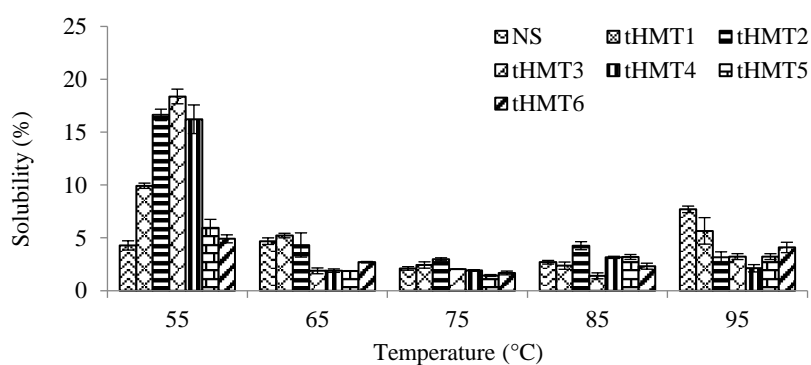


**Fig. 3.4** Effect of heat-moisture treatment on swelling power of potato starches under different moisture content

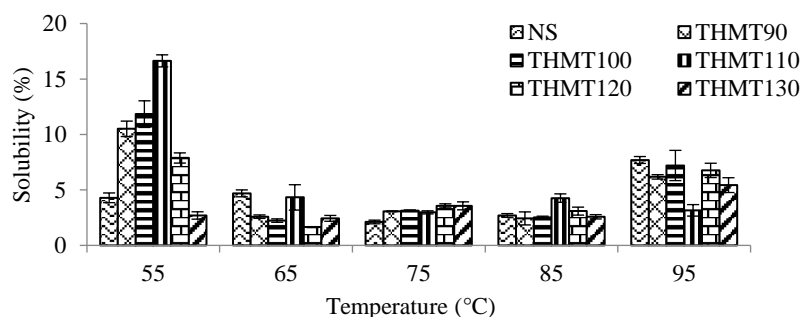
The solubility HMT starches and native potato starch (NS) at 55°C - 95°C were shown in Fig. 3.5, Fig. 3.6 and Fig. 3.7. The solubility of NS and HMT starches showed an increasing trend with an increase of test temperature (65°C - 95°C), but lower than that of low temperature (55°C). HMT starches showed



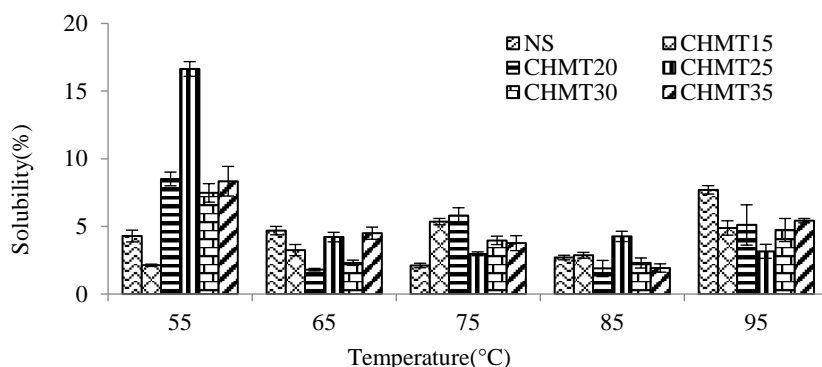
higher solubility in comparison of NS in low test temperature (55°C -75°C), which indicated that HMT destroyed the structure of starch granules, preventing amylose and lipids to form complex at low temperature, thereby increased the solubility. Amylose in starch granules was hard to leach out after HMT, and formed a stable structure with amylopectin in starch molecules [101], thereby the solubility of HMT starch was lower than that of NS, which was consistent with the results of previous researches on the modification of corn starch [99]. In general, HMT decreased starch swelling power and solubility, which were suitable in noodles processing.



**Fig. 3.5** Effect of heat-moisture treatment on solubility of potato starches under different heating time



**Fig. 3.6** Effect of heat-moisture treatment on solubility of potato starches under different heating temperature



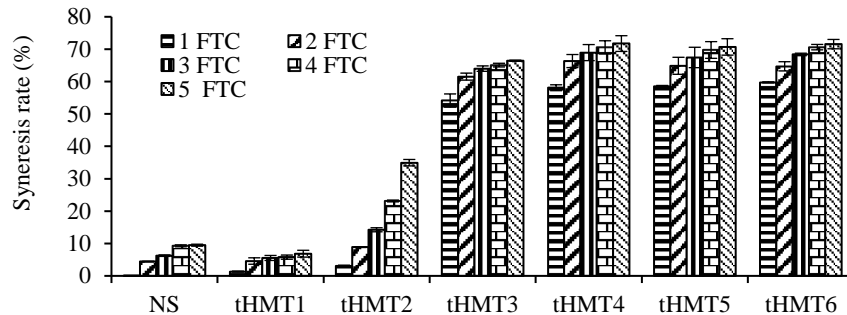
**Fig. 3.7** Effect of heat-moisture treatment on solubility of potato starches under different moisture content

### 3.2.2 Effect of heat moisture treatment on freeze-thaw stability of potato starch

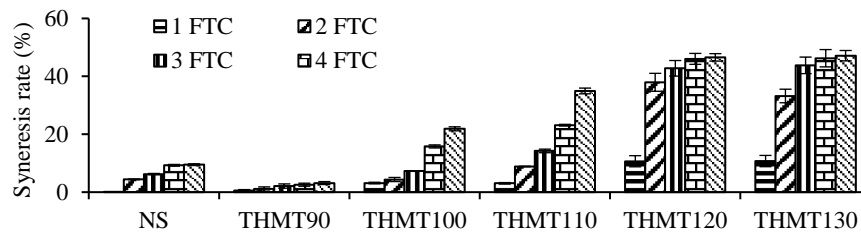
Freeze-thaw stability represents the ability of starch withstand the undesirable physical changes that may occur during freezing and thawing. Thermal energy fluctuation and phase change of water during freeze-thaw are the possible cause of disruption of the gel matrix of starch. When a starch gel is frozen, starch-rich regions are created in the matrix, where water remains partially unfrozen. High solid concentration in the regions facilitates the starch chains to associate forming thick filaments, whereas water molecules coagulate into ice crystals forming a separate phase. Upon thawing, ice transforms to bulk phase water, which can be readily released from the polymeric network (syneresis). The water release consequently leaves the starch gel sponge-like [163].

The freeze-thaw stabilities during FT cycles (1FTC, 2FTC, 3FTC, 4FTC, 5FTC) were shown in Fig. 3.8, Fig. 3.9 and Fig. 3.10. with the percentage of syneresis as an index. The syneresis of all the starch gels including NS increased with the increasing of freezing-thawing cycles. Compared with NS, tHMT1, THMT 90 and CHMT15 starch gels had lower syneresis, which indicated that HMT could improve the freeze-thaw stability of starch gels under such conditions. With further increase of treatment heating time, treatment heating temperature and moisture content, destruction of starch granules increased significantly, starch molecules were rearranged, which limited the ability of starch molecules and

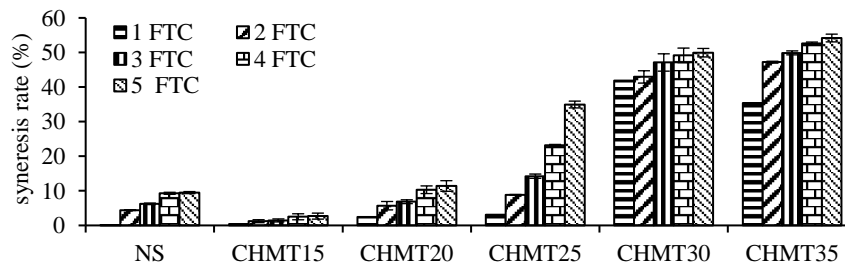
water molecules to bind to each other through hydrogen bonds, thereby the water in starch granules was more easily precipitated, resulting in poor freeze-thaw stability of starch gels.



**Fig. 3.8** Effect of heat-moisture treatment on freeze-thaw stability of potato starches under different heating time



**Fig. 3.9** Effect of heat-moisture treatment on freeze-thaw stability of potato starches under different heating temperature



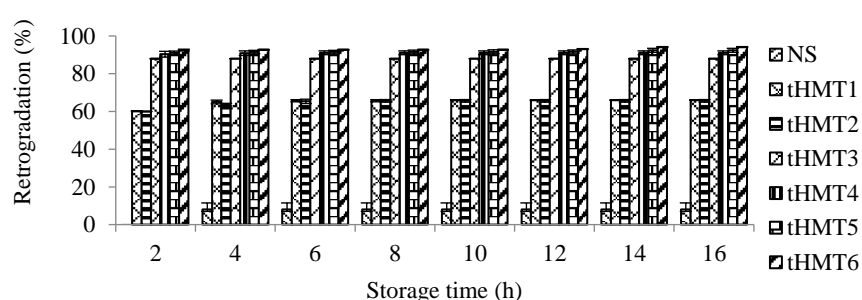
**Fig. 3.10** Effect of heat-moisture treatment on freeze-thaw stability of potato starches under different starch moisture content

### 3.2.3 Effect of heat moisture treatment on retrogradation of potato starch

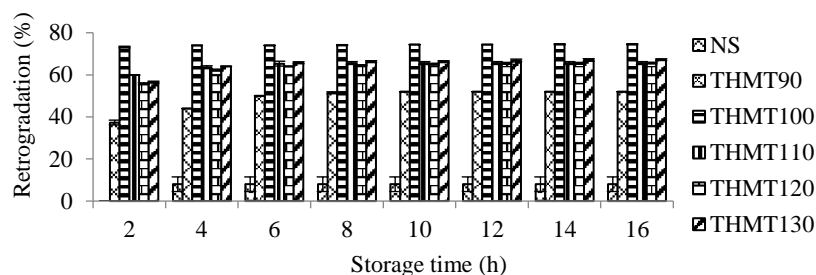
The retrogradation of starch is the process of gelatinized starch molecules from disordered state to orderly rearrangement, finally coagulation and sedimentation. In the gelatinization process of starch by heating, the ordered starch molecules become disordered under the action of water and heat. In the process of cooling and storage, due to the effect of molecular potential energy, the

disorder of high energy states gradually tends to the order of low energy states. NS and HMT starches were gelatinized and stored at 25°C for 16 h and the starch paste supernatant liquid volumes were recorded every 2 h. The retrogradation of HMT starches and native potato starch (NS) were shown in Fig. 3.11, Fig. 3.12 and Fig. 3.13.

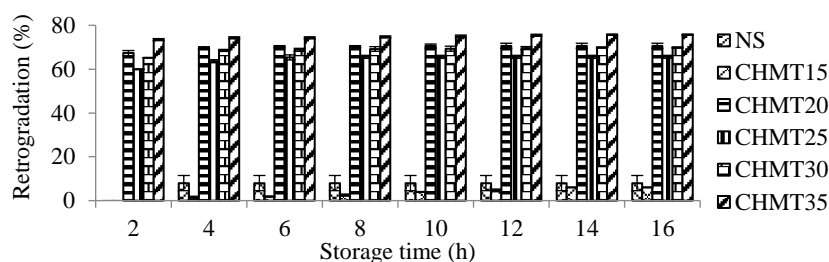
Fig. 3.11, Fig. 3.12 and Fig. 3.13 showed that retrogradation of NS and HMT starches increased and tended to balance with the extension of storage time. HMT destroyed the structure of starch granules and reduced the hydrogen bonds between starch molecules and water molecules, which was prone to retrogradation, resulting in great retrogradation of potato starch. Starch retrogradation was generally considered to have adverse effects on starchy food because it shortens the shelf life of food, reduces the consumer acceptance and sensory evaluation. However, HMT starch can be used to produce vermicelli and other food for its property of easy retrogradation. To certain extent, retrogradation of starch is also nutritionally important because retrograded starch is slowly digested by human digestive enzymes in the upper gut, which can alleviate the release of glucose into the blood stream, resulting in reduced postprandial glycemic and insulin responses [164].



**Fig. 3.11** Effect of heat-moisture treatment on retrogradation of potato starches under different heating time



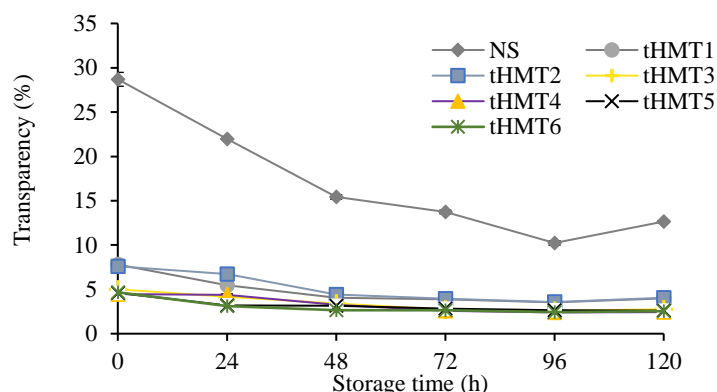
**Fig. 3.12** Effect of heat-moisture treatment on retrogradation of potato starches under different heating temperature



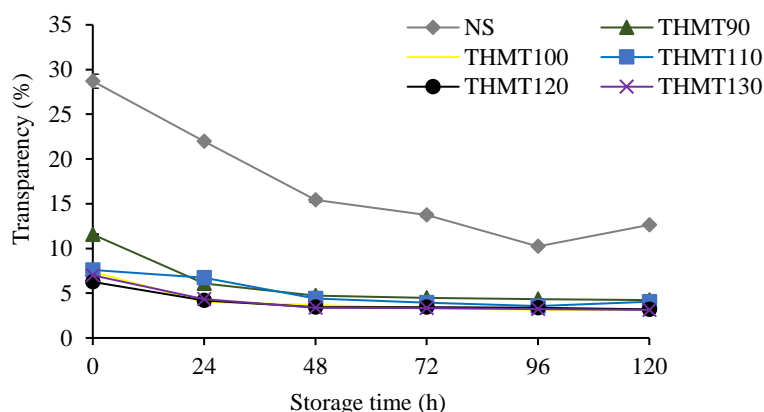
**Fig. 3.13** Effect of heat-moisture treatment on retrogradation of potato starches under different moisture content

### 3.2.4 Effect of heat moisture treatment on transparency of potato starch

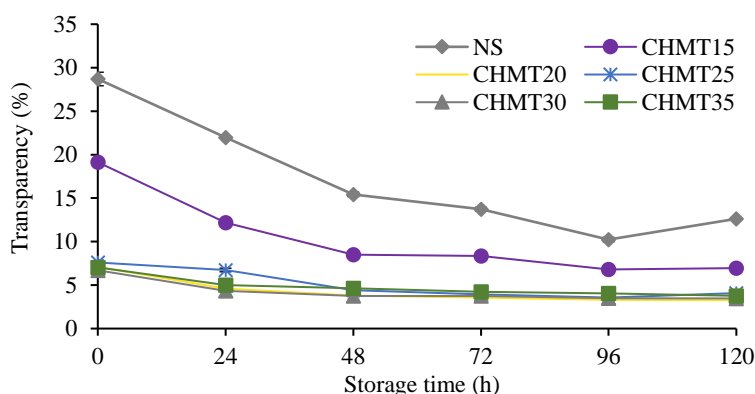
The transparency of starch paste is an important indicator for product characteristics, which is closely related to the appearance and acceptability of starch and its processed products, also reflects the ability of starch molecules to bond with water molecules [165]. As shown in Fig. 3.14, Fig. 3.15 and Fig. 3.16, the starch pastes retrograded during storage which affected the transparency, thereby, the transparency of NS and HMT starches decreased with the extension of storage time. With increase of treatment holding time, treatment holding temperature and moisture content, the transparency of starch pastes decreased significantly. HMT destructed starch granules, which led to rearrangement of starch molecules and destruction of crystal structure. The number of unexpanded starch granules and incompletely broken remaining starch granules increased during the heating gelatinization process, which caused the light scattering when it passed through the starch paste, which reduced the transparency of the starch pastes [166].



**Fig. 3.14** Effect of heat-moisture treatment on transparency of potato starches under different heating time



**Fig. 3.15** Effect of heat-moisture treatment on transparency of potato starches under different heating temperature

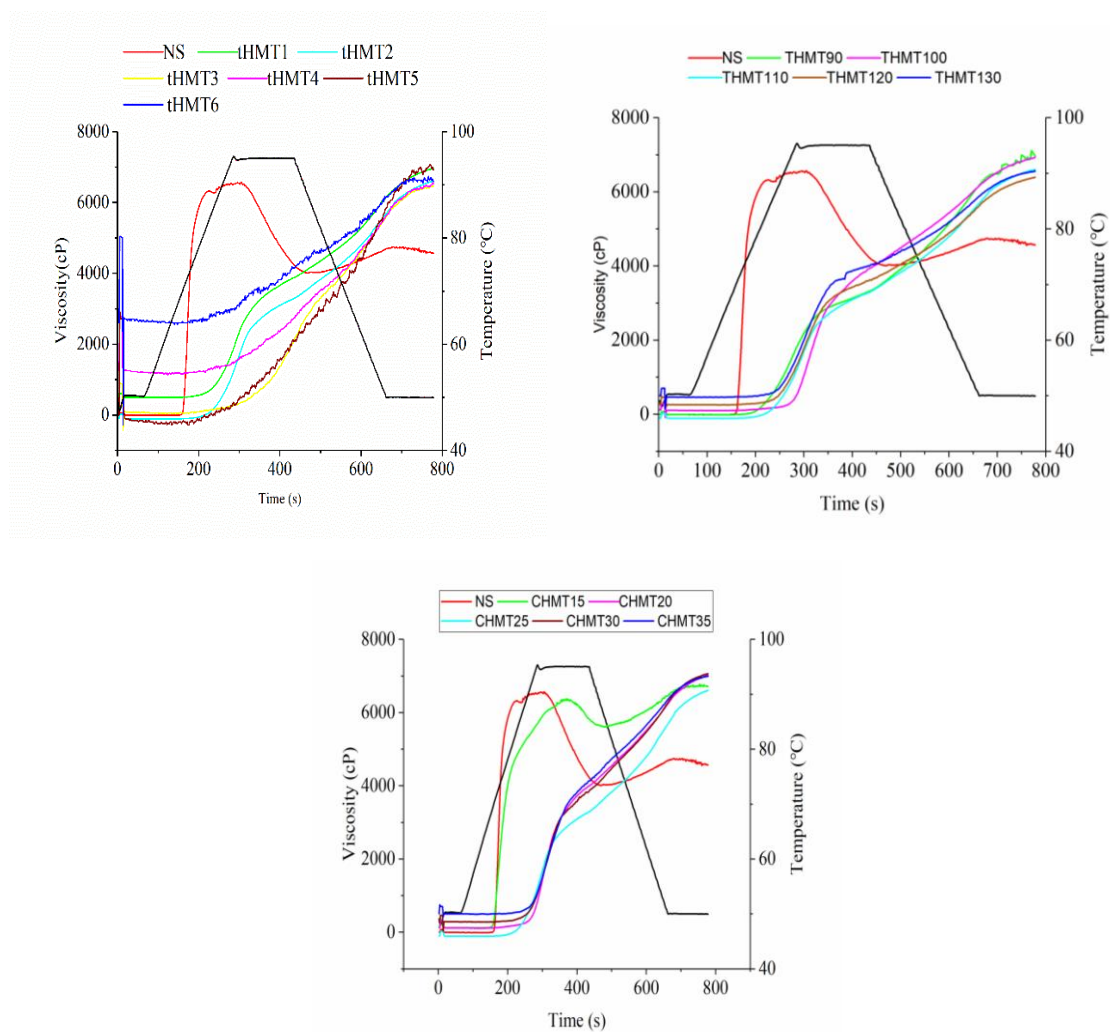


**Fig. 3.16** Effect of heat-moisture treatment on transparency of potato starches under different moisture content

### 3.2.5 Effect of heat moisture treatment on pasting properties of potato starch

The starch viscosity properties are correlations to the gel texture properties, the stability of starch paste and retrogradation tendency. The RVA profiles of NS and HMT starch were shown as Fig.3.17 and the gelatinization parameter values were shown in Table 3.1, Table 3.2 and Table 3.3. As shown in Fig.3.17, the RVA profiles of all HMT samples had great difference with NS starch, the RVA

gelatinization properties of HMT starches were significantly different from each other. Compared with native potato starch, the peak viscosity, hold viscosity and breakdown viscosity of HMT starch decreased with the increasing of moisture content, treatment temperature and time, while the gelatinization temperature increased (Table 3.1, Table 3.2 and Table 3.3), and the final viscosity was higher than peak viscosity, which indicated that potato starch modified by heat-moisture treatment was more prone to retrogradation. The decrease of peak viscosity is supposedly attributed to the strengthening of starch interior interactions by heat moisture treatment enhancing granule integrity, preventing penetrate of water into the starch granules. The breakdown viscosity is related to the thermal stability of the swollen starch granules in the starch paste during heating and shearing, i.e. lower breakdown indicates more resistance to shear force [167-168]. Final viscosity and setback value are represented to the re-association during cooling of the main amylose that released following. The lower viscosity and higher gelatinization temperature indicated that the structure of starch particles was more stable and the internal force between molecules was stronger after heat moisture treatment. Therefore, the starch modified by heat moisture treatment requires more energy to decompose its structure and form paste, thus improving the gelatinization stability of the sample. The setback viscosity firstly increased and then decreased with the extension of treatment time, temperature and moisture content. The setback viscosity has great relativity to the retrogradation properties of starch and has great influence on the tensile strength of vermicelli food [160]. Therefore, it is necessary to select appropriate heat-moisture treatment conditions for the preparation of vermicelli food by using the HMT potato starch in order to obtain better edible quality.



**Fig. 3.17** RVA profiles of native and HMT potato starches

**Table 3.1**

Effect of heat-moisture treatment on rheological properties and relative crystallinity of potato starch under different holding time

Samples	Pasting temperature (°C)	Peak viscosity (cP)	Hold viscosity (cP)	Final viscosity (cP)	Breakdown (cP)	Setback (cP)	Relative crystallinity (%)
NS	68.5±0.2 <sup>d</sup>	6598.0±72.8 <sup>a</sup>	4071.7±25.4 <sup>a</sup>	4567±37 <sup>a</sup>	2526.33±47.34 <sup>a</sup>	495.7±11.6 <sup>c</sup>	19.37
tHMT1	76.1±1.3 <sup>c</sup>	2189.0±63.2 <sup>b</sup>	2145.0±60.9 <sup>b</sup>	4234±71 <sup>b</sup>	44.00±2.65 <sup>b</sup>	2089.0±25.1 <sup>a</sup>	20.08
tHMT2	76.7±1.6 <sup>c</sup>	1888.0±14.0 <sup>c</sup>	1850.0±10.2 <sup>c</sup>	3736±6 <sup>c</sup>	38.00±4.36 <sup>bc</sup>	1886.3±3.5 <sup>b</sup>	23.32
tHMT3	95.0±0.1 <sup>a</sup>	173.7±4.5 <sup>d</sup>	148.7±4.0 <sup>e</sup>	573±10 <sup>d</sup>	25.00±1.00 <sup>bc</sup>	424.3±5.7 <sup>d</sup>	24.43
tHMT4	97.0±0.0 <sup>a</sup>	62.6±4.9 <sup>e</sup>	232.3±22.1 <sup>d</sup>	232±22 <sup>e</sup>	6.67±0.58 <sup>c</sup>	176.3±17.5 <sup>e</sup>	22.83
tHMT5	95.1±0.1 <sup>a</sup>	47.3±5.5 <sup>e</sup>	42.7±3.2 <sup>f</sup>	146±34 <sup>f</sup>	4.67±2.89 <sup>c</sup>	103.0±31.2 <sup>f</sup>	20.98
tHMT6	90.9±4.0 <sup>b</sup>	42.3±1.5 <sup>e</sup>	38.7±0.6 <sup>f</sup>	109±2 <sup>f</sup>	3.67±1.15 <sup>c</sup>	70.7±2.3 <sup>g</sup>	20.75

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).



**Table 3.2**

Effect of heat-moisture treatment on rheological properties and relative crystallinity of potato starch under different holding temperature

Samples	Pasting temperature (°C)	Peak viscosity (cP)	Hold viscosity (cP)	Final viscosity (cP)	Breakdown (cP)	Setback (cP)	Relative crystallinity (%)
NS	68.5±0.2 <sup>d</sup>	6598±73 <sup>a</sup>	4072±25 <sup>a</sup>	4567±37 <sup>b</sup>	2526.3±47.3 <sup>a</sup>	496±12 <sup>f</sup>	19.37
THMT90	72.7±2.0 <sup>c</sup>	3367±56 <sup>b</sup>	3299±53 <sup>b</sup>	6997±81 <sup>a</sup>	67.3±3.1 <sup>b</sup>	3697±71 <sup>a</sup>	21.30
THMT100	67.7±3.0 <sup>d</sup>	1662±17 <sup>d</sup>	1617±15 <sup>d</sup>	3024±13 <sup>d</sup>	44.7±2.1 <sup>bc</sup>	1406±13 <sup>c</sup>	21.43
THMT110	76.7±1.6 <sup>b</sup>	1888±14 <sup>c</sup>	1850±10 <sup>c</sup>	3736±9 <sup>c</sup>	38.0±4.4 <sup>bc</sup>	1886±4 <sup>b</sup>	23.32
THMT120	75.0±1.0 <sup>bc</sup>	1282±35 <sup>c</sup>	1258±38 <sup>c</sup>	2365±30 <sup>c</sup>	23.7±5.5 <sup>c</sup>	1107±12 <sup>d</sup>	25.77
THMT130	81.2±1.61 <sup>a</sup>	1165±25 <sup>f</sup>	1147±24 <sup>f</sup>	2002±59 <sup>f</sup>	18.0±1.7 <sup>c</sup>	855±36 <sup>e</sup>	25.74

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

**Table 3.3**

Effect of heat-moisture treatment on rheological properties and relative crystallinity of potato starch under different moisture content

Samples	Pasting temperature (°C)	Peak viscosity (cP)	Hold viscosity (cP)	Final viscosity (cP)	Breakdown (cP)	Setback (cP)	Relative crystallinity (%)
NS	68.5±0.2 <sup>c</sup>	6598±73 <sup>a</sup>	4072±25 <sup>b</sup>	4567±37 <sup>b</sup>	2526.3±47.3 <sup>a</sup>	496±12 <sup>c</sup>	19.37
CHMT15	67.8±0.3 <sup>c</sup>	5594±31 <sup>b</sup>	4920±49 <sup>a</sup>	5930±73 <sup>a</sup>	674.7±41.9 <sup>b</sup>	1010±30 <sup>d</sup>	21.46
CHMT20	66.3±3.9 <sup>c</sup>	1720±13 <sup>d</sup>	1685±14 <sup>d</sup>	3102±34 <sup>d</sup>	35.0±2.0 <sup>c</sup>	1416±26 <sup>b</sup>	21.71
CHMT25	76.7±1.6 <sup>b</sup>	1888±14 <sup>c</sup>	1850±10 <sup>c</sup>	3736±9 <sup>c</sup>	38.0±4.4 <sup>c</sup>	1886±4 <sup>a</sup>	23.32
CHMT30	83.8±0.2 <sup>a</sup>	1378±5 <sup>c</sup>	1346±8 <sup>c</sup>	2627±52 <sup>c</sup>	34.0±2.6 <sup>c</sup>	1275±36 <sup>c</sup>	24.19
CHMT35	85.3±1.7 <sup>a</sup>	1217±10 <sup>f</sup>	1180±14 <sup>f</sup>	2190±27 <sup>f</sup>	37.0±5.3 <sup>c</sup>	1010±14 <sup>d</sup>	25.09

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

### 3.2.6 Effect of heat moisture treatment on texture properties of potato starch paste

Starch gelatinization refers to the process in which the crystalline structure of the starch-water suspension melts at a certain temperature, and the starch granules are highly swollen or even broken and gelatinized. This is an irreversible process from an ordered structure to a disordered structure. This process is accompanied by water swelling of starch granules, precipitation of amylose, water

association and crystal loss, and finally a network structure gel is formed, which is closely related to the quality characteristics of food.

Textural properties of the native potato starch (NS) gel and HMT starch (THMT, tHMT, CHMT) gels, including hardness, springiness, cohesiveness, gumminess, chewiness and resilience, were investigated as the determination of texture profile analysis (TPA) of starch paste. And the results were shown as Table 3.4, Table 3.5 and Table 3.6.

**Table 3.4**

Textural properties of native potato starch (NS) and tHMT starches

Starch gel	Hardness (g)	Springiness (mm)	Cohesiveness (-)	Gumminess (g)	Chewiness (g·mm)	Resilience (-)
NS	2705.55±3.11 <sup>c</sup>	0.83±0.01 <sup>a</sup>	0.63±0.01 <sup>a</sup>	1700.43±11.44 <sup>c</sup>	1403.77±29.88 <sup>c</sup>	0.40±0.03 <sup>a</sup>
tHMT1	5528.36±3.03 <sup>b</sup>	0.65±0.02 <sup>b</sup>	0.59±0.04 <sup>a</sup>	3286.67±224.62 <sup>b</sup>	2137.88±95.10 <sup>b</sup>	0.41±0.03 <sup>a</sup>
tHMT2	7037.943±1.69 <sup>a</sup>	0.69±0.00 <sup>b</sup>	0.54±0.01 <sup>b</sup>	3765.31±70.58 <sup>a</sup>	2577.18±29.67 <sup>a</sup>	0.34±0.03 <sup>b</sup>
tHMT3	2448.32±6.65 <sup>e</sup>	0.57±0.04 <sup>c</sup>	0.30±0.00 <sup>c</sup>	738.17±7.20 <sup>d</sup>	420.26±22.52 <sup>d</sup>	0.084±0.01 <sup>c</sup>
tHMT4	2639.09±38.55 <sup>d</sup>	0.54±0.03 <sup>c</sup>	0.28±0.00 <sup>c</sup>	750.92±24.03 <sup>d</sup>	403.55±32.02 <sup>d</sup>	0.08±0.01 <sup>c</sup>
tHMT5	2239.92±44.15 <sup>f</sup>	0.49±0.01 <sup>d</sup>	0.24±0.02 <sup>d</sup>	537.14±33.75 <sup>d</sup>	263.57±11.25 <sup>e</sup>	0.08±0.01 <sup>c</sup>
tHMT6	1534.46±12.27 <sup>e</sup>	0.41±0.04 <sup>e</sup>	0.20±0.00 <sup>d</sup>	309.20±3.56 <sup>e</sup>	126.16±2.76 <sup>f</sup>	0.07±0.01 <sup>c</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

**Table 3.5**

Textural properties of native potato starch (NS) and THMT starches

Starch gel	Hardness (g)	Springiness (mm)	Cohesiveness (-)	Gumminess (g)	Chewiness (g·mm)	Resilience (-)
NS	2705.55±3.11 <sup>c</sup>	0.83±0.00 <sup>c</sup>	0.63±0.01 <sup>b</sup>	1700.43±11.44 <sup>d</sup>	1403.77±29.88 <sup>d</sup>	0.40±0.03 <sup>b</sup>
THMT90	10203.52±3.09 <sup>a</sup>	0.93±0.00 <sup>a</sup>	0.82±0.01 <sup>a</sup>	8382.18±105.69 <sup>a</sup>	7782.81±92.20 <sup>a</sup>	0.59±0.04 <sup>a</sup>
THMT100	8308.08±102.65 <sup>b</sup>	0.84±0.01 <sup>b</sup>	0.55±0.01 <sup>c</sup>	4585.48±37.33 <sup>b</sup>	3856.21±14.00 <sup>b</sup>	0.38±0.01 <sup>b</sup>
THMT110	7037.943±1.69 <sup>c</sup>	0.68±0.00 <sup>c</sup>	0.54±0.01 <sup>c</sup>	3765.31±70.58 <sup>c</sup>	2577.18±29.67 <sup>c</sup>	0.34±0.03 <sup>b</sup>
THMT120	2955.38±13.97 <sup>d</sup>	0.72±0.00 <sup>d</sup>	0.40±0.00 <sup>d</sup>	1189.55±11.89 <sup>e</sup>	858.24±4.37 <sup>e</sup>	0.23±0.03 <sup>c</sup>
THMT130	1230.64±2.45 <sup>f</sup>	0.54±0.00 <sup>f</sup>	0.27±0.01 <sup>c</sup>	327.36±9.35 <sup>f</sup>	178.43±6.48 <sup>f</sup>	0.11±0.00 <sup>d</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

**Table 3.6**

Textural properties of native potato starch (NS) and CHMT starches

Starch gel	Hardness (g)	Springiness (mm)	Cohesiveness (-)	Gumminess (g)	Chewiness (g·mm)	Resilience (-)
NS	2705.55±3.11 <sup>f</sup>	0.83±0.01 <sup>a</sup>	0.63±0.00 <sup>b</sup>	1700.43±11.44 <sup>d</sup>	1403.77±29.88 <sup>c</sup>	0.40±0.03 <sup>b</sup>
CHMT15	7349.3460.48 <sup>b</sup>	0.83±0.00 <sup>a</sup>	0.87±0.01 <sup>a</sup>	6383.39±172.05 <sup>a</sup>	5266.54±160.00 <sup>a</sup>	0.61±0.04 <sup>a</sup>
CHMT20	7560.91±14.97 <sup>a</sup>	0.74±0.00 <sup>b</sup>	0.44±0.03 <sup>d</sup>	3307.93±33.28 <sup>c</sup>	2449.48±17.63 <sup>b</sup>	0.28±0.02 <sup>cd</sup>
CHMT25	7037.94±1.69 <sup>c</sup>	0.68±0.00 <sup>c</sup>	0.54±0.01 <sup>c</sup>	3765.31±70.58 <sup>b</sup>	2577.178±29.67 <sup>b</sup>	0.34±0.03 <sup>bc</sup>
CHMT30	3380.40±147.33 <sup>e</sup>	0.58±0.01 <sup>d</sup>	0.33±0.03 <sup>f</sup>	1071.25±46.45 <sup>e</sup>	620.51±14.04 <sup>e</sup>	0.13±0.01 <sup>e</sup>
CHMT35	3774.98±16.10 <sup>d</sup>	0.66±0.02 <sup>c</sup>	0.40±0.01 <sup>c</sup>	1506.15±25.61 <sup>d</sup>	995.78±42.49 <sup>d</sup>	0.22±0.01 <sup>d</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

Table 3.4, Table 3.5 and Table 3.6 showed that the hardness, gumminess, chewiness and resilience of HMT starch gels increased significantly and then decreased with the extension of treatment time. Short heating time (<1.5 h), relatively low heating temperature (<100°C) and low moisture content (<25%) of HMT can significantly enhance the texture properties of HMT starch gels. With the increase of treatment time, temperature and moisture content, the textural property parameters of HMT starch gels decreased gradually. The texture properties of starch gels were related to the type of starch, the structure and proportion of amylose and amylopectin [169]. HMT caused the granular structure of potato starch to be damaged to varying degrees, and the dissolution of amylose were inhibited under HMT. Meanwhile, HMT caused the molecular structure of starch to change and starch gelatinization to occur. Proper heat moisture treatment of starch is beneficial to improve the texture properties of starch gel, which is conducive to the application of HMT modified starch in noodles. Previous studies had found an improvement in the texture (adhesiveness, chewiness, and tensile strength) of the noodles prepared with HMT modified rice starch or sweet potato starch [170].

### 3.2.7 Effect of heat moisture treatment on particle size distributions of potato starch granules

Table 3.7 to Table 3.19 showed that the D50, D(4,3) and D (3,2) of HMT starch were significantly higher than that of NS except the 50 value and D(4,3) of tHMT6, whereas the S.S.A. value of HMT was significantly lower than NS. The large particle size of HMT starch indicated that heat moisture treatment could result in slight swell of potato starch, which might further lead to the rupture, adhesion and partial gelatinization and agglomeration of the granules. This result was agreement with a previous study about lily starch [95].

**Table 3.7**

Effect of heat-moisture treatment on particle size distribution of potato starches under different holding time

Samples	D50( $\mu\text{m}$ )	D(4,3) ( $\mu\text{m}$ )	D(3,2) ( $\mu\text{m}$ )	S.S.A( $\text{m}^2/\text{kg}$ )
NS	34.12 $\pm$ 0.40 <sup>c</sup>	36.60 $\pm$ 0.38 <sup>c</sup>	22.24 $\pm$ 0.56 <sup>d</sup>	99.93 $\pm$ 2.54 <sup>a</sup>
tHMT1	35.33 $\pm$ 0.19 <sup>b</sup>	37.83 $\pm$ 0.18 <sup>ab</sup>	24.02 $\pm$ 0.14 <sup>c</sup>	92.50 $\pm$ 0.52 <sup>b</sup>
tHMT2	35.30 $\pm$ 0.14 <sup>b</sup>	37.84 $\pm$ 0.21 <sup>ab</sup>	24.27 $\pm$ 0.06 <sup>c</sup>	91.52 $\pm$ 0.20 <sup>b</sup>
tHMT3	35.23 $\pm$ 0.94 <sup>b</sup>	37.18 $\pm$ 1.35 <sup>bc</sup>	25.90 $\pm$ 1.25 <sup>ab</sup>	85.99 $\pm$ 4.17 <sup>cd</sup>
tHMT4	36.16 $\pm$ 0.122 <sup>a</sup>	38.75 $\pm$ 0.12 <sup>a</sup>	25.00 $\pm$ 0.21 <sup>bc</sup>	88.87 $\pm$ 0.74 <sup>bc</sup>
tHMT5	35.37 $\pm$ 0.28 <sup>b</sup>	37.85 $\pm$ 0.27 <sup>ab</sup>	24.48 $\pm$ 0.19 <sup>c</sup>	91.63 $\pm$ 0.48 <sup>b</sup>
tHMT6	34.73 $\pm$ 0.46 <sup>bc</sup>	36.50 $\pm$ 0.76 <sup>c</sup>	26.51 $\pm$ 0.39 <sup>a</sup>	83.83 $\pm$ 1.23 <sup>d</sup>

**Notes:** all values are the means of triplicate determinations  $\pm$  standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

**Table 3.8**

Effect of heat-moisture treatment on particle size distribution potato starches under different holding temperature

Samples	D50( $\mu\text{m}$ )	D(4,3) ( $\mu\text{m}$ )	D(3,2) ( $\mu\text{m}$ )	S.S.A( $\text{m}^2/\text{kg}$ )
NS	34.12 $\pm$ 0.40 <sup>c</sup>	36.60 $\pm$ 0.38 <sup>c</sup>	22.24 $\pm$ 0.56 <sup>c</sup>	99.93 $\pm$ 2.54 <sup>a</sup>
THMT90	35.59 $\pm$ 0.30 <sup>ab</sup>	38.06 $\pm$ 0.27 <sup>ab</sup>	24.02 $\pm$ 0.67 <sup>b</sup>	92.54 $\pm$ 2.66 <sup>b</sup>
THMT100	35.60 $\pm$ 0.39 <sup>ab</sup>	38.08 $\pm$ 0.36 <sup>ab</sup>	24.30 $\pm$ 0.26 <sup>b</sup>	91.43 $\pm$ 0.97 <sup>b</sup>
THMT110	35.30 $\pm$ 0.14 <sup>b</sup>	37.84 $\pm$ 0.21 <sup>b</sup>	24.27 $\pm$ 0.06 <sup>b</sup>	91.52 $\pm$ 0.20 <sup>b</sup>
THMT120	35.49 $\pm$ 0.35 <sup>ab</sup>	37.86 $\pm$ 0.42 <sup>b</sup>	26.09 $\pm$ 1.13 <sup>a</sup>	85.27 $\pm$ 3.79 <sup>c</sup>
THMT130	35.87 $\pm$ 0.16 <sup>a</sup>	38.46 $\pm$ 0.18 <sup>a</sup>	24.92 $\pm$ 0.19 <sup>b</sup>	89.15 $\pm$ 0.68 <sup>b</sup>

**Notes:** all values are the means of triplicate determinations  $\pm$  standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

**Table 3.9**

Effect of heat-moisture treatment on particle size distribution of potato starches  
under different moisture content

Samples	D50( $\mu\text{m}$ )	D(4,3) ( $\mu\text{m}$ )	D(3,2) ( $\mu\text{m}$ )	S.S.A( $\text{m}^2/\text{kg}$ )
NS	34.12 $\pm$ 0.40 <sup>d</sup>	36.60 $\pm$ 0.38 <sup>d</sup>	22.24 $\pm$ 0.56 <sup>c</sup>	99.93 $\pm$ 2.54 <sup>a</sup>
CHMT15	35.25 $\pm$ 0.11 <sup>c</sup>	37.71 $\pm$ 0.14 <sup>c</sup>	24.17 $\pm$ 0.11 <sup>b</sup>	91.95 $\pm$ 0.40 <sup>b</sup>
CHMT20	35.72 $\pm$ 0.03 <sup>c</sup>	38.33 $\pm$ 0.02 <sup>bc</sup>	24.67 $\pm$ 0.29 <sup>b</sup>	90.04 $\pm$ 1.06 <sup>b</sup>
CHMT25	35.30 $\pm$ 0.14 <sup>c</sup>	37.84 $\pm$ 0.21 <sup>c</sup>	24.27 $\pm$ 0.06 <sup>b</sup>	91.52 $\pm$ 0.20 <sup>b</sup>
CHMT30	36.38 $\pm$ 0.21 <sup>b</sup>	38.68 $\pm$ 0.46 <sup>b</sup>	24.92 $\pm$ 0.46 <sup>b</sup>	89.17 $\pm$ 1.67 <sup>b</sup>
CHMT35	41.12 $\pm$ 0.42 <sup>a</sup>	45.60 $\pm$ 0.50 <sup>a</sup>	27.00 $\pm$ 0.81 <sup>a</sup>	82.33 $\pm$ 2.45 <sup>c</sup>

**Notes:** all values are the means of triplicate determinations  $\pm$  standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

### 3.2.8 Effect of heat moisture treatment on in vitro digestion of potato starch

The results of RDS, SDS, and RS of cooked native starch and HMT starch sample were shown in Table 3.10. The values of RDS, SDS, and RS fractions in NS were 31.14%, 55.17% and 13.69%, respectively. Compared to the native starch counterpart, HMT starches had less RDS and SDS, but more RS. The total content of SDS and RS of HMT starch was significantly higher than that of NS starch ( $p < 0.05$ ), the holding time of heat-moisture treatment had greatest effect on the content of RS, indicating that heat moisture treatment could rearrange the molecular chain and the newly formed ordered structures (crystallites and helices) increased the resistance of starch to enzymatic hydrolysis, eventually reduce the digestion of starch by transforming part of RDS into SDS and /or RS [161]. In particularly, tHMT1, THMT100 and CHMT15 had the highest content of SDS and RS, and there was no significant difference between the three samples. Therefore, choosing appropriate treatment time, temperature and moisture content can not only save energy, but also obtain modified starch with higher content of SDS and RS.

**Table 3.10**

RDS, SDS and RS contents of native and HMT starches

Samples	RDS (%)	SDS (%)	RS (%)
NS	31.14±0.10 <sup>a</sup>	55.17±0.17 <sup>a</sup>	13.69±0.10 <sup>h</sup>
tHMT1	28.28±0.30 <sup>fg</sup>	51.46±0.21 <sup>fg</sup>	20.25±0.10 <sup>b</sup>
tHMT2	29.91±0.27 <sup>c</sup>	51.16±0.39 <sup>g</sup>	18.93±0.25 <sup>de</sup>
tHMT3	30.46±0.71 <sup>b</sup>	50.28±0.26 <sup>h</sup>	19.25±0.16 <sup>d</sup>
tHMT4	28.73±0.00 <sup>ef</sup>	49.11±0.16 <sup>i</sup>	22.16±0.16 <sup>a</sup>
tHMT5	29.32±0.56 <sup>d</sup>	51.94±0.47 <sup>f</sup>	18.74±0.09 <sup>e</sup>
tHMT6	29.93±0.10 <sup>c</sup>	50.05±0.17 <sup>h</sup>	20.02±0.09 <sup>bc</sup>
THMT90	30.34±0.00 <sup>bc</sup>	52.59±0.48 <sup>e</sup>	17.07±0.48 <sup>g</sup>
THMT100	28.38±0.18 <sup>fg</sup>	54.13±0.25 <sup>b</sup>	17.48±0.38 <sup>f</sup>
THMT110	29.91±0.27 <sup>c</sup>	51.16±0.39 <sup>g</sup>	18.93±0.25 <sup>de</sup>
THMT120	29.89±0.36 <sup>c</sup>	53.35±0.21 <sup>cd</sup>	16.76±0.16 <sup>g</sup>
THMT130	30.30±0.17 <sup>bc</sup>	52.84±0.26 <sup>de</sup>	16.86±0.19 <sup>g</sup>
CHMT15	28.20±0.27 <sup>g</sup>	53.93±0.18 <sup>b</sup>	17.88±0.09 <sup>f</sup>
CHMT20	29.10±0.18 <sup>de</sup>	53.35±0.22 <sup>cd</sup>	17.54±0.09 <sup>f</sup>
CHMT25	29.91±0.27 <sup>c</sup>	51.16±0.39 <sup>g</sup>	18.93±0.25 <sup>de</sup>
CHMT30	28.78±0.50 <sup>ef</sup>	51.44±0.90 <sup>fg</sup>	19.79±0.41 <sup>e</sup>
CHMT35	29.16±0.44 <sup>de</sup>	53.78±0.12 <sup>bc</sup>	17.06±0.35 <sup>g</sup>

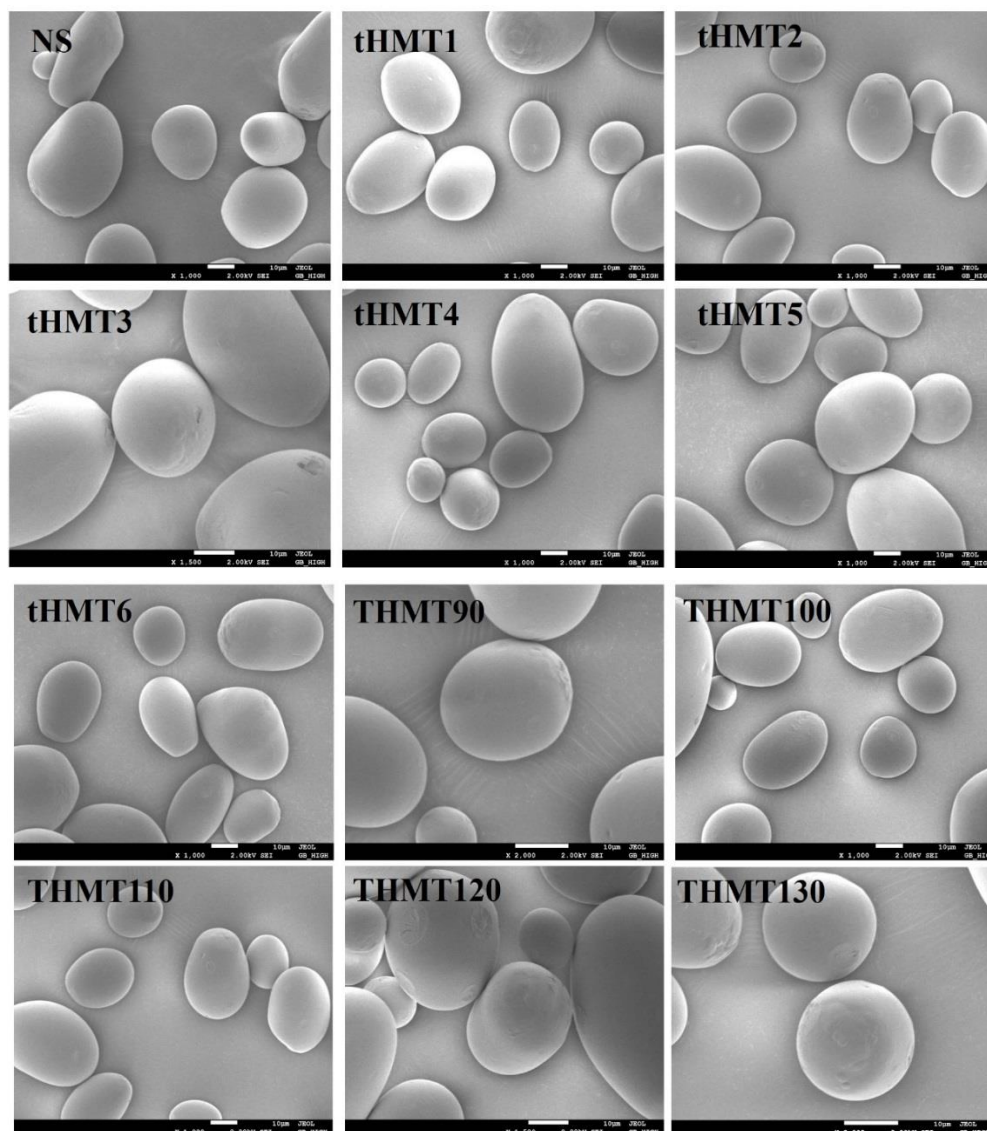
**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

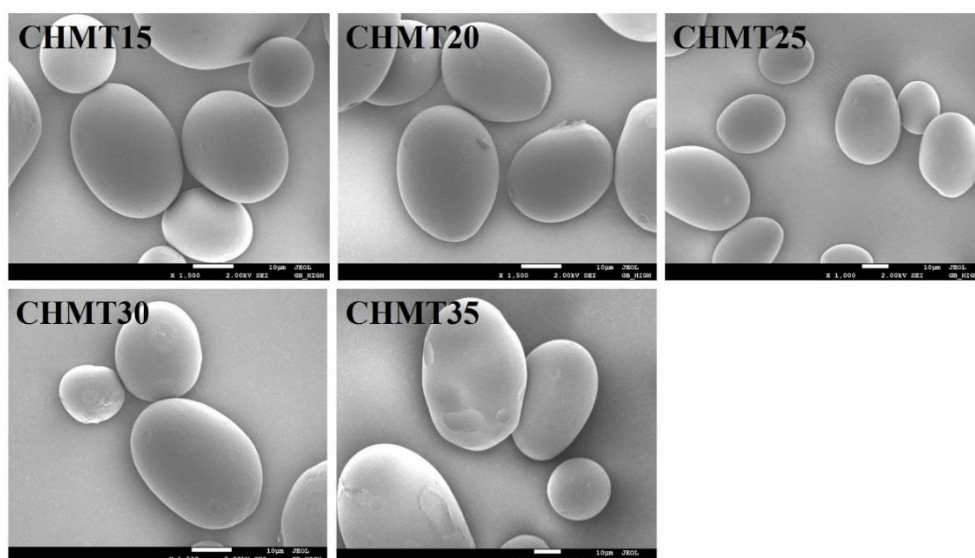
### 3.3 Effects of heat-moisture treatment conditions on the structural properties of potato starch

#### 3.3.1 Morphological properties of potato starch modified by heat-moisture treatment

The microscopic images of the NS and HTM starch samples obtained under SEM were shown in Fig.3.18. Almost all the NS starch showed elliptical shaped granules and some round shaped granules with a smooth surface. After heat moisture treatment, potato starch granules were damaged and showed a negligible change in terms of morphology at low moisture levels ( $\leq 25\%$ ), which was agreement with the results of HMT modified highland barley starch granules at low moisture levels [80]. Relatively high temperature (e.g., 120°C and 130°C)

and high moisture content (e.g., 30% and 35%) led to coarser and rougher granule surface than that of NS. Similar results were obtained for HMT modified rice starch that the granule surface became slightly rougher than that of NS with the increasing agglomeration degree of the starch granules [171].





**Fig. 3.18** Morphology of native potato starch and HMT starches

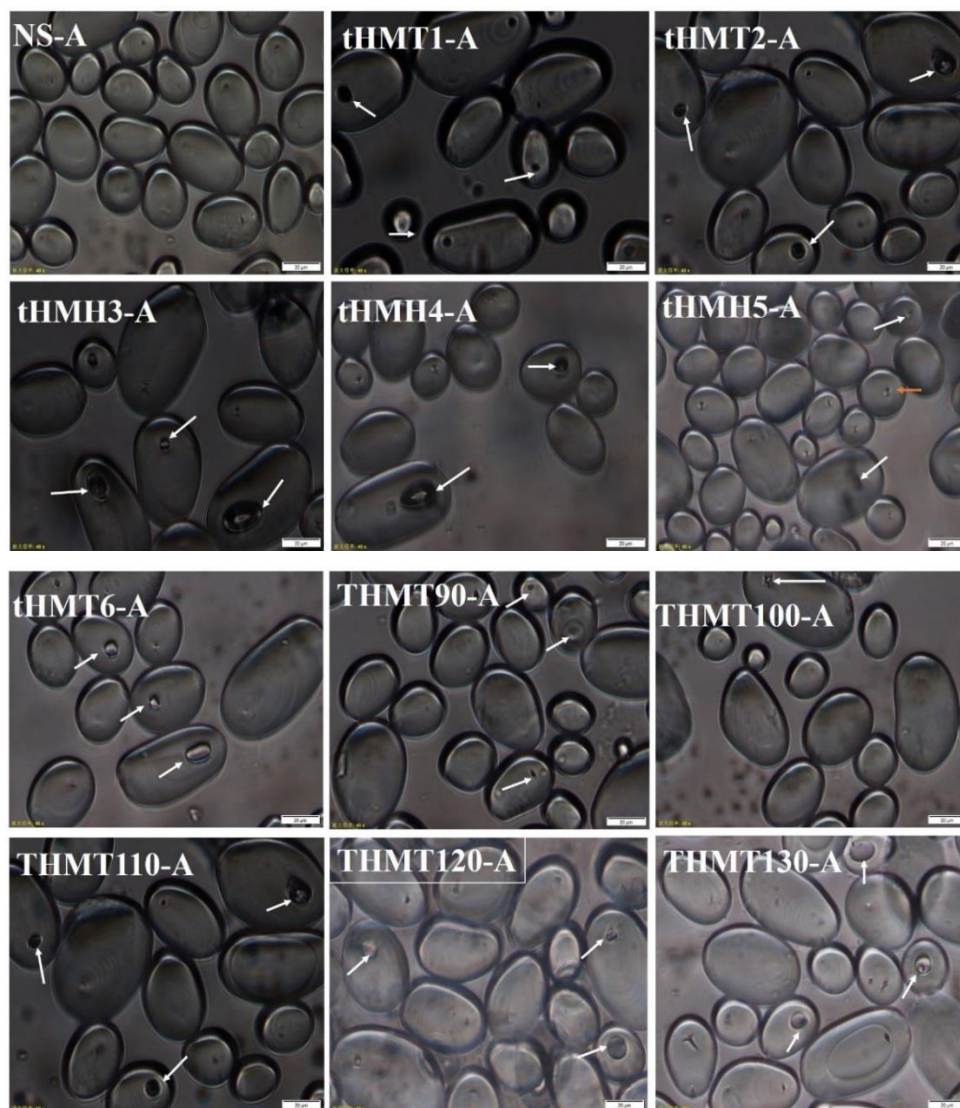
### **3.3.2 Light microscopic properties of potato starch modified by heat-moisture treatment**

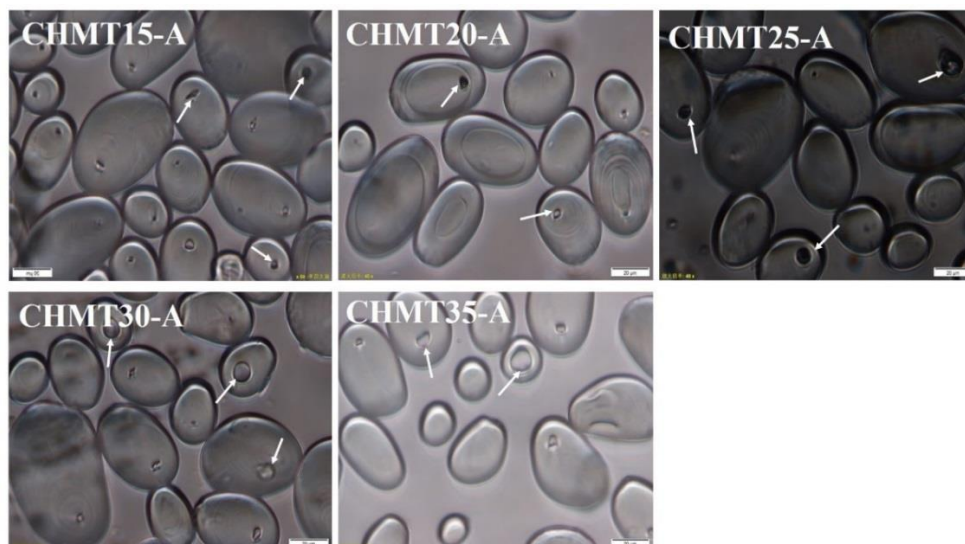
Normal light microscopy (NLM) and polarized light microscopy (PLM) were used to obtain the microscopic images of NS and HMT modified starch (Fig. 3.19 and Fig. 3.20). Smooth surface and hilum structure were obtained from NS granules under normal light. After HMT modification, the hilum structure of HMT potato starch became hollow. The diameter and depth of the hollow region increased with increasing the length of heating, moisture content and heating temperature. Similar results were obtained by Watcharatewinkul [172] and Huan Li [95]. The location of hilum structure which was in the amorphous zone led to relative fragility. Furthermore, high temperature and certain moisture condition during HMT caused partial swelling and disruption of starch granules, which was the reason that hollow central region became large.

Visible black polarization cross like “X” and “+” of NS starch granules was observed under polarized light. Maltese cross with the helium was biased toward to the small end of the starch granules, and the deviation of large granular starch was higher. However, for HMT starch, the contour of the Maltese cross became gradually unclear and showed large black area in the middle of the cross. HMT starch showed high birefringence intensity at moisture contents lower than 20%

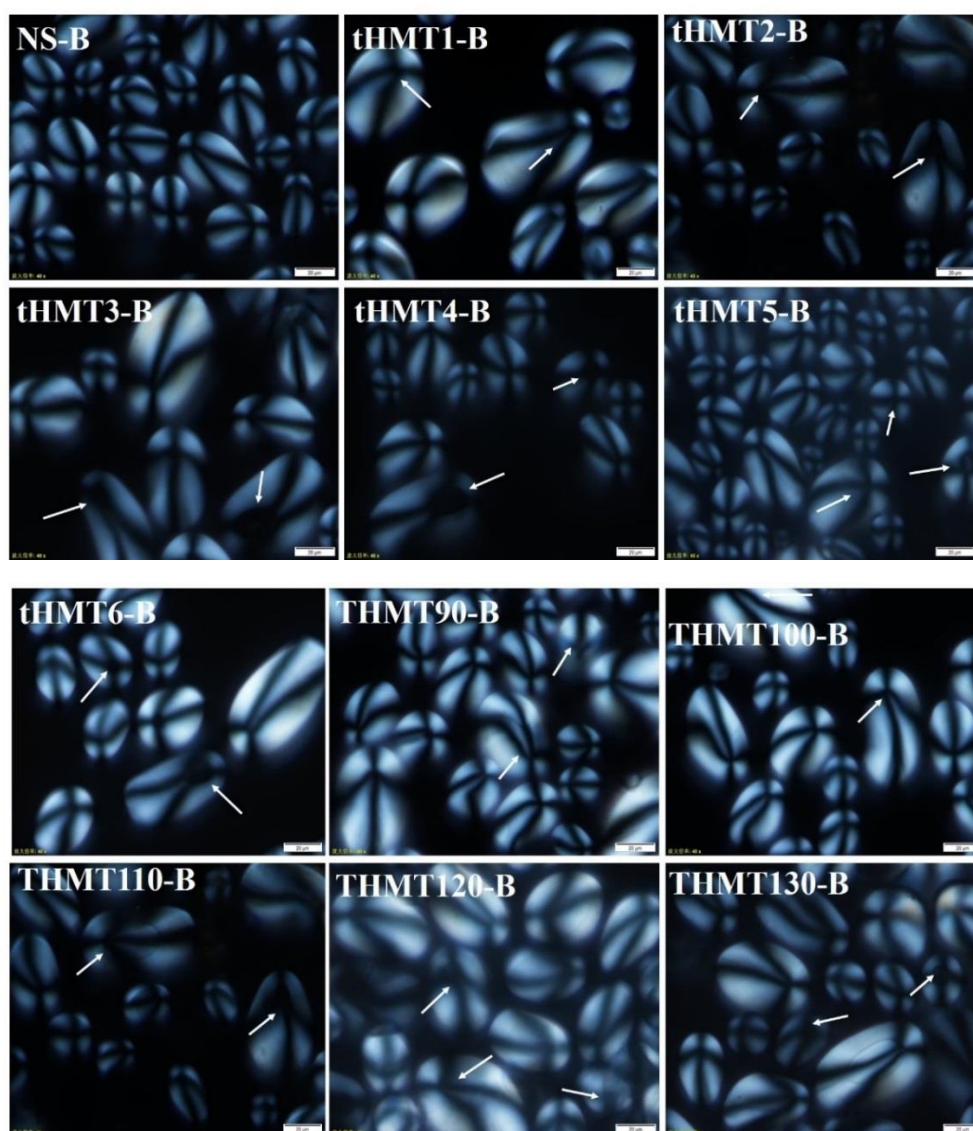


and heating temperature lower than 100°C, whereas a remarkable reduction of birefringence intensity was observed when the moisture content was higher than 25% and 110°C. The Maltese cross contour became unclear by short time HMT modification, and became obvious when heating length was over 5 h. the average orientation of helical structures caused birefringence patterns. The results were consistent with the results of pervious research of K Liu et al [80]. The decrease in birefringence intensity on HMT indicated a change in the radial orientation of double helices due to increased amylopectin chain flexibility.

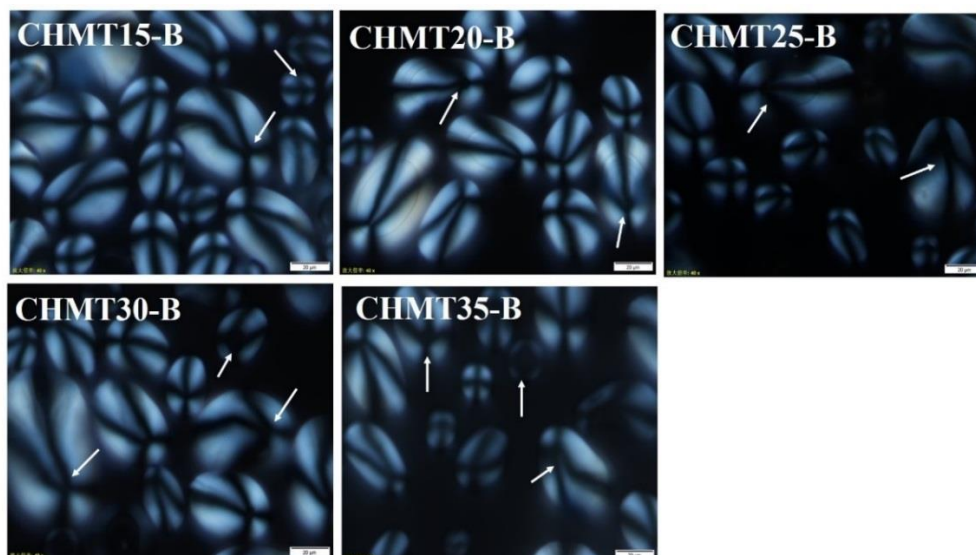




**Fig. 3.19** Morphological characteristics of the heat-moisture treated potato starch granules under normal light  $\times 400$  (A)





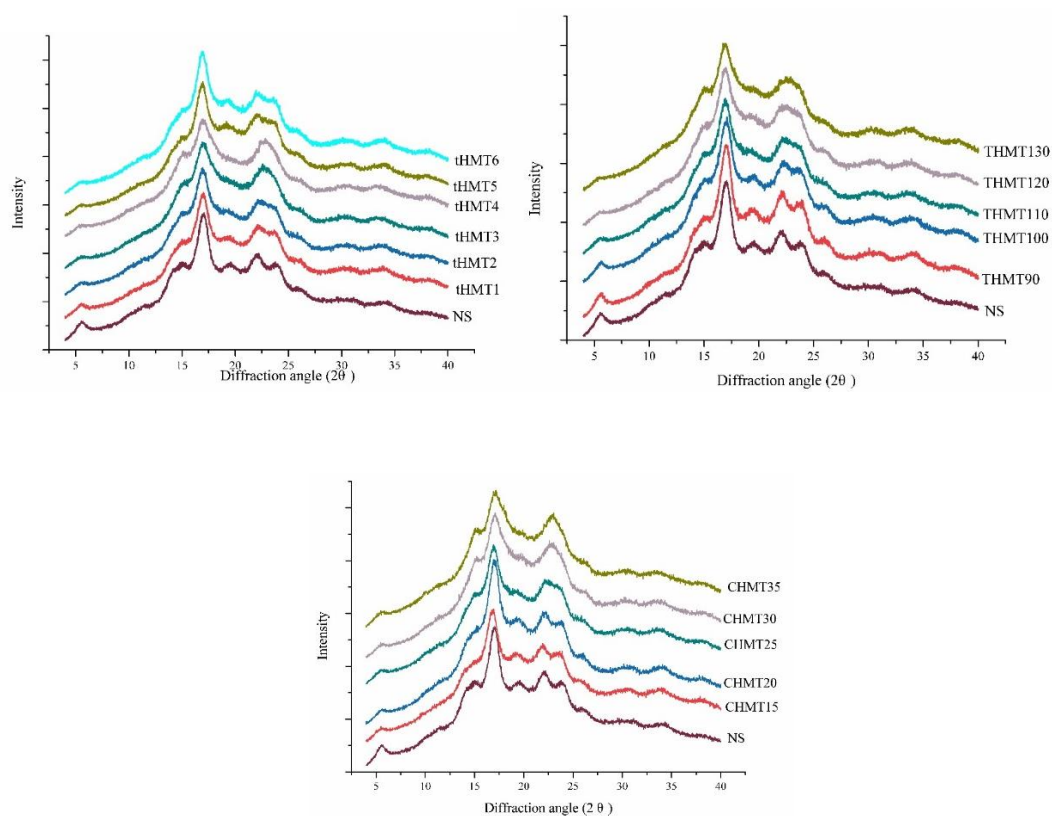


**Fig. 3.20** Morphological characteristics of the heat-moisture treated potato starch granules under polarized light  $\times 400$  (B)

### 3.3.3 X-ray diffraction (XRD) of potato starch modified by heat-moisture treatment

The diffraction patterns of NS and HMT starch samples were illustrated in Fig. 3.21. Four main peaks at  $5.6^\circ 2\theta$ ,  $17^\circ 2\theta$ ,  $22^\circ 2\theta$  and  $24^\circ 2\theta$  can be seen from the NS starch pattern, indicating that the crystal form of potato starch was B-type. Some significant changes in the crystalline patterns of HMT starch were observed. With the extension of heat moisture treatment process, all HMT starch diffraction peaks gradually disappeared at  $5.6^\circ 2\theta$  and  $19.5^\circ 2\theta$ , the diffraction peak at  $15^\circ 2\theta$  became smooth, and the two independent diffraction peaks at  $22^\circ 2\theta$  and  $24^\circ 2\theta$  tended to merge into a single peak near  $23^\circ 2\theta$ , which was a characteristic of A type. When treatment time reached 5 hours, the diffraction peak at  $23^\circ 2\theta$  divided into two diffraction peaks at  $22^\circ 2\theta$  and  $24^\circ 2\theta$  again. These results indicated the crystal structure of potato starch gradually changed from B-type to C-type with the extension heat moisture treatment. When treatment time was too long (more than 5 hours), its crystal structure changed to B-type again. Relative crystallinity for NS was 19.37% and the crystallinity of HMT potato starch initially increased and then decreased with the extension of the treatment time, but all HMT starch had higher crystallinity than that of NS (Table 3.1, Table 3.2

and Table 3.3). With the increase of treatment temperature from 90°C to 130°C, the crystallinity increased from 21.30% to 25.74%. With the increase of moisture content from 15% to 35%, the crystallinity increased from 21.46% to 25.09%. The results indicated that heat moisture treatment destroyed and rearranged the double helix structure of starch molecules, which strengthened the connection between starch chains, produced new and more stable crystalline structure, and ultimately led to an increase in the relative crystallinity of starch. The increased X-ray intensity was agreed with previous studies about HMT mung bean starch[161].

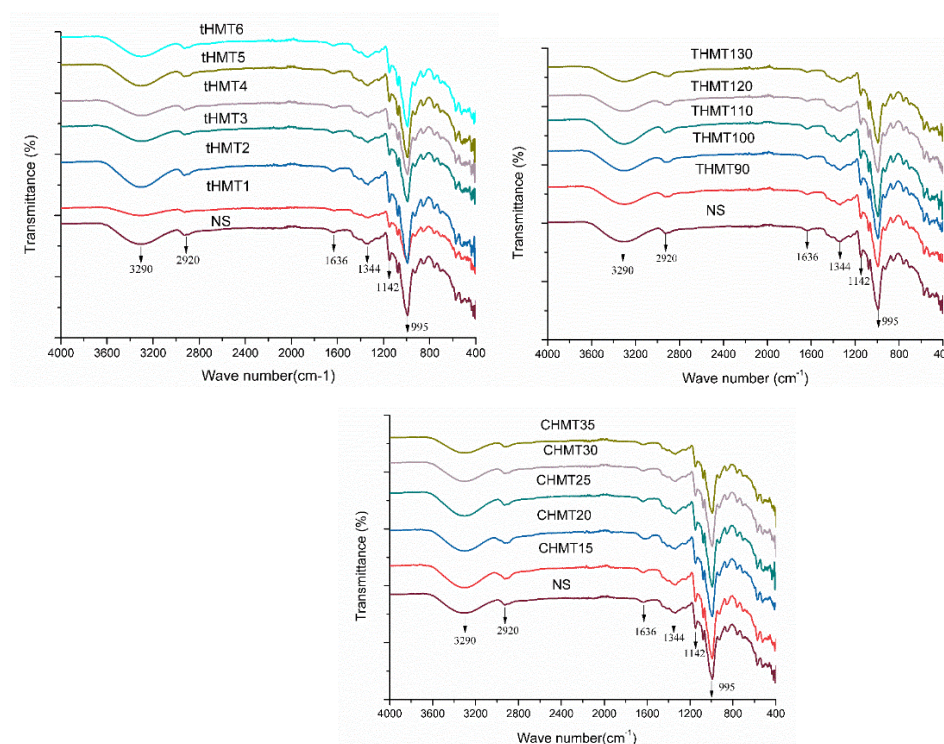


**Fig. 3.21** X-ray diffractograms of native and HMT potato starches

### 3.3.4 FTIR spectroscopy of potato starch modified by heat-moisture treatment

The FTIR spectra of NS and HMT starches were shown as Fig. 3.22. All samples showed the same characteristic peak at 3290, 2920, 1636 and 1344  $\text{cm}^{-1}$  and no new absorption peaks were generated, which indicated the original basic structure of potato starch was not destroyed and no new compounds were

produced after heat moisture treatment. However, it can be seen from the spectrograms within the spectrum range, the intensity of the absorption peaks was different with difference modification conditions, although the position of absorption peaks did not change. The absorption peak strength of potato starch increased or decreased, indicating that the molecular conformation of starch changed in the process of heat moisture treatment. With heat moisture treatment process extend, the infrared absorption peak intensity in the range of  $1000\text{ cm}^{-1}$ - $3300\text{ cm}^{-1}$  showed an overall trend of first weaken and then increased. The absorption peaks at  $1600\text{ cm}^{-1}$ - $1750\text{ cm}^{-1}$  correspond to the carbonyl ( $>\text{C}=\text{O}$ ) stretching vibration area [173]. The absorption peaks at  $2850\text{ cm}^{-1}$ - $3100\text{ cm}^{-1}$  correspond to the CH stretching vibration area and the absorption peaks at  $3100\text{ cm}^{-1}$ - $3700\text{ cm}^{-1}$  correspond to the hydroxyl (OH) stretching vibration area [174]. These results indicated that the heat moisture treatment may result in the breaking of starch molecular chain or the breaking of the associative hydrogen bond of starch molecule, which eventually leads to the increase of the number of carbonyl group and hydroxyl group, and it is possible to increase the new C-H bonds.

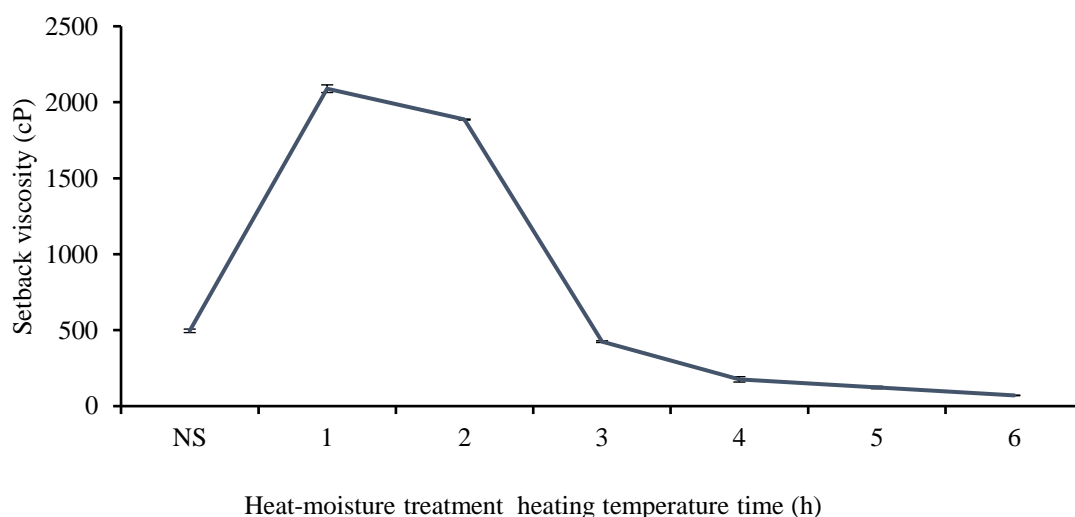


**Fig.3.22** FTIR spectra of native and HMT potato starches

### 3.4 Optimization of heat-moisture treatment processing parameters of potato starch by response surface methodology

#### 3.4.1. Effect of heat-moisture treatment on setback viscosity of potato starch under different heating time

The effect of heat-moisture treatment setback viscosity of potato starch under different holding time was shown as Fig. 3.23. Since starch molecules were in a messy state under the action of water and heat, the appropriate extension of the moisture and heat treatment time were beneficial to enhance the interaction between starch molecules, but too long time would lead to excessive degradation of starch molecules [175].

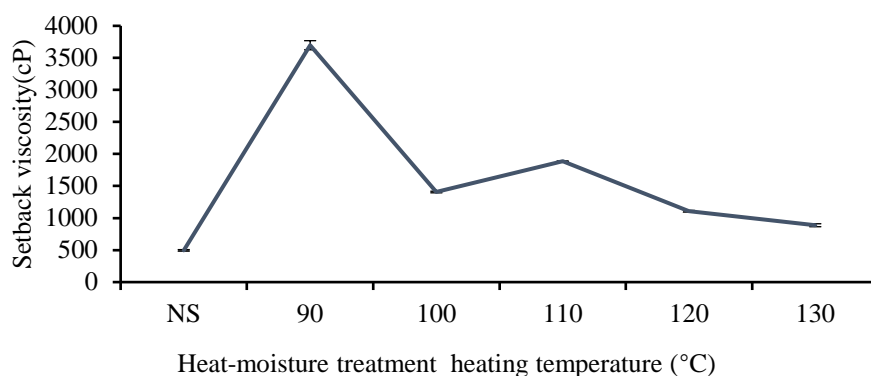


**Fig. 3.23** Effect of heat-moisture treatment on setback viscosity of potato starch under different heating time

As can be seen from Fig. 3.23, HMT heating time had significant effect on the setback viscosity of starch. Setback viscosity decreased with the extension of HMT heating time, the highest setback viscosity was 2089 cP at 1 h. Therefore, 0.5 h, 1 h and 1.5 h were used as the three levels (−1, 0, 1) of response surface, respectively.

#### 3.4.2. Effect of heat-moisture treatment on setback viscosity of potato starch under different heating temperature

The effect of heat-moisture treatment setback viscosity of potato starch under different heating temperature was shown as Fig.3.24.

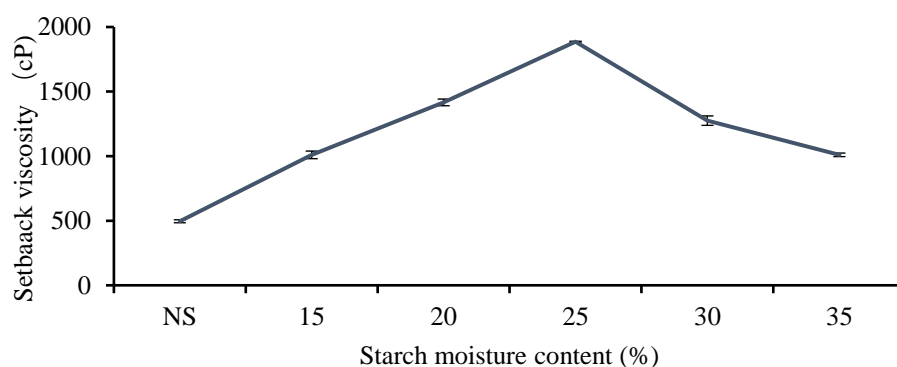


**Fig. 3.24** Effect of heat-moisture treatment setback viscosity of potato starch under different heating temperature

During the process of HMT, the water molecules in starch crystals turned into gaseous, destroyed the  $-1, 6$  and  $-1, 4$  bonds under the action of heating, changed the double helix structure of amorphous and crystalline regions at the meantime [176]. All these changes led to the related properties of starch changed accordingly. As can be seen from Fig. 1, HMT holding temperature could increase the setback viscosity of starch significantly, the highest setback viscosity was 3697 cP when the heating temperature was 90 °C. Considering energy consumption and cost comprehensively, 90 °C, 100 °C and 110 °C were used as the three levels ( $-1, 0, 1$ ) of response surface, respectively.

### 3.4.3. Effect of heat-moisture treatment on setback viscosity of potato starch under different starch moisture content

The effect of heat-moisture treatment setback viscosity of potato starch under starch moisture content was shown as Fig. 3.25.



**Fig. 3.25** Effect of heat-moisture treatment on setback viscosity of potato starch under different starch moisture content

As can be seen from Fig. 3.25, HTM can increased setback viscosity

significantly, with the extension of moisture content, the setback viscosity increased, the highest setback viscosity was 1886 cP when the moisture content was 25%. As a plasticizer, water had a significant impact on the structure and properties of starch during the HMT process. The presence of water was conducive to the destruction of the starch crystal structure and promoted the movement of starch molecular chains. The rearrangements of starch molecules in amorphous regions were formed more crystals. However, too low moisture content was not conducive to the interaction between the molecular chains in the amorphous regions; starch was gelatinized easily with too high moisture content which led to excessive degradation of starch molecules [177]. Therefore, 20%, 25% and 30% were used as the three levels (−1, 0, 1) of response surface, respectively.

#### 3.4.4. Experimental analysis of response surface and model establishment

On the basis of single factor experiments, Box-Behnken design (BBD) with three variables (temperature, time and moisture content) at three different levels were used to find a possible correlation among these variables and obtained a response surface. The factors and levels in the response surface analysis were shown as Table 3.11.

**Table 3.11**

The factors and levels in the response surface analysis.

Levels	Factors		
	<i>A</i> : Temperature (°C)	<i>B</i> : Time (h)	<i>C</i> : Moisture content (%)
−1	90	0.5	20
0	100	1	25
1	110	1.5	30

The central point experiment was set to be repeated for five times and total of 17 experiments with three different levels (coded as −1, 0, 1) of each factor, which were shown as Table 3.12.



**Table 3.12**

Experimental design and corresponding results for RSM

Run	<i>A</i> : Temperature (°C)	<i>B</i> : Time (h)	<i>C</i> : Moisture content (%)	Setback viscosity (cP)
1	−1	−1	0	1711.667
2	1	−1	0	2525.333
3	−1	1	0	3890.667
4	1	1	0	858.000
5	−1	0	−1	1320.000
6	1	0	−1	1740.000
7	−1	0	1	2788.667
8	1	0	1	835.333
9	0	−1	−1	966.333
10	0	1	−1	3352.667
11	0	−1	1	3072.333
12	0	1	1	1304.333
13	0	0	0	2785.333
14	0	0	0	3009.667
15	0	0	0	2901.000
16	0	0	0	2848.667
17	0	0	0	2835.333

All experiments were performed in triplicate. The relationships between the responses and the independent variables were constituted by means of the quadratic polynomial equation. The optimum conditions were deduced using multiple response analysis.

Response surface methodology (RSM) was used to optimize the process of HMT potato starch under the effect of the independent variables (heating temperature, heating time and moisture content) with setback viscosity as an index. The central point experiment was set to be repeated for five times and total of 17 experiments with three different levels (−1, 0, 1) of each factor were listed in Table 3.12 and the analysis of variance (ANOVA) results of quadratic regression model for RSM was listed in Table 3.13. All experiments were performed in triplicate.

**Table 3.13**

## ANOVA for response surface quadratic model

Source	Sum of Squares	<i>df</i>	Mean Square	<i>F</i> Value	<i>P</i> value Prob> <i>F</i>	Significance
Model	14698408	9	1633156	94.20812	<0.0001	**
<i>A</i>	1760001	1	1760001	101.5251	<0.0001	**
<i>B</i>	159612.5	1	159612.5	9.207197	0.019	*
<i>C</i>	48308.68	1	48308.68	2.786671	0.139	—
<i>AB</i>	3698570	1	3698570	213.3509	<0.0001	**
<i>AC</i>	1408178	1	1408178	81.2303	<0.0001	**
<i>BC</i>	4314621	1	4314621	248.8876	<0.0001	**
<i>A</i> <sup>2</sup>	1350059	1	1350059	77.87774	<0.0001	**
<i>B</i> <sup>2</sup>	16888.89	1	16888.89	0.97423	0.3565	—
<i>C</i> <sup>2</sup>	1717901	1	1717901	99.0966	<0.0001	**
Residual	121349.4	7	17335.62	—	—	—
Lack of Fit	92236.25	3	30745.42	4.224271	0.0989	—
Pure Error	29113.11	4	7278.278	—	—	—
Cor Total	14819758	16	<i>R</i> -Squared	0.991812	Adj <i>R</i> - Squared	0.9813

**Notes:** \* – indicated significant ( $P_{value} < 0.05$ ); \*\* – indicated extremely significant ( $P_{value} < 0.01$ ); *A* – temperature; *B* – time; *C* – moisture content

The setback viscosity of HMT starch was influenced by holding temperature, time and moisture content were fitted with the quadratic polynomial model represented by the following equations:

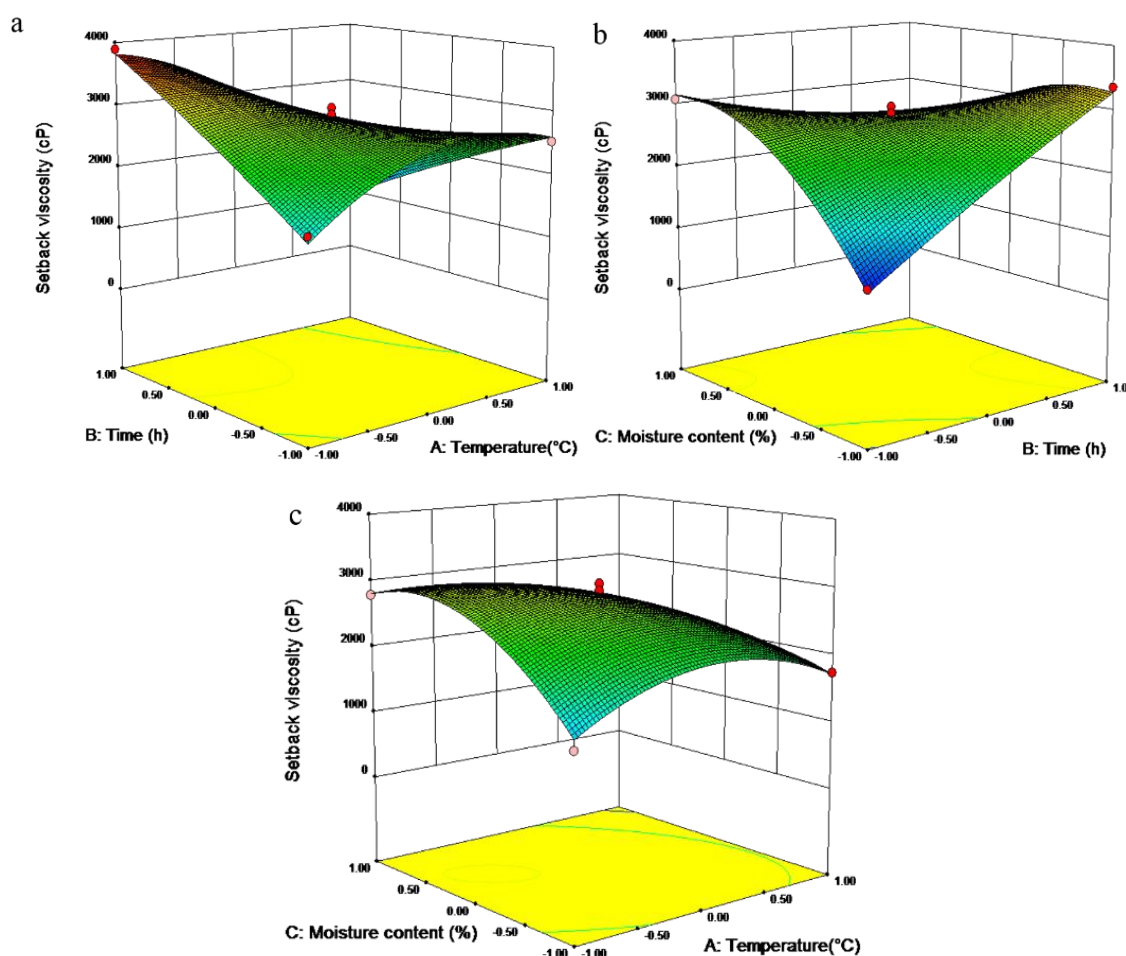
$$S_v = 2876 - 469.04 \cdot A + 141.25 \cdot B + 77.71 \cdot C - 961.58 \cdot A \cdot B - 593.33 \cdot A \cdot C - 1038.58 \cdot B \cdot C - 566.25 \cdot A^2 - 63.33 \cdot B^2 - 638.75 \cdot C^2,$$

where  $S_v$  is setback viscosity (cP); *A*, *B*, *C* were the HMT holding temperature (°C), time (h) and moisture content (%), respectively.

ANOVA was performed for the model (Table 3.13) and observed that the model *F*-value of 94.21 implied the model was significant. There was only a 0.01% chance that a «Model *F*-Value» this large could occur due to noise. Values of «Prob>*F*» less than 0.0500 indicated model terms were significant. In this case *A*, *B*, *AB*, *AC*, *BC*, *A*<sup>2</sup>, *C*<sup>2</sup> were significant model terms. The «Lack of Fit *F*-value» of 4.22 implied there was a 9.89% chance that a «Lack of Fit *F*-value» this large

could occur due to noise. Lack of fit was significant ( $P=0.0989>0.05$ ), the model fitted well. The «Pred  $R$ -Squared» of 0.8973 was in reasonable agreement with the «Adj  $R$ -Squared» of 0.9813. «Adeq Precision» measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio of 31.015 indicated an adequate signal. This model can be used to navigate the design space.

The response surfaces of interaction of variables on setback viscosity were shown in Fig. 3.26.



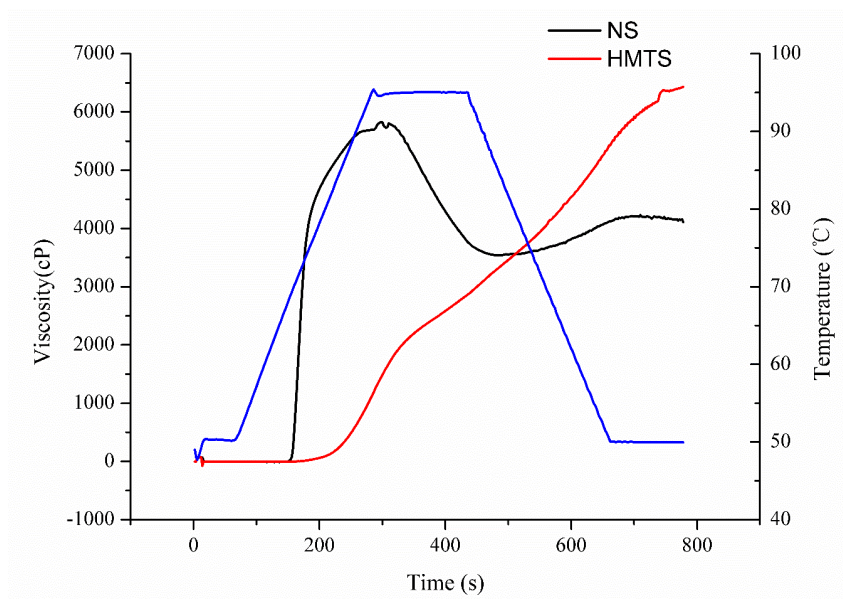
**Fig. 3.26** The response surfaces for the interaction of various factors on setback viscosity of HMT modified starches: a – the interaction of HMT heating temperature and heating time; b – the interaction of HMT heating time and starch moisture content; c – the interaction of HMT heating temperature and starch moisture content

The Design-Expert 8.0.6 software was used to optimize the setback viscosity equation model formula, and the optimized process parameters of setback viscosity of HMT were as follow: the temperature was 90°C (coded –1), the time was 1.5 h (coded 1) and the moisture content was 23.56% (coded –0.288), under

such conditions the maximum theoretical setback viscosity value was 3871 cP. In order to verify the reliability of the model, triplicate experiments were carried out with the optimal process parameters (the temperature was 90°C, the time was 1.5 h and the moisture content was 23.56%). The mean setback viscosity was 3677 cP and there was little error between the actual setback viscosity and theoretical setback viscosity. Therefore, it was feasible to use RSM to optimize the process of HMT on potato starch and had practical application value.

### 3.4.5 Comparative analysis of starch pasting properties

The starch pasting properties are correlated to the gel texture properties, the stability of starch paste, and retrogradation tendency. The pasting properties of native potato starch (NS) gel and optimized heat-moisture treatment modified starch (HMTS) were listed in Table 3.14 and the rapid viscosity analysis (RVA) pasting profiles were illustrated in Fig. 3.27.



**Fig. 3.27** Rapid viscosity analysis (RVA) pasting profiles of native and modified potato starches

As shown in Fig. 3.27, the RVA profiles of HMTS had great difference with that of NS. Compared with native potato starch (NS), HMTS had lower peak viscosity (2966 cP), lower hold viscosity (2882 cP) and lower breakdown viscosity (84.5 cP), but higher paste temperature (71.1°C), higher final viscosity

(6559 cP) and setback viscosity (3677 cP). HMTS had lower viscosity but higher gelatinization temperature, indicating that the starch modified by heat moisture treatment requires more energy to decompose its structure and form paste, thus improving the gelatinization of the sample. Heat moisture treatment could enhance the interior interactions of starch, preventing the penetration of water into starch granules, thus resulting in decrease of peak viscosity. As shown in Table 4, the peak viscosity of HMTS was lower than final viscosity, while the peak viscosity of NS was higher than final viscosity, indicating that HMTS was more prone to retrogradation than NS [178].

**Table 3.14**

Pasting properties of native and modified potato starches.

Samples	Paste temperature (°C)	Peak viscosity (cP)	Hold viscosity (cP)	Final viscosity (cP)	Breakdown viscosity (cP)	Setback viscosity (cP)
NS	68.5±0.2 <sup>b</sup>	6598±73 <sup>a</sup>	4072±25 <sup>a</sup>	4567±37 <sup>b</sup>	2526.3±47.3 <sup>a</sup>	496±12 <sup>b</sup>
HMTS	71.1±0.8 <sup>a</sup>	2966±46 <sup>b</sup>	2882±44 <sup>b</sup>	6559±61 <sup>a</sup>	84.5±2.9 <sup>b</sup>	3677±21 <sup>a</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ )

The breakdown viscosity is related to the thermal stability of the swollen starch granules in the starch paste during heating and shearing, lower breakdown indicates more resistance to shear force [167-168]. The setback viscosity has great relativity to the retrogradation properties of starch and has a great influence on the tensile strength of vermicelli food or noodles, higher setback viscosity indicates higher tensile strength [160].

### 3.4.6 Comparative analysis of textural properties

Textural properties of the native potato starch (NS) gel and optimized HMT potato starch (HMTS) gels, including hardness, springiness, cohesiveness, gumminess, chewiness and resilience, were determined by TPA test (Table 3.15). The textural properties of starch gels depend on the constituents of starch and amylose, the volume and deformation of de granules and the interaction between the continuous and dispersed phases [179].

**Table 3.15****Textural properties native and modified potato starches**

Starch gel	Hardness (g)	Springiness (mm)	Cohesiveness (-)	Gumminess (g)	Chewiness (g·mm)	Resilience (-)
NS	2706±3 <sup>a</sup>	0.83±0.01 <sup>a</sup>	0.63±0.00 <sup>b</sup>	1700±11 <sup>b</sup>	1404±30 <sup>b</sup>	0.40±0.03 <sup>a</sup>
HMTS	6082±8 <sup>b</sup>	0.81±0.00 <sup>a</sup>	0.73±0.00 <sup>a</sup>	4920±2 <sup>a</sup>	3570±12 <sup>a</sup>	0.42±0.03 <sup>a</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

Gel hardness generally related to the retrogradation of starch. Previous study reported a positive correlation between gel hardness and the amylose content of the starch and high-amylose starches produced a harder gel [180]. Linear amylose molecules re-associate more easily than high-branched amylopectin molecules. As can be seen, the HMTS gel showed significantly higher hardness than NS gel, the difference between the hardness of NS gel and HMTS gel can be attributed to higher amylose content of HMTS.

Springiness reflects the ability of starch gel to recover from its deformation with a period of time after being squeezed. The springiness of starch gel is affected by the number of crosslinking points and the density of crosslinking points of the network structure formed by starch molecules. The more effective crosslinking points, the greater springiness of the gel. There was no significant difference of springiness between NS gel and HMTS gel, although NS gel showed greater springiness than HMTS gel.

Cohesiveness is a measure of how well a starch gel resists the second deformation according to its behavior in the first deformation, which means cohesiveness is a criterion of how well a starch gel can keep its structure after the first bite [181]. Starch gels with good gel setting and high acceptability in starchy food are identified by high cohesiveness. HMTS gel represented the higher amount of cohesiveness (0.7255) than NS gel (0.6285), which indicated that the HMTS has a better ability to withstand deformations.

Gumminess ( $\text{hardness} \times \text{springiness}$ ) is the energy required to break up a semi-solid food to ready it for swallowing [179]. HMTS gel showed higher gumminess than NS gel about 189.4%, which indicating that HMTS gel needs more energy to be ready for swallowing about 2.89-fold than NS gel. Chewiness ( $\text{hardness} \times \text{springiness} \times \text{cohesiveness}$ ) is the energy required for disintegration and mastication of semi-solid foods. HMTS gel showed higher than NS gel about 154.27%, which indicating that HMTS gel needs more energy to be ready for swallowing about 2.54-fold than NS gel. The variations in gumminess and chewiness values showed a similar trend as hardness, due to the high relative weight of hardness in calculating these textural parameters [182]. Resilience is other criterion of TPA test, measure how well the starch gel fights to regain its original position. HMTS gel showed higher resilience than NS gel, but there was no significant difference between them.

The changes in the textural characteristic of HMTS gel were mainly due to the rearrangement of starch molecules, and starch chains was cross-linked with non-starch molecules such as protein and fat, making the starch structure denser after the gel treatment, thus enhanced the textual properties of starch.

### **3.4.7. Comparative analysis of retrogradation**

The retrogradation of starch is the process of gelatinized starch molecules from disordered state to orderly rearrangement, finally coagulation and sedimentation. In the gelatinization process of starch by heating, the ordered starch molecules become disordered under the action of water and heat. In the process of cooling and storage, due to the effect of molecular potential energy, the disorder of high energy states gradually tends to the order of low energy states.

As can be seen from Table 3.16, retrogradation of NS and HMTS increased with the extension of storage time, and the HMTS had higher retrogradation than that of NS, indicating that potato starch modified by heat-moisture treatment was more prone to retrogradation. This result was consistent with viscosity properties of NS and HMTS. Heat-moisture treatment destroyed the starch granules structure

and reduced the hydrogen bonds between starch molecules and water molecules, which was prone to retrogradation, resulting in poor the retrogradation stability of potato starch.

**Table 3.16**

Retrogradation of native and modified potato starches

Samples	Retrogradation (%)							
	2 h	4 h	6 h	8 h	10 h	12 h	14 h	16 h
NS	0.00±0.00 <sup>b</sup>	8.00±0.00 <sup>b</sup>	8.00±0.00 <sup>b</sup>	8.00±0.00 <sup>b</sup>	8.00±0.00 <sup>b</sup>	8.00±0.00 <sup>b</sup>	8.00±0.00 <sup>b</sup>	8.00±0.00 <sup>b</sup>
HMTS	12.80±1.06 <sup>a</sup>	22.53±1.29 <sup>a</sup>	28.00±1.83 <sup>a</sup>	30.80±1.74 <sup>a</sup>	32.00±0.80 <sup>a</sup>	34.00±1.20 <sup>a</sup>	34.53±1.29 <sup>a</sup>	35.33±1.67 <sup>a</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

The results of this research indicate that it is feasible to use RSM to optimize the process of HMT on potato starch and has practical application value. And the HMT starch prepared under the optimized conditions is suitable for the production of vermicelli, noodles and other products that require good retrogradation characteristics, which is of great significance in promoting potato as staple food. However, the modified potato starch is not suitable for products that do not need good retrogradation properties for the HMT modified gel is easy prone to retrogradation.

### 3.4.8 Comparative analysis of *in vitro* digestibility

Intrinsic and extrinsic factors affect the starch digestibility, of which external factors include the effect of processing of conditions, modified treatments and additives [183]. RDS will cause a sudden rise of blood glucose level after ingestion can be used as a proxy indicator of GI value [184]. SDS is slowly broken down and completely digested in human small intestine, instead of causing a rapid rise of blood glucose level, it maintains blood glucose at steady level, thus playing an important role in the diet of type 2 diabetic patients [185]. RS is the portion of starch that is not absorbed in the small intestine of healthy individuals[186], but it can be used as a substitute of dietary fiber in foods to improve the processing characteristics and nutritional value [187].



As shown in Table 3.17, the optimized HMT potato starch (HMTS) had higher SDS content and RS content than that of native potato starch (NS), but lower RDS content. Compared to the native potato starch, the RDS decreased by 10.05%, the SDS increased by 5.06% and the RS increased by 2.48%, respectively in the optimized HMT potato starch. The increase of SDS and RS in the optimized HMT potato starch indicated that the HMT could enhance the interactions between amylose–amylose and amylose–amylopectin, which may partially restrict the accessibility of starch chains to the enzymes [100].

**Table 3.17**

RDS, SDS and RS contents of native and modified potato starches

Samples	RDS (%)	SDS (%)	RS (%)
NS	31.14±0.10 <sup>a</sup>	55.17±0.17 <sup>b</sup>	13.69±0.10 <sup>b</sup>
HMTS	28.01±0.73 <sup>b</sup>	57.96±0.44 <sup>a</sup>	14.03±0.16 <sup>a</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

### Conclusions to section 3

1. In this section, the effect of HMT on the swelling power, solubility, freeze-thawing stability, retrogradation, transparency and textural properties of NS and HMT starch were investigated. The research results show that the transparency and retrogradation stability of potato starch after HMT were reduced, solubility and swelling power varied with the gelatinization temperature. The HMT potato starch pasting properties results indicated that it is necessary to select appropriate heat-moisture treatment conditions for the preparation of vermicelli food by using the HMT potato starch in order to obtain better edible quality. HMT can significantly affect the textural properties of potato starch and the hardness, gumminess, chewiness and resilience of HMT starch gels first increased significantly and then decreased with the extension of treatment time. Short heating time ( $<1.5$  h), relatively low heating temperature ( $<100^{\circ}\text{C}$ ) and low moisture content ( $<25\%$ ) of HMT can significantly enhance the texture properties of HMT starch gels. The changes of physicochemical and textural properties of HMT starches could account for destruction of starch granules, the rearrangement of starch molecule or / and re-associations of starch chains caused by HMT, which limited the ability of starch molecules and water molecules to bind to each other through hydrogen bonds.

2. HMT had great effect on the particle size, morphological, structural, and digestive properties of potato starch. HMT led to large particle size, rough surface of starch granules and resulted in hollow structure located at the hilum of potato starch granules. XRD results showed an increased relative crystallinity and transformed crystalline structure from B-type to C-type with the extension heat moisture treatment. FTIR spectroscopy results indicated that the heat moisture treatment may result in the breaking of starch molecular chain or the breaking of the associative hydrogen bond of starch molecule, which eventually leads to the increase of the number of carbonyl group and hydroxyl group, and it is possible

to increase the new C-H bonds. In vitro digestion results showed that RDS content significantly decreased, whereas RS content in HMT starch increased, indicating HMT starches could potentially become sources of industrial-resistant starch and as low-calorie food ingredients.

3. Response surface methodology (RSM) was used to optimize the process of HMT potato starch with setback viscosity as an index, and the optimized process parameters of setback viscosity of HMT were as follow: the temperature was 90°C (coded -1), the time was 1.5 h (coded 1) and the moisture content was 23.56% (coded -0.288). Under such conditions the maximum theoretical setback viscosity value was 3871 cP. The verification experiment showed the actual mean setback viscosity was 3677 cP and there was little error between the actual setback viscosity and theoretical setback viscosity. Therefore, it was feasible to use RSM to optimize the process of HMT on potato starch and had practical application value.

4. The RVA profiles of HMTS had great difference with that of NS. Compared with native potato starch (NS), HMTS had lower peak viscosity (2966 cP), lower hold viscosity (2882 cP) and lower breakdown viscosity (84.5 cP), but higher paste temperature (71.1°C), higher final viscosity (6559 cP) and setback viscosity (3677 cP). TPA tests demonstrated that HMT can enhance the textural properties of starch gel. Compared with the NS gel, the hardness, cohesiveness, gumminess, chewiness and resilience of HMTS gel were increased significantly, and there was no significant difference in springiness. The retrogradation of NS and HMTS increased with the extension of storage time, and the HMTS had higher retrogradation than that of NS, indicating that potato starch modified by heat-moisture treatment was more prone to retrogradation. The optimized HMT potato starch (HMTS) had higher SDS content and RS content than that of native potato starch (NS), but lower RDS content.

## **SECTION 4 EFFECTS OF HEAT-MOISTURE TREATMENT COMBINED WITH MICROWAVE PRE-AND POST-TREATMENT ON PHYSICOCHEMICAL, STRUCTURAL AND DIGESTIVE PROPERTIES OF POTATO STARCH**

The effects of single HMT and MW on functionalities and structural properties of starch have been investigated by many researchers as reviewed by Schafranski *et al.* [74] and Oyeyinka *et al.* [22]. However, the mechanism of dual modification combined with HMT and MW is far from being fully understood. Therefore, the purpose of this research was to evaluate the effects of HMT assisted by MW pre- and post-treatment on the morphological, physicochemical and *in vitro* digestion properties of potato starch. This research presented a comprehensive understanding on the effects of HMT and MW bi-directional modifications on functional and digestibility properties of starch, as well as the mechanism, which would provide a useful theoretical basis for further studies on improving the application of microwave technology in starch modification.

### **4.1 Preparation of modified starch**

#### **4.1.1 Microwave treatment modified potato starch preparation**

The moisture content of native potato starch was adjusted to 25% as described above and the starch mixture was equilibrated at 25°C for 24 h. 70 g moisture-balanced starch was placed flat into a lab dish (diameter: 18 cm), covered with microwave-specific plastic wrap, and 10 holes were evenly pierced with toothpicks. Then, the lab dish with starch was placed in a microwave oven (G80F20CN2L-B8(RO), Guangdong Galanz Microwave Appliance Manufacturing Co., LTD, Foshan, Guangdong, China) for 5 min at 400 W power (increasing the power or time of microwave heating will cause the starch granules to burn). The treated starch samples were dried and stored according to the method mentioned above in single HMT. The prepared starches were named MWS.

#### **4.1.2 Dual modification of starch**

(1) Heat-moisture treatment assisted by pre- treatment of microwave (MW-HMT): the moisture-balanced MWS samples (25%) were subjected to HMT at 90°C for 1.5 h, 4 h, 8 h and 12 h, next, dried and stored according to the procedure described above for single HMT. The prepared starches were named as MW-HMT1.5, MW-HMT4, MW-HMT8 and MW-HMT12, respectively.

(2) Heat-moisture treatment assisted by poste-treatment of microwave (HMT-MW): The moisture-balanced (25%) native potato starch was subjected to the HMT at 90°C for 1.5 h, 4 h, 8 h and 12 h, respectively to obtained single HMT modified potato starch (HMT1.5, HMT4, HMT8 and HMT12, respectively). To obtain HMT-MW starches, the moisture content of HMT1.5, HMT4, HMT8 and HMT12 samples was adjusted to 25%. After equilibration at 25°C for 24 h, the starch mixtures were subjected to MW according to the description above for single MW. The starch samples were dried and named as HMT1.5-MW, HMT4-MW, HMT8-MW and HMT12-MW, respectively.

## **4.2 Effects of heat-moisture treatment combined with microwave pre- and post- treatment on the physicochemical properties of potato starch**

### **4.2.1 Effects of eat-moisture treatment combined with microwave pre- and post- treatment on swelling power and solubility of potato starch**

Differences in cohesive force within starch granules can be characterized by differences in swelling and solubility. The swelling power of native and modified potato starches is shown in Table 4.1 and the solubility of native and modified potato starches is shown in Table 4.2.

The swelling power of native starch and modified starches increased with increase of test temperature. The modified starch showed lower swelling power than native starch when the test temperature was 65°C, 75°C and 85°C, while opposite results were obtained at 95°C, and the MWS sample showed the highest swelling power (21.58%) at 95°C. MWS had the higher swelling power than MW-HMT modified starch at the same test temperature; moreover, the swelling power

of HMT modified starch (HMT, MW-HMT and HMT-MW) generally decreased with the treatment time when test temperature was same. The decrease in starch swelling power can be attributed to the enhancement of molecular interactions between amylose and amylopectin, and the amylose-lipid complexes formed during MW and HMT [7]. The swelling power of starch granules can also be affected by starch granules physical destruction, starch molecule rearrangement or/and starch chains re-associations induced by MW and HMT [21].

**Table 4.1****Swelling power of the native and modified potato starches**

Sample	Swelling power (%)				
	55°C	65°C	75°C	85°C	95°C
NS	2.28±0.03 <sup>h</sup>	12.13±0.79 <sup>a</sup>	14.14±0.16 <sup>a</sup>	17.10±0.12 <sup>a</sup>	13.29±0.57 <sup>f</sup>
MWS	4.02±0.23 <sup>a</sup>	12.94±0.19 <sup>b</sup>	12.59±0.20 <sup>c</sup>	15.74±0.26 <sup>b</sup>	21.58±0.04 <sup>a</sup>
MW-HMT1.5	3.05±0.01 <sup>b</sup>	11.20±0.17 <sup>c</sup>	12.31±0.32 <sup>cd</sup>	12.92±0.11 <sup>d<sup>ef</sup></sup>	16.37±0.14 <sup>c</sup>
MW-HMT4	2.84±0.05 <sup>c</sup>	9.58±0.19 <sup>ef</sup>	11.65±0.55 <sup>e</sup>	12.90±0.09 <sup>def</sup>	14.27±0.14 <sup>e</sup>
MW-HMT8	2.63±0.07 <sup>ef</sup>	9.04±0.24 <sup>gh</sup>	10.56±0.16 <sup>gh</sup>	11.27±0.23 <sup>g</sup>	14.12±0.83 <sup>e</sup>
MW-HMT12	2.52±0.02 <sup>fg</sup>	8.69±0.23 <sup>h</sup>	10.70±0.02 <sup>fg</sup>	11.19±0.12 <sup>g</sup>	14.35±0.38 <sup>e</sup>
HMT1.5	2.46±0.10 <sup>g</sup>	11.13±0.39 <sup>cd</sup>	13.28±0.64 <sup>b</sup>	13.76±0.70 <sup>c</sup>	18.27±0.31 <sup>b</sup>
HMT4	2.48±0.06 <sup>g</sup>	10.00±0.03 <sup>c</sup>	12.36±0.19 <sup>cd</sup>	13.35±0.16 <sup>cd</sup>	16.36±0.09 <sup>b</sup>
HMT8	2.47±0.04 <sup>g</sup>	9.18±0.15 <sup>gh</sup>	11.56±0.03 <sup>c</sup>	13.16±0.25 <sup>de</sup>	15.53±0.25 <sup>d</sup>
HMT12	2.47±0.02 <sup>g</sup>	8.85±0.30 <sup>h</sup>	10.89±0.30 <sup>f</sup>	12.58±0.08 <sup>f</sup>	15.20±0.177 <sup>d</sup>
HMT1.5-MW	2.79±0.00 <sup>cd</sup>	10.61±0.25 <sup>d</sup>	11.84±0.31 <sup>de</sup>	13.08±0.01 <sup>def</sup>	18.34±0.03 <sup>b</sup>
HMT4-MW	2.69±0.04 <sup>de</sup>	9.95±0.11 <sup>c</sup>	10.25±0.33 <sup>gh</sup>	12.85±0.20 <sup>def</sup>	16.39±0.06 <sup>c</sup>
HMT8-MW	2.42±0.02 <sup>g</sup>	9.45±0.11 <sup>efg</sup>	10.04±0.13 <sup>h</sup>	12.73±0.11 <sup>ef</sup>	16.81±0.4 <sup>c</sup>
HMT12-MW	2.41±0.08 <sup>g</sup>	8.99±0.32 <sup>gh</sup>	9.95±0.09 <sup>i</sup>	12.56±0.16 <sup>f</sup>	16.56±0.20 <sup>c</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

The leaching of soluble molecules of starch granules, such as amylose, sugars can be characterized by solubility. Modified potato starch showed generally higher solubility than native starch when the test temperature was  $>75^{\circ}\text{C}$ , and the solubility of starch samples increased respectively with the increase in test temperature from  $75^{\circ}\text{C}$  to  $95^{\circ}\text{C}$  (Table 4.2). This indicates that

temperature increase could induce the leaching of amylose or sugars from starch granules. MW, HMT, HMT combined with MW pre- and post-treatment could cause depolymerization of starch molecules, resulting in a higher ratio of shorts chains which had greater tendency to hydration than native starch molecules [7]. At the same test temperature, the solubility of HMT and HMT-MW starch increased with the length of heating time, which indicated that long time heat-moisture treatment could enhance the molecular depolymerization of starch. The MW, HMT and HMT combined with MW pre- and post-treatment can cause the weathering of starch granules, which consequently leads to the improvement of solubility [113]. However, high gelatinization temperature of modified starch could lead to low solubility at low test temperature. The modified starch had not been gelatinized at low temperature, and the soluble substances cannot be leached out from the starch granules, resulting in low solubility.

**Table 4.2**

## Solubility of native and modified potato starches

Sample	Solubility (%)				
	55°C	65°C	75°C	85°C	95°C
NS	4.29±0.44 <sup>a</sup>	4.69±0.31 <sup>a</sup>	2.11±0.17 <sup>j</sup>	2.70±0.17 <sup>i</sup>	7.70±0.31 <sup>g</sup>
MWS	3.59±0.47 <sup>b</sup>	3.24±0.21 <sup>b</sup>	2.77±0.27 <sup>i</sup>	5.64±0.63 <sup>f</sup>	7.27±0.13 <sup>g</sup>
MW-HMT1.5	0.77±0.06 <sup>g</sup>	3.29±0.07 <sup>b</sup>	2.73±0.07 <sup>i</sup>	5.94±0.33 <sup>f</sup>	8.70±0.30 <sup>f</sup>
MW-HMT4	1.53±0.15 <sup>f</sup>	3.18±0.11 <sup>b</sup>	2.98±0.03 <sup>i</sup>	6.70±0.24 <sup>e</sup>	9.94±0.37 <sup>e</sup>
MW-HMT8	2.37±0.02 <sup>de</sup>	3.09±0.06 <sup>bc</sup>	3.51±0.31 <sup>h</sup>	7.29±0.19 <sup>d</sup>	11.36±0.46 <sup>b</sup>
MW-HMT12	2.29±0.04 <sup>e</sup>	3.02±0.04 <sup>bc</sup>	2.32±0.13 <sup>j</sup>	7.18±0.05 <sup>de</sup>	13.54±0.02 <sup>b</sup>
HMT1.5	2.40±0.08 <sup>de</sup>	2.80±0.14 <sup>cd</sup>	5.63±0.15 <sup>f</sup>	4.59±0.11 <sup>g</sup>	7.35±0.30 <sup>g</sup>
HMT4	2.32±0.00 <sup>e</sup>	3.08±0.18 <sup>bc</sup>	6.91±0.25 <sup>d</sup>	4.70±0.15 <sup>g</sup>	11.28±0.31 <sup>b</sup>
HMT8	2.38±0.07 <sup>de</sup>	2.65±0.11 <sup>d</sup>	8.35±0.08 <sup>b</sup>	5.86±0.09 <sup>f</sup>	14.05±0.18 <sup>b</sup>
HMT12	2.66±0.11 <sup>cd</sup>	2.70±0.15 <sup>d</sup>	6.14±0.31 <sup>c</sup>	3.57±0.30 <sup>h</sup>	8.69±0.28 <sup>f</sup>
HMT1.5-MW	3.75±0.17 <sup>b</sup>	3.11±0.07 <sup>bc</sup>	3.01±0.13 <sup>i</sup>	7.14±0.47 <sup>de</sup>	8.43±0.39 <sup>f</sup>
HMT4-MW	2.94±0.06 <sup>c</sup>	3.09±0.08 <sup>bc</sup>	4.42±0.12 <sup>g</sup>	8.09±0.13 <sup>c</sup>	10.53±0.36 <sup>d</sup>
HMT8-MW	1.63±0.12 <sup>f</sup>	4.68±0.14 <sup>a</sup>	7.64±0.21 <sup>c</sup>	15.28±0.13 <sup>b</sup>	13.69±0.14 <sup>b</sup>
HMT12-MW	1.37±0.10 <sup>f</sup>	4.94±0.34 <sup>a</sup>	15.66±0.39 <sup>a</sup>	34.83±0.33 <sup>a</sup>	15.65±0.18 <sup>a</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

#### **4.2.2 Effects of heat-moisture treatment combined with microwave pre- and post- treatment on freeze-thaw stability of potato starch**

Starch with good freeze-thaw stability can be used as emulsifiers in food products such as sauce, yogurt, and jellies. High syneresis rate indicates low freeze-thaw stability of the food products [188]. The freeze-thaw stabilities of potato starch modified by heat-moisture treatment combined with microwave pre- and post- treatment during freeze-thaw cycles (1FTC, 2FTC, 3FTC, 4FTC, 5FTC) were shown in Table 4.3 with the percentage of syneresis as an index. The syneresis of all the starch pastes including NS increased with the increasing of freezing-thawing cycles. During the whole freezing-thawing cycles, there was no water separation from MWS samples. In addition, MW-HMT1.5 and HMT1.5 had lower syneresis rate than that of NS after two freezing-thawing cycles, all these results indicating that single MW, short-time single HMT and short-time HMT combined with MW pretreatment can enhance the repeated freeze-thaw stability of potato starch pastes. Similar to the results of section 3.2.2, the water separation of modified potato starch increased with the extension of HMT heating time, indicating that long- time HMT ( $\geq 4$ h) could weaken the freeze-thaw stability of potato starch. Moreover, both HMT combined with MW pretreatment and HMT combined with MW post-treatment had stronger weakening effects on the freeze-thaw stability of potato starch than that of long-time single HMT. The freeze-thaw stability of starch paste was not only positively correlated with the amylopectin and amylose ratio in starch [19], but also positively correlated with the chain length of starch molecules [189]. Therefore, from the increased syneresis rate of starch paste, it can be inferred that long- time single HMT, long- time single HMT combined with MW not only decreased the amylopectin and amylose ratio of potato starch, but also destroyed the molecular chains and the spatial structure of potato starch.



**Table 4.3****Freeze-thaw stability of native and modified potato starches**

Sample	Syneresis rate (%)				
	1FTC	2FTC	3FTC	4FTC	5FTC
NS	0.05±0.00 <sup>h</sup>	4.42±0.11 <sup>i</sup>	6.24±0.22 <sup>i</sup>	9.28±0.30 <sup>g</sup>	9.48±0.30 <sup>i</sup>
MWS	-	-	-	-	-
MW-HMT1.5	0.57±0.18 <sup>gh</sup>	2.37 ±0.10 <sup>k</sup>	3.47±0.12 <sup>k</sup>	4.49±0.01 <sup>h</sup>	5.13±0.38 <sup>j</sup>
MW-HMT4	4.25±0.44 <sup>f</sup>	5.61±0.07 <sup>h</sup>	8.60±0.23 <sup>gh</sup>	9.74±0.14 <sup>g</sup>	10.02 ±0.45 <sup>i</sup>
MW-HMT8	17.65±1.13 <sup>b</sup>	21.12±0.42 <sup>c</sup>	25.99±0.13 <sup>c</sup>	35.89±0.25 <sup>b</sup>	40.02±0.20 <sup>b</sup>
MW-HMT12	27.11±0.30 <sup>a</sup>	35.42±0.92 <sup>a</sup>	37.73±0.52 <sup>a</sup>	39.23±0.84 <sup>a</sup>	41.17±1.61 <sup>a</sup>
HMT1.5	0.24±0.04 <sup>gh</sup>	3.59±0.18 <sup>j</sup>	4.30±0.21 <sup>j</sup>	4.72±0.06 <sup>h</sup>	4.93±0.33 <sup>j</sup>
HMT4	0.95±0.05 <sup>g</sup>	3.23±0.16 <sup>j</sup>	8.74±0.21 <sup>g</sup>	17.20±0.18 <sup>f</sup>	20.18±0.47 <sup>g</sup>
HMT8	9.72±0.21 <sup>e</sup>	13.39±0.40 <sup>g</sup>	21.37±0.44 <sup>f</sup>	24.89±1.83 <sup>e</sup>	27.01±0.27 <sup>f</sup>
HMT12	10.84±0.49 <sup>d</sup>	15.44±0.11 <sup>f</sup>	33.75±0.33 <sup>b</sup>	34.26±0.07 <sup>c</sup>	36.90±0.11 <sup>c</sup>
HMT1.5-MW	0.99±0.04 <sup>g</sup>	4.33±0.33 <sup>i</sup>	7.88±0.16 <sup>h</sup>	9.74±0.41 <sup>g</sup>	13.31±0.31 <sup>h</sup>
HMT4-MW	14.47±0.63 <sup>c</sup>	16.50±0.27 <sup>e</sup>	23.03±0.13 <sup>e</sup>	25.99±0.10 <sup>e</sup>	28.12±0.29 <sup>e</sup>
HMT8-MW	15.20±0.18 <sup>c</sup>	18.48±0.07 <sup>d</sup>	24.99±1.01 <sup>d</sup>	29.15±0.95 <sup>d</sup>	32.34±0.46 <sup>d</sup>
HMT12-MW	27.42±01.2 <sup>a</sup>	31.41±0.28 <sup>b</sup>	33.63±0.05 <sup>b</sup>	35.91±0.37 <sup>b</sup>	37.28±0.35 <sup>c</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

### **4.2.3 Effect of heat-moisture treatment combined with microwave pre- and post- treatment on retrogradation of potato starch**

The retrogradation of NS and all the treated potato starches increased and tended to balance with the extension of storage time (Table 4.4). HMT heating time had great significant effect on the retrogradation of starch, all the HMT modified potato starch (including single HMT and HMT combined with MW pre- and post-treatment) with longer heating time showed stronger retrogradation characteristics. Dual modification of HMT and MW had greater effects on starch retrogradation than that of single HMT or single MW. Moreover, starch modified by HMT combined with MW pretreatment was more prone to aging than starch modified by HMT combined with MW post-treatment. Starch retrogradation refers to the process that involves the recombination of amylose and amylopectin chains into ordered structures and characterize the potential to reduce starch

digestibility [190]. The starch granules reformed double helices during retrogradation process, inhibiting catalysis and reducing the efficiency of digestive, thus reducing the digestion rate of cooked starch [191]. All these of this research results indicated that HMT combined with MW could enhance the content of slowly digestible starch and resistant starch, which was confirmed in the results of *in vitro* digestibility (Section 4.2.10), indicating that HMT and MW modified starch had huge potential application value in the daily diet of patients with insulin-independent diabetic.

**Table 4.4**

### Retrogradation of native and modified potato starches

Sample	Retrogradation (%)							
	2 h	4 h	6 h	8 h	10 h	12 h	14 h	16 h
NS	0.00±0.00 <sup>j</sup>	8.00±0.00 <sup>h</sup>	8.00±0.00 <sup>h</sup>	8.00±0.00 <sup>h</sup>	8.00±0.00 <sup>h</sup>	8.00±0.00 <sup>h</sup>	8.00±0.00 <sup>h</sup>	8.00±0.00 <sup>h</sup>
MW	28.00±3.46 <sup>h</sup>	42.93±1.85 <sup>f</sup>	45.33±0.46 <sup>f</sup>	46.27±0.23 <sup>f</sup>	47.20±0.00 <sup>f</sup>	47.60±0.00 <sup>f</sup>	48.00±0.00 <sup>f</sup>	48.00±0.00 <sup>f</sup>
MW-HMT1.5	48.00±0.00 <sup>f</sup>	53.73±0.46 <sup>e</sup>	55.20±0.69 <sup>e</sup>	55.73±0.46 <sup>e</sup>	55.87±0.23 <sup>e</sup>	56.27±0.23 <sup>e</sup>	56.27±0.23 <sup>e</sup>	56.27±0.23 <sup>e</sup>
MW-HMT4	57.33±1.15 <sup>d</sup>	61.73±0.92 <sup>d</sup>	62.27±1.15 <sup>d</sup>	62.67±1.15 <sup>d</sup>	62.80±1.39 <sup>d</sup>	62.93±1.62 <sup>d</sup>	62.93±1.62 <sup>d</sup>	62.93±1.62 <sup>d</sup>
MW-HMT8	72.53±0.46 <sup>b</sup>	73.20±0.40 <sup>b</sup>	73.60±0.40 <sup>b</sup>	73.73±0.46 <sup>b</sup>	73.73±0.46 <sup>b</sup>	73.73±0.46 <sup>b</sup>	73.73±0.46 <sup>b</sup>	73.73±0.46 <sup>b</sup>
MW-HMT12	76.67±0.61 <sup>a</sup>	77.07±0.23 <sup>a</sup>	77.47±0.23 <sup>a</sup>	77.47±0.23 <sup>a</sup>	77.47±0.23 <sup>a</sup>	77.47±0.23 <sup>a</sup>	77.47±0.23 <sup>a</sup>	77.47±0.23 <sup>a</sup>
HMT1.5	12.53±1.29 <sup>i</sup>	24.80±1.74 <sup>g</sup>	32.40±0.69 <sup>e</sup>	35.60±0.69 <sup>e</sup>	37.47±0.92 <sup>e</sup>	37.73±0.46 <sup>e</sup>	38.00±0.40 <sup>e</sup>	38.40±0.40 <sup>e</sup>
HMT4	53.07±0.46 <sup>c</sup>	59.73±0.46 <sup>d</sup>	60.27±0.23 <sup>d</sup>	60.67±0.23 <sup>d</sup>	60.80±0.40 <sup>d</sup>	60.93±0.23 <sup>d</sup>	60.93±0.23 <sup>d</sup>	60.93±0.23 <sup>d</sup>
HMT8	54.13±0.23 <sup>c</sup>	60.00±0.00 <sup>d</sup>	60.40±0.40 <sup>d</sup>	60.93±0.46 <sup>d</sup>	61.47±0.61 <sup>d</sup>	61.47±0.61 <sup>d</sup>	61.47±0.61 <sup>d</sup>	61.47±0.61 <sup>d</sup>
HMT12	66.53±0.92 <sup>c</sup>	68.27±0.46 <sup>c</sup>	68.80±0.80 <sup>c</sup>	68.93±0.61 <sup>c</sup>	69.20±0.80 <sup>c</sup>	69.20±0.80 <sup>c</sup>	69.20±0.80 <sup>c</sup>	69.20±0.80 <sup>c</sup>
HMT1.5-MW	44.00±0.00 <sup>g</sup>	53.33±0.23 <sup>c</sup>	55.03±0.77 <sup>e</sup>	55.47±0.23 <sup>c</sup>	55.87±0.23 <sup>c</sup>	56.00±0.00 <sup>c</sup>	56.00±0.00 <sup>c</sup>	56.00±0.00 <sup>c</sup>
HMT4-MW	57.07±0.92 <sup>d</sup>	61.60±0.69 <sup>d</sup>	62.13±0.23 <sup>d</sup>	62.53±0.61 <sup>d</sup>	62.53±0.61 <sup>d</sup>	62.67±0.83 <sup>d</sup>	62.67±0.83 <sup>d</sup>	62.80±1.06 <sup>d</sup>
HMT8-MW	72.27±0.46 <sup>b</sup>	72.80±0.69 <sup>b</sup>	73.07±0.83 <sup>b</sup>	73.20±0.80 <sup>b</sup>	73.20±0.80 <sup>b</sup>	73.20±0.80 <sup>b</sup>	73.20±0.80 <sup>b</sup>	73.20±0.80 <sup>b</sup>
HMT12-MW	73.07±1.01 <sup>b</sup>	73.47±0.92 <sup>b</sup>	73.47±0.92 <sup>b</sup>	73.73±1.15 <sup>d</sup>	73.73±1.15 <sup>b</sup>	73.73±1.15 <sup>b</sup>	73.73±1.15 <sup>b</sup>	73.87±1.29 <sup>b</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

#### 4.2.4 Effect of heat-moisture treatment combined with microwave pre- and post- treatment on transparency of potato starch

As showed in Table 4.5, The transparency of NS and all the treated potato starch decreased with the extension of storage time. Similar to the results of the retrogradation of starch, HMT heating time had great significant effect on starch transparency. The longer heating time, the lower transparency of starch after

gelatinization. Moreover, dual modification of HMT combined with MW had greater effect on the transparency of starch paste than that of single HMT and MW. After being modified by HMT and MW, the molecular structure of amylose and amylopectin chains was rearranged, and the starch granules underwent aggregation and recrystallization, resulting in an increase of amount of residual starch granules that were not expanded or completely broken during gelatinization process. Light refraction caused by the unexpanded starch granules and light refraction caused by the association between starch molecular chains effortlessly resulted in low transmittance of starch paste [192].

**Table 4.5**

Transparency of native and modified potato starches

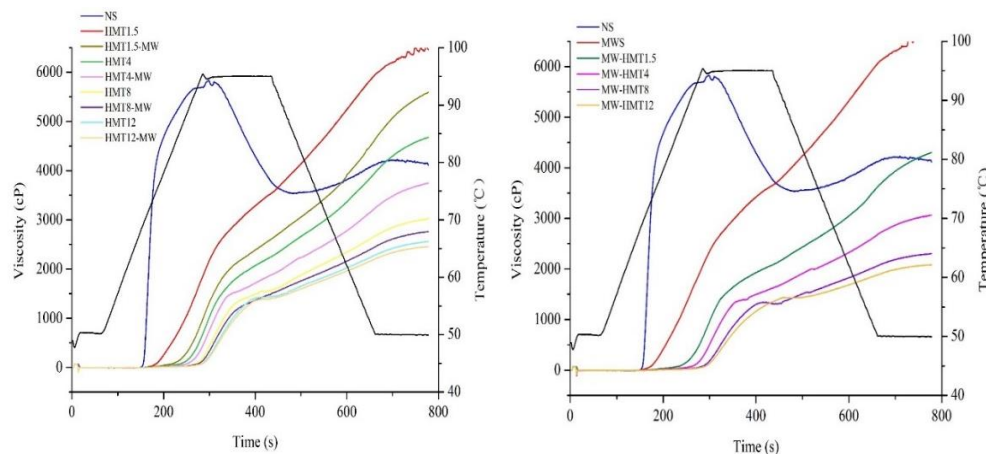
Sample	Transparency (%)					
	0 h	24 h	48 h	72 h	96 h	120 h
NS	28.70±0.78 <sup>a</sup>	21.97±0.06 <sup>a</sup>	15.43±0.25 <sup>a</sup>	13.73±0.21 <sup>a</sup>	10.23±0.25 <sup>a</sup>	12.63±0.12 <sup>a</sup>
MW	18.90±0.10 <sup>b</sup>	13.47±0.15 <sup>b</sup>	10.77±0.06 <sup>b</sup>	10.27±0.06 <sup>b</sup>	9.80±0.10 <sup>b</sup>	9.00±0.10 <sup>c</sup>
MW-HMT1.5	12.57±0.06 <sup>f</sup>	8.47±0.06 <sup>f</sup>	7.43±0.06 <sup>e</sup>	6.57±0.06 <sup>e</sup>	6.27±0.06 <sup>f</sup>	6.23±0.06 <sup>f</sup>
MW-HMT4	10.53±0.12 <sup>h</sup>	7.37±0.06 <sup>h</sup>	6.33±0.06 <sup>g</sup>	5.73±0.06 <sup>f</sup>	5.47±0.15 <sup>g</sup>	5.33±0.06 <sup>h</sup>
MW-HMT8	9.27±0.12 <sup>j</sup>	5.63±0.06 <sup>l</sup>	4.97±0.06 <sup>k</sup>	4.57±0.06 <sup>i</sup>	4.57±0.06 <sup>i</sup>	4.33±0.06 <sup>k</sup>
MW-HMT12	8.63±0.06 <sup>k</sup>	5.10±0.00 <sup>m</sup>	4.70±0.10 <sup>l</sup>	4.43±0.06 <sup>i</sup>	4.33±0.06 <sup>j</sup>	4.33±0.06 <sup>k</sup>
HMT1.5	18.03±0.23 <sup>c</sup>	12.57±0.06 <sup>c</sup>	10.83±0.06 <sup>b</sup>	10.40±0.10 <sup>b</sup>	9.57±0.06 <sup>c</sup>	9.40±0.10 <sup>b</sup>
HMT4	14.17±0.06 <sup>c</sup>	9.57±0.06 <sup>c</sup>	7.97±0.06 <sup>d</sup>	7.60±0.00 <sup>d</sup>	7.60±0.10 <sup>c</sup>	7.30±0.10 <sup>d</sup>
HMT8	11.00±0.10 <sup>e</sup>	6.47±0.12 <sup>j</sup>	5.47±0.06 <sup>i</sup>	5.33±0.06 <sup>g</sup>	4.73±0.06 <sup>i</sup>	4.70±0.10 <sup>j</sup>
HMT12	10.03±0.21 <sup>i</sup>	6.13±0.12 <sup>k</sup>	5.17±0.06 <sup>j</sup>	4.93±0.06 <sup>g</sup>	4.70±0.00 <sup>i</sup>	4.60±0.00 <sup>j</sup>
HMT1.5-MW	14.70±0.10 <sup>d</sup>	10.37±0.06 <sup>d</sup>	8.90±0.00 <sup>c</sup>	8.00±0.10 <sup>c</sup>	7.97±0.06 <sup>d</sup>	7.07±0.06 <sup>e</sup>
HMT4-MW	12.37±0.12 <sup>f</sup>	8.23±0.12 <sup>g</sup>	7.20±0.10 <sup>f</sup>	6.53±0.06 <sup>e</sup>	6.27±0.06 <sup>f</sup>	5.87±0.12 <sup>g</sup>
HMT8-MW	10.43±0.12 <sup>hi</sup>	6.70±0.10 <sup>i</sup>	5.87±0.06 <sup>h</sup>	5.70±0.10 <sup>f</sup>	5.33±0.06 <sup>g</sup>	4.97±0.06 <sup>i</sup>
HMT12-MW	9.60±0.10 <sup>j</sup>	6.27±0.06 <sup>k</sup>	5.47±0.12 <sup>i</sup>	5.27±0.12 <sup>g</sup>	4.93±0.06 <sup>h</sup>	4.87±0.12 <sup>i</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

#### 4.2.5 Effect of heat-moisture treatment combined with microwave pre- and post- treatment on pasting properties of potato starch

The pasting properties of native and modified potato starch are listed in Table 4.6 and the rapid viscosity analysis (RVA) pasting profiles are illustrated in Fig. 4.1. The RVA pasting profiles and the pasting properties of potato starch were significantly changed by single MW, single HMT and HMT combined with MW pre- and post-treatment. The peak viscosity of native potato starch was 6598 cP, which was higher than that of all the treated starch samples. The pasting temperature of native potato starch was 68.5°C, which was significantly increased to 73.5–93.0°C after MW and HMT except for the MWS (68.8°C) and the HMT1.5 (71.0°C). Higher pasting temperature indicated interactions between starch chains enhanced by MW and HMT, and more energy was required to destroy the enhanced starch structure during gelatinization process. All the MW-treated starch samples and HMT-treated starch samples showed lower peak viscosity, holding viscosity and breakdown viscosity than that of native potato starch. Similar results were reported for HMT mango kernel starch and MW millet starch [21, 193]. The decrease in peak viscosity of the samples may be attributed to the recombination and rearrangement in starch granules after MW, HMT, MW-HMT and HMT-MW, which limited the leaching of starch components from granules into the medium, consequently resulting in a decreased peak viscosity. Reduction in holding viscosity representing starch degradation upon application of high temperature and shear, indicated a decrease in peak viscosity [21, 101]. The low breakdown viscosity indicated high thermal stability, resistance development against shear exerted from heating and lower deterioration tendency [20]. Compared with native potato starch setback viscosity (496 cP), all the treated starch samples had higher setback viscosity ranging from 807 to 3168 cP, with the highest values noted for HMT1.5-MW and HMT1.5, and the lowest one for MW-HMT12 (Table 4.6). Additionally, the duration of HMT affected the pasting properties. Peak viscosity, hold viscosity and final viscosity of HMT modified starch decreased successively with the extension of HMT duration. Moreover, HMT combined with MW pre- and post-treatment resulted in lower peak viscosity

than that of starch modified by single MW or single HMT. Starch modified by HMT combined with MW pre-treatment had lower peak viscosity, hold viscosity and final viscosity than the starch modified by HMT combined with MW post-treatment or the starch modified by single HMT, indicating that MW-HMT starch had lower resistance to heat and shear than HMT-MW starch or HMT starch.



**Fig. 4.1** RVA pasting profiles of native and modified potato starches

**Table 4.6**

Pasting properties of native and modified potato starches

Samples	Pasting temperature (°C)	Peak viscosity (cP)	Hold viscosity (cP)	Final viscosity (cP)	Breakdown viscosity (cP)	Setback viscosity(cP)
NS	68.5±0.2 <sup>f</sup>	6598±73 <sup>a</sup>	4072±25 <sup>a</sup>	4567±37 <sup>c</sup>	2526.3±47.3 <sup>a</sup>	496±12 <sup>j</sup>
MWS	68.8±0.1 <sup>f</sup>	3662±27 <sup>b</sup>	3597±31 <sup>b</sup>	6644±13 <sup>a</sup>	65.7±3.5 <sup>bc</sup>	3047±42 <sup>b</sup>
MW-HMT1.5	80.8±1.5 <sup>bc</sup>	2145±9 <sup>e</sup>	2090±9 <sup>e</sup>	4559±88 <sup>c</sup>	54.3±1.5 <sup>bcde</sup>	2439±39 <sup>c</sup>
MW-HMT4	88.50±1.0 <sup>a</sup>	1591±22 <sup>g</sup>	1548±18 <sup>g</sup>	3027±56 <sup>e</sup>	42.3±3.8 <sup>cdef</sup>	1479±40 <sup>e</sup>
MW-HMT8	92.9±2.0 <sup>a</sup>	1339±6 <sup>jk</sup>	1309±2 <sup>k</sup>	2290±26 <sup>h</sup>	30.3±6.4 <sup>ef</sup>	985±50 <sup>h</sup>
MW-HMT12	93.0±0.6 <sup>a</sup>	1315±18 <sup>k</sup>	1256±19 <sup>l</sup>	2064±18 <sup>i</sup>	58.3±1.5 <sup>bcd</sup>	807±4 <sup>i</sup>
HMT1.5	71.0±0.4 <sup>ef</sup>	3522±8 <sup>c</sup>	3443±11 <sup>c</sup>	6553±88 <sup>a</sup>	78.3±3.5 <sup>b</sup>	3110±79 <sup>ab</sup>
HMT4	80.5±5.6 <sup>bc</sup>	2173±58 <sup>e</sup>	2111±56 <sup>e</sup>	4528±150 <sup>c</sup>	61.7±2.1 <sup>bcd</sup>	2417±98 <sup>c</sup>
HMT8	76.5±1.5 <sup>cd</sup>	1582±21 <sup>g</sup>	1545±18 <sup>g</sup>	3019±14 <sup>e</sup>	38.0±5.6 <sup>def</sup>	1474±17 <sup>e</sup>
HMT12	76.8±0.4 <sup>cd</sup>	1433±2 <sup>hi</sup>	1416±16 <sup>i</sup>	2573± 23 <sup>g</sup>	17.3±14.6 <sup>f</sup>	1157±27 <sup>g</sup>
HMT1.5-MW	73.5±2.7 <sup>de</sup>	2591±24 <sup>d</sup>	2516±23 <sup>d</sup>	5684±91 <sup>b</sup>	75.3±2.1 <sup>b</sup>	3168±102 <sup>a</sup>
HMT4-MW	81.9±5.3 <sup>b</sup>	1854±16 <sup>f</sup>	1799±15 <sup>f</sup>	3696±48 <sup>d</sup>	55.0±2.0 <sup>bcde</sup>	1936±50 <sup>d</sup>
HMT8-MW	75.5±2.0 <sup>de</sup>	1475±58 <sup>h</sup>	1457±36 <sup>g</sup>	28033±97 <sup>f</sup>	55.0±8.9 <sup>bcde</sup>	1345±67 <sup>f</sup>
HMT12-MW	82.4±3.7 <sup>b</sup>	1392±8 <sup>ij</sup>	1373±13 <sup>j</sup>	2465±9 <sup>g</sup>	18.7±11.6 <sup>f</sup>	1092±22 <sup>g</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

#### **4.2.6 Effect of heat-moisture treatment combined with microwave pre- and post- treatment on textural properties of potato starch paste**

The volume fraction and rigidity of gelatinized starch particles, the rheological properties of the continuous phase, as well as the interactions between the dispersed and continuous phase of gels determine the mechanical properties of starch gels [194]. These were significantly affected by HMT and MW, leading to obvious changes in gel texture. Texture, especially the hardness, is an important quality attribute affecting the palatability, mouthfeel, and swallowing characteristics of food [195]. A high hardness value indicates a harder structure and more chewable quality of food [196].

From Table 4.7, it is clear that single MW modified potato starch gel (MWS) and short-time HMT modified starch gels (HMT1.5, MW-HMT1.5, HMT-MW1.5) had higher hardness value than that of NS and the other treated samples, which was consistent with the results of Hongwei Cao [65] using appropriate MW to improve the gel hardness of quinoa starch and Rungarun Hormdok [170] using HMT to improve the gel hardness of rice starch, indicating that appropriate MW and HMT modification could increase gel hardness of potato starch. The increased hardness of single MW modified potato starch gels (Table 4.7, MWS) and short-time single HMT modified potato starch gels (Table 4.7, HMT1.5, MW-HMT1.5, HMT-MW1.5) could attributed to the increased cross-linking between starch chains in the particular amylose portion during HMT and MW process. Trends for HMT modified potato starch gels were observed that hardness increased for some extent and then gradually decreased with the extension of treatment time, which was consistent with the results of Rungarun Hormdok [170] that long time HMT led to low hardness value. This was probably due to partial gelatinization in starch granules at long time HMT, resulting in the collapse of some starch structure and ultimately resulting in a less rigid starch gel.

The hardness, cohesiveness, gumminess and chewiness of all the HMT modified potato starch gel (including single HMT, HMT combined with MW)

decreased with the extension of heating time (Table 4.7). The HMT potato starch pretreated by MW had higher hardness value than that of HMT potato starch post-treated by MW, which indicated that the processing sequence of dual modification of HMT and MW also affected the textural properties of the treated potato gels.

**Table 4.7**

**Pasting properties of native and modified potato starches**

Starch gel	Hardness (g)	Springiness (mm)	Cohesiveness (-)	Gumminess (g)	Chewiness (g·mm)	Resilience (-)
NS	2705.55±3.11 <sup>d</sup>	0.83±0.01 <sup>a</sup>	0.64±0.00 <sup>a</sup>	1700.43±11.44 <sup>d</sup>	1403.77±29.88 <sup>c</sup>	0.40±0.03 <sup>b</sup>
MWS	4645.49±33.14 <sup>a</sup>	0.81±0.00 <sup>a</sup>	0.49±0.00 <sup>de</sup>	2254.68±31.72 <sup>a</sup>	1821.85±33.03 <sup>a</sup>	0.36±0.01 <sup>b</sup>
MW-HMT1.5	2917.43±22.16 <sup>c</sup>	0.63±0.00 <sup>de</sup>	0.62±0.00 <sup>a</sup>	1810.32±28.19 <sup>c</sup>	1139.57±13.91 <sup>d</sup>	0.48±0.01 <sup>a</sup>
MW-HMT4	2550.09±55.74 <sup>c</sup>	0.56±0.00 <sup>f</sup>	0.61±0.00 <sup>b</sup>	1545.24±22.96 <sup>c</sup>	858.34±7.19 <sup>f</sup>	0.46±0.01 <sup>a</sup>
MW-HMT8	2269.93±33.04 <sup>g</sup>	0.63±0.01 <sup>de</sup>	0.34±0.01 <sup>h</sup>	761.69±28.74 <sup>k</sup>	480.09±10.04 <sup>j</sup>	0.16±0.01 <sup>d</sup>
MW-HMT12	1990.54±0.25 <sup>i</sup>	0.50±0.00 <sup>g</sup>	0.28±0.00 <sup>j</sup>	564.32±1.34 <sup>m</sup>	284.14±3.47 <sup>k</sup>	0.12±0.00 <sup>de</sup>
HMT1.5	4393.92±6.67 <sup>b</sup>	0.81±0.00 <sup>a</sup>	0.50±0.00 <sup>d</sup>	2177.20±18.79 <sup>b</sup>	1771.14±13.75 <sup>b</sup>	0.36±0.01 <sup>b</sup>
HMT4	2702.25±4.81 <sup>d</sup>	0.77±0.03 <sup>b</sup>	0.47±0.00 <sup>f</sup>	1267.37±13.72 <sup>g</sup>	981.16±51.84 <sup>e</sup>	0.36±0.02 <sup>b</sup>
HMT8	2323.64 ±3.18 <sup>fg</sup>	0.65±0.00 <sup>d</sup>	0.36±0.001 <sup>g</sup>	841.15±11.99 <sup>j</sup>	547.56±4.24 <sup>hi</sup>	0.16±0.00 <sup>d</sup>
HMT12	1778.86±18.91 <sup>k</sup>	0.81±0.01 <sup>a</sup>	0.35±0.00 <sup>g</sup>	630.64±12.99 <sup>l</sup>	512.04±6.98 <sup>ij</sup>	0.10±0.00 <sup>e</sup>
HMT1.5-MW	2755.19±6.93 <sup>d</sup>	0.68±0.02 <sup>c</sup>	0.52±0.00 <sup>c</sup>	1418.91±8.12 <sup>f</sup>	969.92±38.66 <sup>e</sup>	0.37±0.03 <sup>b</sup>
HMT4-MW	2346.49±24.26 <sup>c</sup>	0.62±0.01 <sup>c</sup>	0.51±0.00 <sup>c</sup>	1190.89±20.61 <sup>h</sup>	740.63±1.03 <sup>g</sup>	0.30±0.01 <sup>c</sup>
HMT8-MW	2054.64±5.07 <sup>h</sup>	0.58±0.00 <sup>f</sup>	0.48±0.01 <sup>ef</sup>	978.03±22.75 <sup>i</sup>	566.83±18.03 <sup>h</sup>	0.29±0.03 <sup>c</sup>
HMT12-MW	1848.64±51.41 <sup>j</sup>	0.52±0.00 <sup>g</sup>	0.32±0.00 <sup>i</sup>	598.89±11.43 <sup>lm</sup>	311.13±7.21 <sup>k</sup>	0.14±0.04 <sup>de</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

#### 4.2.7 Effect of heat-moisture treatment combined with microwave pre- and post- treatment on particle size distributions of potato starch granules

The variation of particle size could reflect the changes in agglomeration of the starch before and after modification. The particle size distribution parameters of native and treated potato starch were showed in Table 4.8. There was no obvious trend in the influence of HMT heating time and the processing sequence of dual modification of HMT and MW on the particle size distribution of starch granules, but in general, after being modified by single MW, single HMT and dual modification of HMT combined with MW, the particle size of starch granules could be increased. The D50, D (4,3) and D (3,2) of all treated starch were higher than NS, while the value of S.S.A. was significantly decreased by MW and HMT,

which was agreed with precious studies about HMT treated lily starch [95] MW treated waxy hull-less barley starch [197]. The reason for the large particle size of MW and HMT modified starch was that the internal temperature of the granules rose rapidly during the MW and HMT treatment, and the internal pressure increased, causing the starch granules to expand, resulting in partial gelatinization and agglomeration of the granules[95, 197].

**Table 4.8**

The particle size distribution of native and modified potato starches

Samples	D50( $\mu\text{m}$ )	D (4,3) ( $\mu\text{m}$ )	D (3,2) ( $\mu\text{m}$ )	S.S.A.( $\text{m}^2/\text{kg}$ )
NS	34.12 $\pm$ 0.40 <sup>c</sup>	36.60 $\pm$ 0.38 <sup>f</sup>	22.24 $\pm$ 0.56 <sup>h</sup>	99.93 $\pm$ 2.54 <sup>a</sup>
MWS	34.29 $\pm$ 0.16 <sup>bc</sup>	36.97 $\pm$ 0.07 <sup>cdef</sup>	24.06 $\pm$ 0.07 <sup>g</sup>	92.35 $\pm$ 0.26 <sup>b</sup>
MW-HMT1.5	34.38 $\pm$ 0.09 <sup>bc</sup>	37.24 $\pm$ 0.21 <sup>bedef</sup>	29.67 $\pm$ 0.12 <sup>def</sup>	74.87 $\pm$ 0.32 <sup>cd</sup>
MW-HMT4	34.53 $\pm$ 0.03 <sup>abc</sup>	37.35 $\pm$ 0.08 <sup>abcde</sup>	30.51 $\pm$ 0.02 <sup>abc</sup>	72.83 $\pm$ 0.06 <sup>de</sup>
MW-HMT8	34.91 $\pm$ 0.27 <sup>ab</sup>	37.79 $\pm$ 0.26 <sup>ab</sup>	30.88 $\pm$ 0.23 <sup>a</sup>	71.96 $\pm$ 0.53 <sup>e</sup>
MW-HMT12	35.14 $\pm$ 0.68 <sup>a</sup>	37.95 $\pm$ 0.65 <sup>a</sup>	30.76 $\pm$ 0.56 <sup>ab</sup>	72.24 $\pm$ 1.31 <sup>e</sup>
HMT1.5	34.61 $\pm$ 0.28 <sup>abc</sup>	37.48 $\pm$ 0.19 <sup>abcd</sup>	30.28 $\pm$ 0.17 <sup>abcd</sup>	73.38 $\pm$ 0.41 <sup>cde</sup>
HMT4	34.82 $\pm$ 0.21 <sup>ab</sup>	37.52 $\pm$ 0.22 <sup>abc</sup>	29.51 $\pm$ 0.23 <sup>ef</sup>	75.29 $\pm$ 0.59 <sup>e</sup>
HMT8	34.73 $\pm$ 0.49 <sup>abc</sup>	37.33 $\pm$ 0.67 <sup>abcde</sup>	29.35 $\pm$ 0.25 <sup>f</sup>	75.71 $\pm$ 0.65 <sup>e</sup>
HMT12	34.64 $\pm$ 0.23 <sup>abc</sup>	37.42 $\pm$ 0.17 <sup>abcde</sup>	29.96 $\pm$ 0.44 <sup>cdef</sup>	74.18 $\pm$ 1.08 <sup>cde</sup>
HMT1.5-MW	34.36 $\pm$ 0.13 <sup>bc</sup>	37.03 $\pm$ 0.18 <sup>cdef</sup>	23.97 $\pm$ 0.42 <sup>g</sup>	92.72 $\pm$ 1.60 <sup>b</sup>
HMT4-MW	34.29 $\pm$ 0.19 <sup>bc</sup>	37.00 $\pm$ 0.07 <sup>cdef</sup>	29.41 $\pm$ 0.24 <sup>ef</sup>	75.55 $\pm$ 0.62 <sup>e</sup>
HMT8-MW	34.32 $\pm$ 0.11 <sup>bc</sup>	36.82 $\pm$ 0.09 <sup>def</sup>	30.11 $\pm$ 0.23 <sup>bcd</sup>	73.63 $\pm$ 0.8 <sup>4cde</sup>
HMT12-MW	34.54 $\pm$ 0.07 <sup>abc</sup>	36.75 $\pm$ 0.09 <sup>ef</sup>	30.27 $\pm$ 0.46 <sup>abcd</sup>	73.41 $\pm$ 1.10 <sup>cde</sup>

**Notes:** all values are the means of triplicate determinations  $\pm$  standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

#### 4.2.8 Effect of heat-moisture treatment combined with microwave pre- and post- treatment on color characteristics of potato starch

The color values of the native potato starch (as control) and treated starch were showed in Table 4.9. In general, HMT caused a slight increase of lightness ( $L^*$  values), while single MW treatment caused a slight, although significant, decrease of lightness, indicating that the color of all the HMT treated samples became brighter and the color of the single MW treated sample (MWS) became



darker. The  $a^*$  values of treated samples varied significantly in different ways depending on the treated ways. Although there was no significant difference of  $a^*$  values in starch samples with the same treated method, the color of MW- HMT samples became more reddish, while MWS, HMT and HMT-MW samples became more greenish. As can be seen from Table 4.9, all the treated samples became more yellowish, as significant increase of  $b^*$  values were obtained for treated samples. Although there were significant differences of the difference of color ( $\Delta E$ ), it could conclude that all treatments did not markedly change potato starch color from the analysis of  $\Delta E$ , given that  $\Delta E$  was always below 3, indicating that color differences was no visible differentiated of all the treated starch samples [198-199].

**Table 4.9**

The color characteristics of native and treated potato starch samples

Samples	$L^*$	$a^*$	$b^*$	$\Delta E$
NS	99.38±0.05 <sup>cd</sup>	4.80±0.01 <sup>bcd</sup>	-2.38±0.01 <sup>i</sup>	-
MWS	98.71±0.02 <sup>c</sup>	4.73±0.03 <sup>s</sup>	-2.21±0.03 <sup>h</sup>	0.70±0.02 <sup>c</sup>
MW-HMT1.5	99.31±0.24 <sup>d</sup>	4.81±0.03 <sup>abc</sup>	-2.02±0.06 <sup>f</sup>	0.42±0.05 <sup>f</sup>
MW-HMT4	99.46±0.20 <sup>bcd</sup>	4.84±0.02 <sup>a</sup>	-1.94±0.05 <sup>e</sup>	0.48±0.03 <sup>c</sup>
MW-HMT8	99.51±0.20 <sup>abcd</sup>	4.83±0.01 <sup>ab</sup>	-1.71±0.03 <sup>bc</sup>	0.70±0.02 <sup>c</sup>
MW-HMT12	99.54±0.06 <sup>abcd</sup>	4.80±0.03 <sup>abcd</sup>	-1.60±0.01 <sup>a</sup>	0.80±0.02 <sup>a</sup>
HMT1.5	99.38±0.07 <sup>cd</sup>	4.76±0.03 <sup>defg</sup>	-2.15±0.02 <sup>s</sup>	0.24±0.02 <sup>s</sup>
HMT4	99.59±0.02 <sup>abc</sup>	4.77±0.02 <sup>cdef</sup>	-2.03±0.02 <sup>f</sup>	0.41±0.01 <sup>f</sup>
HMT8	99.49±0.12 <sup>abcd</sup>	4.78±0.02 <sup>cde</sup>	-1.85±0.01 <sup>d</sup>	0.55±0.03 <sup>d</sup>
HMT12	99.66±0.08 <sup>ab</sup>	4.75±0.04 <sup>efg</sup>	-1.68±0.04 <sup>b</sup>	0.76±0.03 <sup>ab</sup>
HMT1.5-MW	99.70±0.05 <sup>a</sup>	4.74±0.01 <sup>fg</sup>	-2.14±0.01 <sup>s</sup>	0.41±0.03 <sup>f</sup>
HMT4-MW	99.50±0.05 <sup>abcd</sup>	4.75±0.01 <sup>efg</sup>	-1.85±0.01 <sup>d</sup>	0.55±0.02 <sup>d</sup>
HMT8-MW	99.68±0.13 <sup>ab</sup>	4.77±0.01 <sup>defg</sup>	-1.89±0.01 <sup>d</sup>	0.58±0.06 <sup>d</sup>
HMT12-MW	99.70±0.11 <sup>a</sup>	4.77±0.03 <sup>def</sup>	-1.74±0.02 <sup>c</sup>	0.72±0.05 <sup>bc</sup>

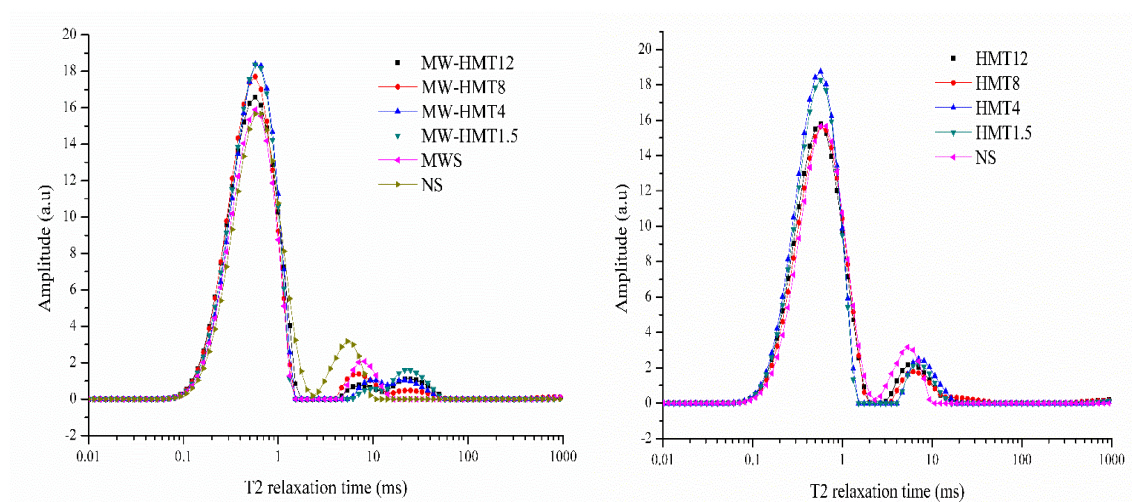
**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

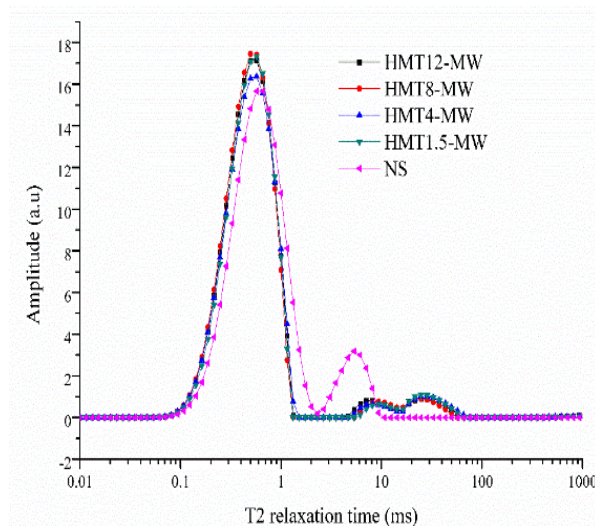
#### 4.2.9 Effect of heat-moisture treatment combined with microwave pre- and post- treatment on water distribution of potato starch

In starch-based foods, the physical properties of water can significantly affect the processing quality of starch. LF-NMR technology is the most effective technology for evaluating the distribution and state of water in the starch-water system. Relaxation time ( $T_2$ ) is the time required for an excited spin-spin proton to reach dynamic equilibrium after energy exchange with adjacent protons, reflects the difference in the degrees of freedom of water. The less bounded hydrogen protons or higher degree of freedom is associated with longer transverse relaxation time, while the more bounded hydrogen protons or lower degree of freedom corresponds to a shorter transverse relaxation time [200-202].

As shown in Fig. 4.2 and Table 4.10, three peaks were observed in  $T_2$  of MW treated starch (MWS, MW-HMT and HMT-MW), while two peaks were observed in  $T_2$  of NS and HMT samples. The first peak  $T_{21}$  (0.01-1 ms) corresponded to bound water, which interacted with the surface of amylose and amylopectin chains [203]. It can be seen from Table 3 that the MW and HMT treatments of potato starch caused the  $T_{21}$  shifted toward faster relaxation times compared with native starch (NS), indicating that MW and HMT treatments enhanced the starch-water interaction and self-binding of starch, and consequently resulting in lower mobility of water protons. The second peak  $T_{22}$  (1-20 ms) corresponded to “semi-crystalline lamellae water” [203], which existed in the narrow space between crystals. Due to different spaces, the water in them exhibited different mobilities. Peak of NS was closer to the left than treated starch; they were followed by the single HMT treated starch (HMT1.5, HMT4, HMT8 and HMT12). The peaks  $T_{22}$  of MW treated starch were close to the right and connected with its next peak. The third peak  $T_{23}$  was located in  $T_2$  between 20 ms and 100 ms was attributed to immobilized water, which was regarded as water inside the hexagonal channels of B-type crystal clusters and was defined as “channel water in B-type crystal” by a previous study [204]. It can be observed from Fig. 4.2, the native and HMT treated starch did not have channel water in B-type crystal, while all the MW treated starch contained a certain amount of channel water in B-type crystal.

The proportion of different state water in native and treated starch were summarized in Table 4.10. The areas under different component represented the proportion of different state water distribution, namely  $PT_{21}$  (bound water),  $PT_{22}$  (semi-crystalline lamellae water),  $PT_{23}$  (channel water in B-type crystal), respectively. Although the bound water in all starch samples was the main water which at least accounted for 90%, the MW treated starch had three different state water, NS and single HMT treated starch only had two different state water. There were significant differences of  $PT_{21}$  and  $PT_{22}$  between NS and all treated starch, NS had the lowest  $PT_{21}$  but highest  $PT_{22}$ , indicating MW and HMT treatments could change the water distribution and improve the interaction between starch and water. No significant differences of  $PT_{21}$  were observed between MW treated starch and single HMT treated, but all MW treated starch had lower  $PT_{22}$  than single HMT treated starch; furthermore, all the MW treated starch contained a certain amount of channel water in B-type crystal ( $PT_{23}$ ). These results indicated that MW treatment could result in a certain level of semi-crystalline lamellae water shifting to channel water in B-type crystal.





**Fig.4.2** Water distribution of native and modified potato starches

**Table 4.10**

Relaxation times ( $T_2$ ) and corresponding peak areas percentages of water from native and modified starch

Samples	Relaxation Time (ms)			Proportion of water in different state (%)		
	$T_{21}$	$T_{22}$	$T_{23}$	$PT_{21}$	$PT_{22}$	$PT_{23}$
NS	$0.62 \pm 0.06^a$	$5.74 \pm 0.57^b$	-	$90.48 \pm 0.98^c$	$9.52 \pm 0.98^a$	-
MWS	$0.59 \pm 0.01^{ab}$	$8.43 \pm 3.24^{ab}$	$30.61 \pm 3.02^a$	$92.56 \pm 0.80^{ab}$	$1.14 \pm 0.06^c$	$6.29 \pm 0.74^a$
MW-HMT1.5	$0.57 \pm 0.00^{ab}$	$7.73 \pm 2.26^{ab}$	$25.01 \pm 4.90^a$	$93.06 \pm 0.04^{ab}$	$1.58 \pm 0.39^{dc}$	$5.36 \pm 0.35^{ab}$
MW-HMT4	$0.60 \pm 0.05^{ab}$	$8.52 \pm 0.70^{ab}$	$24.71 \pm 2.74^b$	$93.39 \pm 0.52^{ab}$	$3.92 \pm 0.88^c$	$2.69 \pm 0.72^c$
MW-HMT8	$0.57 \pm 0.00^{ab}$	$7.60 \pm 0.65^{ab}$	$24.66 \pm 0.16^a$	$93.11 \pm 0.75^{ab}$	$3.57 \pm 1.42^c$	$3.33 \pm 2.17^{bc}$
MW-HMT12	$0.57 \pm 0.00^{ab}$	$8.06 \pm 0.00^{ab}$	$25.76 \pm 1.40^a$	$93.57 \pm 0.30^{ab}$	$2.85 \pm 0.64^{cd}$	$3.57 \pm 0.94^{bc}$
HMT1.5	$0.57 \pm 0.00^{ab}$	$7.10 \pm 0.99^{ab}$	-	$93.32 \pm 0.18^{ab}$	$6.68 \pm 0.18^b$	-
HMT4	$0.57 \pm 0.00^{ab}$	$6.75 \pm 0.53^{ab}$	-	$92.59 \pm 0.26^{ab}$	$7.41 \pm 0.26^b$	-
HMT8	$0.57 \pm 0.00^{ab}$	$6.44 \pm 0.53^{ab}$	-	$93.31 \pm 1.33^{ab}$	$6.69 \pm 1.33^b$	-
HMT12	$0.57 \pm 0.00^{ab}$	$6.60 \pm 0.65^{ab}$	-	$92.37 \pm 0.94^b$	$7.63 \pm 0.94^b$	-
HMT1.5-MW	$0.57 \pm 0.00^{ab}$	$8.72 \pm 0.86^a$	$24.77 \pm 0.00^a$	$93.61 \pm 0.29^{ab}$	$2.32 \pm 0.26^{cde}$	$4.07 \pm 0.04^{abc}$
HMT4-MW	$0.54 \pm 0.05^b$	$8.72 \pm 0.86^a$	$28.61 \pm 0.18^a$	$93.97 \pm 0.53^a$	$2.99 \pm 1.13^{cd}$	$3.05 \pm 1.66^{bc}$
HMT8-MW	$0.54 \pm 0.05^b$	$7.73 \pm 2.26^{ab}$	$28.76 \pm 5.64^a$	$93.77 \pm 0.88^{ab}$	$2.52 \pm 0.13^{cde}$	$3.71 \pm 1.01^{bc}$
HMT12-MW	$0.54 \pm 0.04^b$	$8.52 \pm 0.70^{ab}$	$27.33 \pm 2.22^a$	$93.77 \pm 0.14^{ab}$	$3.19 \pm 0.40^{cd}$	$3.04 \pm 0.41^{bc}$

**Notes:** all values are the means of triplicate determinations  $\pm$  standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

#### 4.2.10 Effect of heat-moisture treatment combined with microwave pre- and post- treatment on *in vitro* digestion of potato starch

Based on the rate and degree of *in vitro* digestion, starch is commonly classified into three categories: RDS, SDS and RS, among which SDS and RS are considered to be beneficial to human health [205]. The contents of RDS, SDS and RS in native potato starch were 32.14%, 55.17% and 13.69%, respectively (Table 4.11), which were inconsistent with the results of previous research reported by Wang *et al.*[109] showing RDS, SDS and RS contents in native potato starch at 22.5%, 12.8% and 64.7% respectively. The unusual results of RDS, SDS and RS contents in native potato starch might be attributed to the method of *in vitro* digestion. Gelatinized and non-gelatinized starch samples showed different *in vitro* digestibility, which was confirmed by Ji & Yu [94] and by Piecyk & Domian [206], while the results of Chen's [207] research directly indicated that gelatinized and non-gelatinized potato starch showed different contents of RDS, SDS and RS. The gelatinized starch showed higher RDS content than non-gelatinized starch for their granular structure had been already disrupted during the cooking process and thus became more susceptible to enzymatic hydrolysis [95].

The RDS content of all modified potato starches was significantly ( $p<0.5$ ) lower than that of native potato starch, while the RS content was generally higher (Table 4.11). When the heating time of HMT was less than 12 h, the RS content increased successively with the prolongation of heat-moisture treatment time. Previous studies have reported that HMT could increase the content of RS and the total content of SDS and RS in maize starch[142] and sweet potato starch [208], while MW also increased the content of RS and the total content of SDS and RS in *Canna edulis* Ker starch [124] and debranched mung-bean starch [16]. Amylopectin in starch was partly degraded, the hydrogen bonds between starch molecules were broken, and the molecular chains in starch granules were separated by microwave heating, all these resulting in swelling and gelatinization of starch particles. HMT further rearranged the molecular structures, transformed some RDS fractions into SDS and/or RS fractions and increased the resistance of starch to enzymatic hydrolysis [80, 115]. Furthermore, dual modification of HMT

and MW increased the total content of SDS and RS but decreased the content of RDS in potato starch (Table 4.11). It was worth mentioning that under the same HMT heating time, the RS content of HMT-MW starch was higher than that of HMT starch, *e.g.*, the RS content of HMT1.5-MW (19.05%) was higher than that of HMT1.5 (14.02%). Additionally, HMT-MW starch also had a higher RS content than that of MW-HMT starch under the same HMT heating time, *e.g.*, the RS content of HMT1.5-MW (19.05%) was higher than that of MW-HMT1.5 (15.86%). All these results indicated that dual starch modification via HMT and MW had greater effects on starch digestion than single MW or HMT, and the sequence of dual modification also affected starch digestibility.

**Table 4.11**

Fourier transform infrared spectroscopy intensity ratios, relative crystallinity and digestibility properties of native and modified potato starches

Starch	1047/1022 (cm <sup>-1</sup> )	1047/1035 (cm <sup>-1</sup> )	Relative crystallinity (%)	Digestibility properties		
				RDS (%)	SDS (%)	RS (%)
NS	1.0361±0.0001 <sup>a</sup>	1.0083±0.0001 <sup>a</sup>	19.39±0.04 <sup>a</sup>	31.14±0.10 <sup>a</sup>	55.17±0.17 <sup>bc</sup>	13.69±0.10 <sup>h</sup>
MWS	1.0150±0.0001 <sup>b</sup>	0.9992±0.0000 <sup>c</sup>	15.35±0.07 <sup>b</sup>	29.14±0.10 <sup>dc</sup>	55.90±0.37 <sup>b</sup>	14.97±0.43 <sup>fg</sup>
MW-HMT1.5	1.0099±0.0001 <sup>k</sup>	0.9965±0.0001 <sup>i</sup>	15.55±0.00 <sup>fg</sup>	28.91±0.10 <sup>ef</sup>	55.23±0.34 <sup>bc</sup>	15.86±0.38 <sup>f</sup>
MW-HMT4	1.0100±0.0002 <sup>k</sup>	0.9957±0.0000 <sup>j</sup>	15.65±0.02 <sup>c</sup>	28.43±0.10 <sup>gh</sup>	52.17±0.09 <sup>d</sup>	19.40±0.16 <sup>c</sup>
MW-HMT8	1.0180±0.0001 <sup>f</sup>	0.9980±0.0002 <sup>f</sup>	15.74±0.01 <sup>d</sup>	27.66±0.60 <sup>i</sup>	50.05±1.02 <sup>f</sup>	22.29±0.53 <sup>c</sup>
MW-HMT12	1.0176±0.0000 <sup>g</sup>	0.9978±0.0001 <sup>g</sup>	15.83±0.01 <sup>c</sup>	28.54±0.10 <sup>fg</sup>	57.25±0.68 <sup>a</sup>	14.21±0.78 <sup>gh</sup>
HMT1.5	1.0268±0.0000 <sup>b</sup>	1.0008±0.0000 <sup>c</sup>	18.17±0.04 <sup>b</sup>	28.11±0.31 <sup>h</sup>	57.87±0.35 <sup>a</sup>	14.02±0.38 <sup>gh</sup>
HMT4	1.0232±0.0002 <sup>c</sup>	1.0002±0.0002 <sup>d</sup>	15.80±0.06 <sup>cd</sup>	29.41±0.10 <sup>d</sup>	54.57±0.44 <sup>c</sup>	16.02±0.42 <sup>f</sup>
HMT8	1.0217±0.0000 <sup>d</sup>	0.9992±0.0001 <sup>c</sup>	15.60±0.02 <sup>ef</sup>	29.80±0.20 <sup>c</sup>	47.36±0.93 <sup>g</sup>	22.85±0.72 <sup>c</sup>
HMT12	1.0206±0.0000 <sup>e</sup>	0.9981±0.0001 <sup>f</sup>	15.17±0.04 <sup>i</sup>	30.23±0.10 <sup>b</sup>	47.33±1.20 <sup>g</sup>	22.44±1.10 <sup>c</sup>
HMT1.5-MW	1.0235±0.0000 <sup>c</sup>	1.0042±0.0000 <sup>b</sup>	15.64±0.03 <sup>c</sup>	29.52±0.10 <sup>cd</sup>	51.43±0.01 <sup>de</sup>	19.05±0.09 <sup>e</sup>
HMT4-MW	1.0205±0.0003 <sup>c</sup>	1.0003±0.0000 <sup>d</sup>	15.50±0.01 <sup>g</sup>	28.16±0.10 <sup>gh</sup>	50.75±1.37 <sup>ef</sup>	21.09±1.47 <sup>d</sup>
HMT8-MW	1.0135±0.0000 <sup>i</sup>	0.9969±0.0000 <sup>h</sup>	15.39±0.03 <sup>b</sup>	28.88±0.26 <sup>ef</sup>	43.29±0.32 <sup>h</sup>	27.83±0.47 <sup>a</sup>
HMT12-MW	1.0130±0.0005 <sup>j</sup>	0.9957±0.0000 <sup>j</sup>	15.35±0.04 <sup>b</sup>	29.12±0.10 <sup>de</sup>	46.37±0.33 <sup>g</sup>	24.50±0.37 <sup>b</sup>

**Notes:** all values are the means of triplicate determinations ±standard deviation. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

### 4.3 Effects of heat-moisture treatment combined with microwave pre- and post- treatment on the structural properties of potato starch

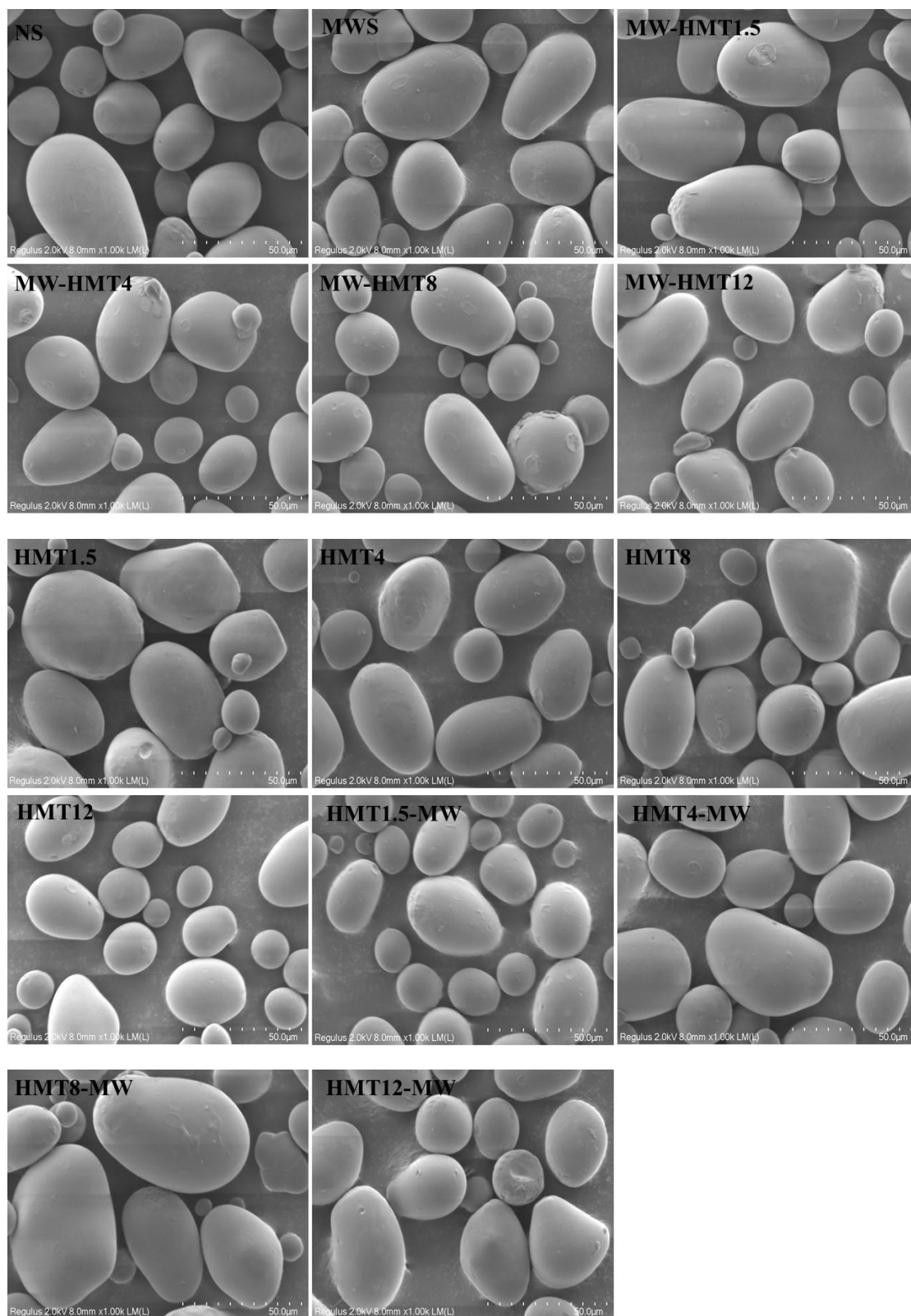
#### 4.3.1 Morphological properties of potato starch modified by heat-moisture

### **treatment combined with microwave pre- and post- treatment**

The surface structural characteristics of native potato starch and modified potato starch samples observed by SEM are presented in Fig. 4.3. The native potato starch granules showed oval or spherical-like shape with no obvious fissures and grooves on the smooth surface. This result was consistent with previous findings reported by Xu *et al.* [209]. Although there were no noteworthy changes in the structure of starch after the MW, HMT and dual MW and HMT, the surface of treated starch granules became rougher with a different degree of pitting and indentation compared with native starch (Fig.4.3). Similar results were obtained for millet starch and rice starch modified by MW and HMT, respectively [21, 171]. Dual MW and HMT modification made the surface of starch granules rougher with more serious depressions or scallops than single MW or HMT modification, especially in the case of the double modified starch granules *via* HMT assisted by MW pre-treatment (Fig.4.3).

For HMT-treated starch, the changes on starch granules surface can be attributed to the partial gelatinization caused by pressure and thermal energy, consequently leading to inconsistent swelling and/or aggregation/fusion of starch particles and rough surface, or even to concavities on the granules surface [171]. For MW-treated starch, the surface roughness and deformation were mainly related to the penetration of microwave energy. When the microwave energy was high enough, the molecular chains that constituted the starch granule structure would break, eventually resulting in pore formation and possible collapse in starch granules [197].





**Fig. 4.3** Morphology of native and modified potato starches

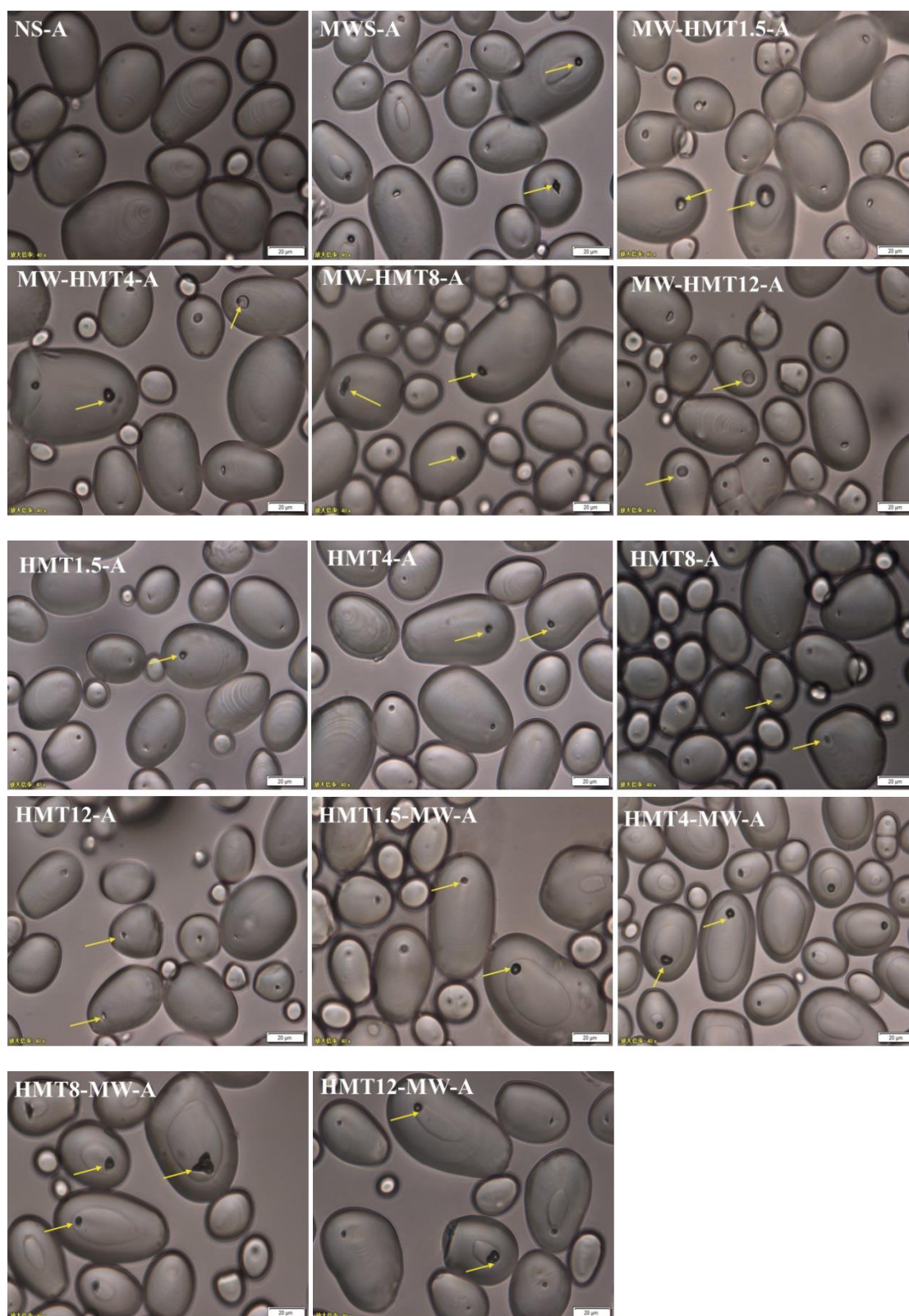
#### **4.3.2 Light microscopic properties of potato starch modified by heat-moisture treatment combined with microwave pre- and post- treatment**



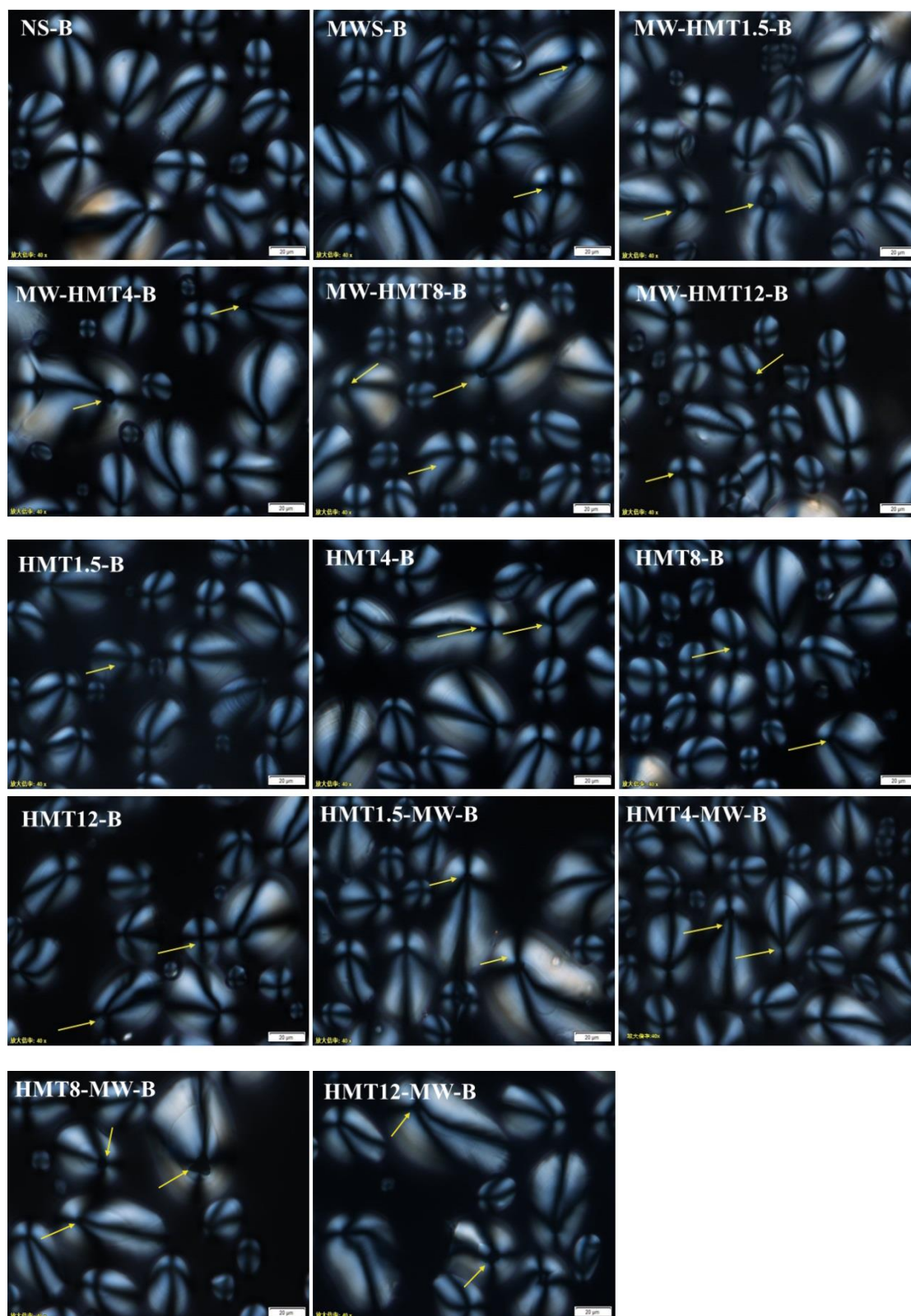
The micrographs of native potato starch and modified potato starch granules observed under normal light microscopy are shown in Fig.4.4 and the micrographs of all the starch samples observed under polarized light microscopy are shown in Fig.4.5.

The normal light microscopy image of native starch (Fig.4.4) exhibited hilum structure with smooth surface, while all treated starch granules showed obvious hollow structure at hilum. The hilum is located in the amorphous zone with relatively fragile structure [95]. Due to this fragile structure, coupled with partial swelling and disruption of starch granules caused by the MW, HMT and dual modification, the large hollow structure appeared at the umbilicus section of starch granules (Fig.4.4). Because of the penetration of microwave irradiation, single MW modification had greater effects on hollow structure at the hilum of starch granules than single HMT. Compared with single modified starch, this phenomenon was more obvious in double modified starch, especially in the HMT starch pretreated by MW.

Although the polarized light microscopy of native potato starch granules (Fig.4.5) and all the modified starches showed a typical Maltese cross with black polarization cross or birefringence, the contour of the Maltese cross of all the modified starches became distorted and fuzzy after MW, HMT and dual modification, and the black zone in the center of the cross became relatively larger than that of native potato starch. These results were similar to those from the previous research of HMT lily starch [95]. Thermal and microwave energy generated by HMT and MW might induce changes in radial orientation of double helices and amylopectin chains, and eventually changed intensity of birefringence [94].



**Fig.4.4** Morphological characteristics of native and modified potato starch granules under normal light  $\times 400$  (A)



**Fig. 4.5** Morphological characteristics of native and modified potato starch granules under polarized light  $\times 400$  (B)

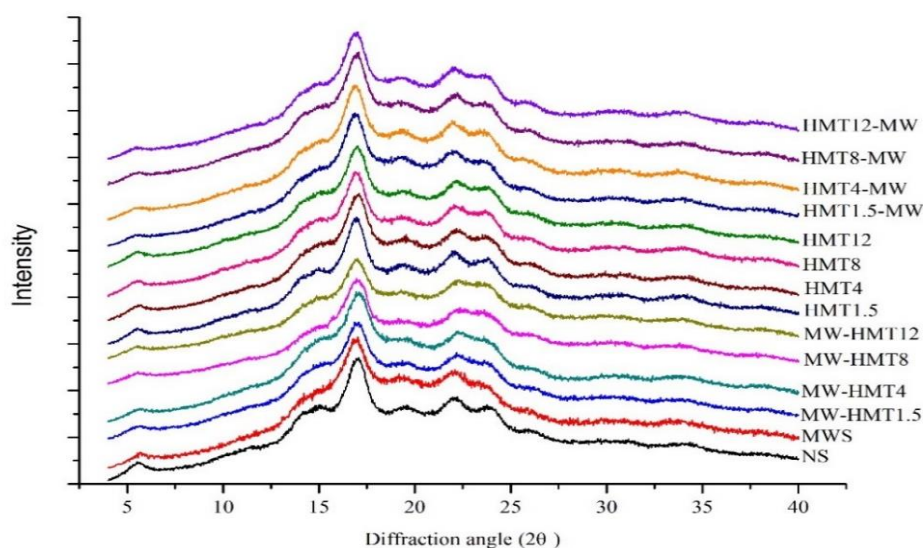
### 4.3.3 X-ray diffraction (XRD) of potato starch modified by heat-moisture treatment combined with microwave pre- and post- treatment



X-Ray diffraction (XRD) analysis is an important technique used to evaluate changes in starch structure. The patterns of native starch and modified starch analyzed by XRD are shown in Fig. 4.6 and the corresponding relative crystallinity was listed in Table 4. Native potato starch exhibited a B-type of X-ray diffraction pattern, which was characterized with a small peak at  $5.6^{\circ} 2\theta$ , a peak at  $17^{\circ} 2\theta$ , and a doublet at  $22^{\circ} 2\theta$  and  $24^{\circ} 2\theta$  [95, 210]. There were some significant changes in the intensities of peaks between native potato starch and modified potato starch. Similar reduction in peak intensities of taro starch after HMT and MW was observed by Deka & Sit [113]. Significant changes in the XRD patterns of all the modified starches were observed (Fig. 4.6). The diffraction peak at  $15^{\circ} 2\theta$  of all the MW-HMT starch and MWS became smooth, the diffraction peaks at  $19.5^{\circ} 2\theta$  gradually disappeared and the double peaks at  $22^{\circ} 2\theta$  and  $24^{\circ} 2\theta$  merged into a broad peak. Similar changes were observed between HMT and HMT-MW starch, the diffraction peak at  $19.5^{\circ} 2\theta$ , and the double peaks at  $22^{\circ} 2\theta$  and  $24^{\circ} 2\theta$  gradually became smooth with the prolongation of heat-moisture treatment time. These results indicate that the X-ray diffraction of potato starch could be changed from B-type to a mixture of A+B type after HMT or/and MW modification. Similar results were obtained by Li *et al.* [95] regarding lily starch modification by HMT. Thirty six water molecules in a central channel of the B-unit cell vaporized during the modification process of MW, HMT, MW-HMT and HMT-MW, and then a pair of double helices moved into the central channel which was originally occupied by the vaporized water molecules, leading to the change of crystalline orientation and destruction of crystalline regions, which eventually induced changes in the XRD pattern of potato starch (B $\rightarrow$ A+B) [79, 95]

All modified potato starch granules had lower relative crystallinity than that of native starch, which was 19.39% (Table 4.11). The relative crystallinity of MWS, MW-HMT1.5, MW-HMT4, MW-HMT8, MW-HMT12 was 15.35%, 15.55%, 15.65%, 15.74% and 15.83% respectively, which indicated that the

relative crystallinity of MW-HMT starch increased with the prolongation of heat-moisture treatment time. Moreover, HMT-MW starch had lower relative crystallinity than that of the single HMT starch at the same heat moisture treatment condition. The reduction of relative crystallinity of microwave-irradiated millet starch was also observed by Li *et al.* [21]. The source of starch and HMT conditions affected the changes observed in starch crystallinity. The results of this research showed that the effect of HMT on potato starch relative crystallinity was consistent with the previous study on the effect of HMT on that of normal maize starch and waxy maize starch [142]. HMT disrupted the amylopectin crystallites and induced the instability of the lamellar arrangement of starch granules, which made the relative crystallinity of HMT starch lower than that of native potato starch [12]. The decrease in crystallinity of the MW starch was attributed to the vibrational motion of the polar molecules induced by the microwave radiation directly impacted the crystalline lamellae inside the granule and destroyed their radial crystalline structure [211-212]. What is more, under the combined effect of thermal energy, microwave radiation and moisture, irreversible damage occurred in crystalline regions of starch granules, inducing the growth of the amorphous or semi-crystalline regions, and consequently reducing the relative crystallinity of starch[213].

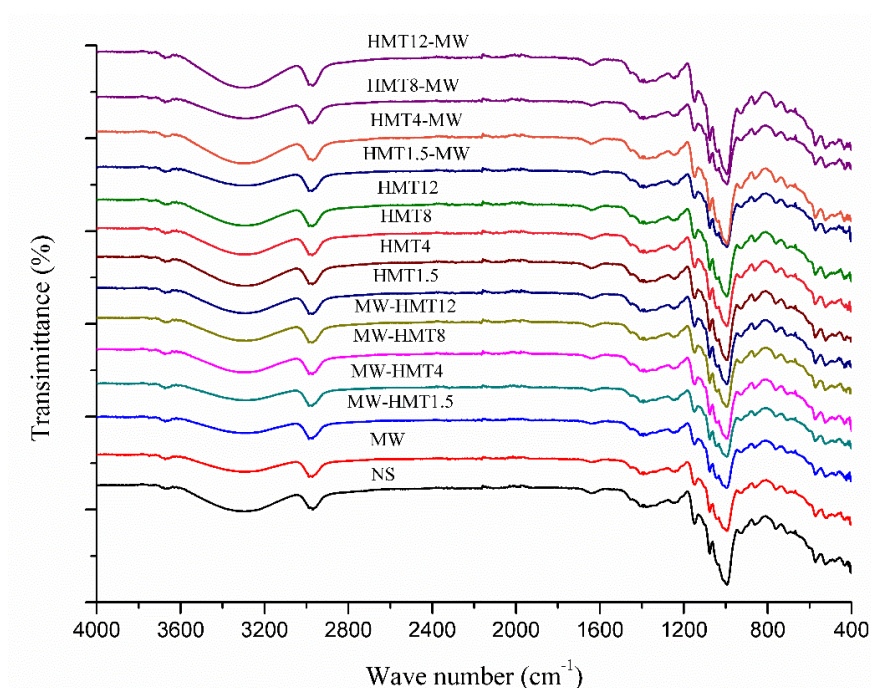


**Fig. 4.6** X-ray diffractograms of native and modified potato starches

#### 4.3.4 FTIR spectroscopy of potato starch modified by heat-moisture treatment combined with microwave pre- and post- treatment

FT-IR is used to monitor the appearance, type and the strength of hydrogen bonds, which reflect the changes in starch molecule structure [214]. The ratio of crystalline regions to amorphous regions of starch granules can be indicated by the absorbance ratio of  $1047/1022\text{ cm}^{-1}$ , while the short-range order can be indicated by the absorbance ratio of  $1047/1035\text{ cm}^{-1}$  [114]. A higher absorbance ratio of  $1047/1022\text{ cm}^{-1}$  indicates larger crystalline region and a higher absorbance ratio of  $1047/1035\text{ cm}^{-1}$  indicates a higher short-range order of starch. The FT-IR spectra of native potato starch and modified potato starch are shown in Fig. 4.7 and their corresponding absorbance ratios of  $1047/1022\text{ cm}^{-1}$  and  $1047/1035\text{ cm}^{-1}$  are summarized in Table 4.11.

Absorption peaks of characteristic groups of all starch granules had similar position and shape (Fig. 4.7), and there was no new absorption peak observed in the spectra, which indicated that any of the modification treatments (single MW, single HMT and HMT combined with MW pre- and post-treatment) neither created new functional groups nor altered the primary structure of the starch.



**Fig. 4.7** FTIR spectra of native and modified potato starches

All modified starch granules had lower absorbance ratios of  $1047/1022\text{ cm}^{-1}$  than that of native starch, and HMT combined with MW pre- and post-treatment had stronger effects on the decrease in the absorbance ratios of  $1047/1022\text{ cm}^{-1}$  than single MW or single HMT (Table 4.11). What is more, with the increase in HMT duration, the ratios of  $1047/1022\text{ cm}^{-1}$  of single HMT starch samples and HMT starch samples post-treated by MW decreased to different extents, while opposite results were observed for HMT starch samples pre-treated by MW. The ratios of  $1047/1035\text{ cm}^{-1}$  of all the starch samples had the same variation trend as that of  $1047/1022\text{ cm}^{-1}$ . These results indicated that microwave irradiation and heat-moisture treatment might have destroyed the double helix structure and crystal region of starch granules [215]. Under different treatment conditions, the changing trend of the ratios of  $1047/1022\text{ cm}^{-1}$  and  $1047/1035\text{ cm}^{-1}$  was consistent with the changing trend of relative crystallinity analyzed by XRD (Table 4.11), which further confirmed that single MW, single HMT and HMT combined with MW pre- and post- treatment destroyed the crystal structure of potato starch.

### Conclusions to section 4

1. In this section, the effect of heat-moisture treatment combined with microwave pre- and post- treatment on the swelling power, solubility, freeze-thawing stability, retrogradation, transparency and textural properties of NS and all the modified starch were investigated. The research results showed that all the modified starch showed lower swelling power than that of native starch when the test temperature was 65–85°C, while opposite results were obtained at 95°C. All the modified starch samples showed higher solubility than that of native starch when test temperature was 75–95°C. Single MW, short-time single HMT and short-time HMT combined with MW pretreatment can enhance the repeated freeze-thaw stability of potato starch pastes, while long- time HMT ( $\geq 4$ h) could weaken the freeze-thaw stability of potato starch. Dual modification of HMT and MW had greater effects on starch retrogradation than that of single HMT or single MW, moreover, HMT combined with MW pretreatment also had greater effects on starch retrogradation than that HMT combined with MW post-treatment. Similar to the results of the retrogradation of starch, HMT heating time had great significant effect on starch transparency and dual modification of HMT combined with MW had greater effect on the transparency of starch paste than that of single HMT and MW. The hardness, cohesiveness, gumminess and chewiness of all the HMT modified potato starch gel (including single HMT, HMT combined with MW) decreased with the extension of heating time. The HMT potato starch pretreated by MW had higher hardness value than that of HMT potato starch post-treated by MW.

2. Although color differences was no visibly differentiated between NS and all treated starch, HMT treatment caused a slight increase of lightness ( $L^*$  values), while single MW treatment caused a slight decrease of lightness, indicating that the color of all the HMT treated samples (HMT, MW-HMT, HMT-MW) became brighter and the color of the single MW treated sample (MWS) became darker.



Although there were significant differences of the differences of color ( $\Delta E$ ), it could conclude that all treatments did not markedly change potato starch color for  $\Delta E$  was always below 3, indicating that color difference was no visible differentiated of all the treated starch samples. The results of particle size distribution showed that D50, D (4,3) and D (3,2) of all treated starch were higher than NS, while the value of S.S.A. was significantly decreased by MW and HMT, indicating that MW and HMT treatments can caused expansion, partial gelatinization and agglomeration of starch granules, resulting in large particle size of starch granules. Although the bound water in all starch samples was the main water which at least accounted for 90%, three peaks were observed in  $T_2$  of MW treated starch (MWS, MW-HMT and HMT-MW), two peaks were observed in  $T_2$  of native and single HMT treated starch, which indicated the MW treated starch had three different state water, while NS and single HMT treated starch only had two different state water. There were significant differences of  $PT_{21}$  and  $PT_{22}$  between NS and all treated starch, NS had the lowest  $PT_{21}$  but highest  $PT_{22}$ , indicating MW and HMT treatments could change the water distribution and improve the interaction between starch and water.

3. HMT, MW and HMT combined with MW pre- and post-treatment had significant effects on the microstructure, crystalline and structural properties and digestibility of potato starch. As can be observed from the scanning electron microscopy, normal light and polarized light microscopy, some depressions or potholes appeared on the surface of starch granules after modification, and the center of polarized cross structure slowly expanded. Dual starch modification *via* MW and HMT made the surface of its granules rougher and caused more serious depressions or scallops than single modification with MW or HMT, especially in the case of HMT-MW. All the treatments increased the pasting temperature and setback viscosity but decreased peak viscosity and breakdown viscosity of starch. The FT-IR and XRD spectra implied that HMT and MW destroyed the double helices and crystalline structure of potato starch. All treatments increased the

content of RS but reduced the content RDS of potato starch. Under the same HMT heating duration, the RS content of starch modified by HMT combined by with MW post-treatment was significantly higher than that of starch modified by HMT combined by MW pre-treatment and single HMT. The information obtained in this research might be beneficial to the industrial applications of microwave and heat-moisture techniques deployed to modify starch and eventually produce new starch materials satisfying the potential consumer requirements.

## SECTION 5 APPLICATIONS OF PHYSICALLY MODIFIED POTATO STARCH IN FOOD PRODUCTS

Wheat flour is an important ingredient in many food products, the global commercial flour market is expected to reach \$53 billion USD by 2025, with wheat flour accounting for the majority of the market share [216]. However, the high glycemic index (GI) of food products restricts the consumption of patients with diabetes and cardiovascular disease [149]. The nutritional quality of wheat products can be improved through partially substitution of wheat flour with whole flour or other functional ingredients, e.g. modified starch, of which modification of starches can promote reduction of GI and improving the quality characteristics of the foods [30]. Englyst *et al.*[217] classified starch as rapidly digestible starch (RDS), slowly digestible starch (SDS) and resistant starch (RS) according to the rate of glucose release when starch is hydrolyzed by starch digestive enzymes. RDS can cause a sudden large fluctuations of blood glucose level after ingestion, leading to the occurrence of many chronic diseases. SDS can be completely digested in the human small intestine, but its digestion rate is slower than that of RDS, therefore SDS has special physiological functions of continuously and slowly releasing energy, stabilizing blood glucose level for a long time and prolonging satiety, which makes SDS has a positive effect on weight and obesity control. Besides, SDS plays an role in the diet of type 2 diabetes patients, because it can reduce the risk of cardiovascular disease and coronary heart disease [186]. RS cannot be absorbed in small intestine of healthy human, but it can be fermented by microorganisms in large intestine to produce butyric acid and other short chain fatty acids, which is conducive to the growth of beneficial bacteria and has positive effects of promoting intestinal peristalsis [218].

In our previous researches, potato starch was modified by heat-moisture treatment (HMT) and microwave treatment (MW), and the results indicated that both HMT and MW could increase the SDS content and RS content of potato starch. Therefore, the addition of HMT modified potato starch (HMTS) or MW

modified potato starch (MWS) is beneficial to the improvement of cookies, noodles and steamed bread quality.

Cookies, noodles and steamed breads are three typical types of food products. Among them, cookies refer to confectionery products, noodles refer to pasta products, steamed bread refers to bakery products. Most recipes for cookies include high sugar and high fat, resulting in high calories. The high calorie and high sugar content of cookies is not conducive to the stability of blood sugar, and long-term consumption of such high-sugar and high-fat foods can easily lead to obesity and increase the risk of diabetes [219]. Noodles have been widely consumed for its simplicity, convenience and easy cooking [220]. The widespread consumption of noodles enriches human dietary life, but also accelerates the appearance of hypertension, hypercholesterolemia and hyperglycaemia, which is harmful to human health [152]. Steamed bread becomes popular in the world markets and is considered as healthy food possible due to low oil and low sodium content, and also due to relatively low steaming temperature (100°C) makes steamed bread not containing Maillard reaction products such as acrylamide and furan [221-222]. Since most of the wheat products belong to high glycemic index (GI) foods, enhancing cookies, noodles and steamed bread with functional components has the potential to be beneficial [30]. Strategies for developing cookies, noodles and steamed bread with low GI remain to be developed to help people with diabetes and other diseases [31].

Modified starch can be used as a kind of food products quality improving agent. Partial substitution of wheat flour with modified starch can enhance the nutritional quality of cookies, noodles and steamed bread. However, the dough rheological properties and the product quality may be altered by substitution of wheat flour with other types of low-gluten flour [32]. Therefore, this study investigated the effects of substitution of wheat flour with potato starch modified by heat-moisture treatment (HMTS) and microwave treatment (MWS) on the quality characteristics of three typical food products including cookies, fresh

noodles and steamed bread. In this research the HMT modified potato starches (HMTS) were prepared by HMT at 90°C for 1.5 h with 23.56% moisture content of starch (the optimized process parameters of HMT, the results were showed in Section 3), whereas the MW modified potato starches (MWS) were prepared by MW at 400W power for 5min with 25% moisture content of starch. According to the Table 3.18, the content of RDS, SDS and RS in HMTS were 28.01%, 57.69% and 14.97%, whereas the content of RDS, SDS and RS in MWS were 29.14 %, 55.90% and 14.97% (Table 4.11). According to experimental determination, the total starch content in low protein flour and wheat flour was 73.92% and 73.15% respectively. The content of RDS, SDS and RS was in low protein flour was 54.35%, 13.08% and 6.5%, while in wheat flour was 53.82%, 12.98% and 6.35%, respectively.

## **5.1 Preparation of cookies, fresh noodles and steamed bread**

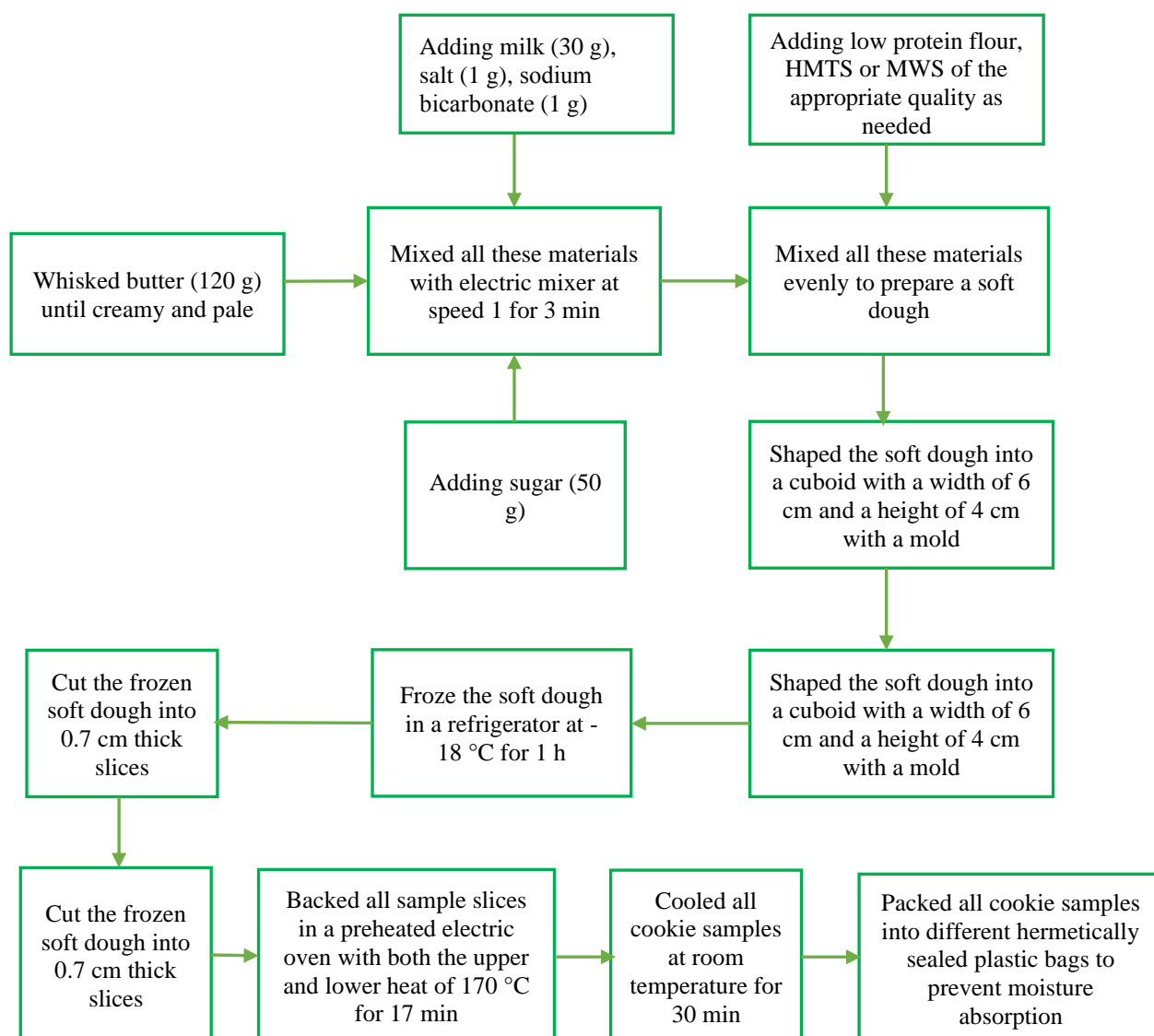
### **5.1.1 Preparation of fresh cookies**

The cookies had the following formulation: low protein flour (180 g, (the protein content was  $7.0\% \pm 1.5\%$ , 12% moisture content), unsalted butter (120 g), powdered white granulated sugar (50 g), pure milk (30 g), salt (1 g), sodium bicarbonate(1 g), HMTS or MWS substituted low protein flour with equal amounts of 5%, 15%, and 25% to make experimental cookies. According to the different substitution of HMTS and MWS, the experimental cookies were named as HMT-5, HMT-15, HMT-25, MW-5, MW-15 and MW-25 respectively. Cookies made entirely of low protein flour without HMTS and MWS were used as control. The recipe of cookies was showed as Table 5.1 and the manufacturing process of cookies as shown in Fig.5.1.

**Table 5.1**

## The recipe of cookies

Cookie samples	Type of raw material							
	Low protein flour (g)	HMTS (g)	MWS (g)	Butter (g)	Sugar (g)	Milk (g)	Salt (g)	Sodium bicarbonate (g)
Control	180	0	0	120	50	30	1	1
HMT-5	171	9	0	120	50	30	1	1
HMT-15	153	27	0	120	50	30	1	1
HMT-25	135	45	0	120	50	30	1	1
MW-5	171	0	9	120	50	30	1	1
MW-15	153	0	27	120	50	30	1	1
MW-25	135	0	45	120	50	30	1	1

**Fig.5.1** The manufacturing process of cookies

Whisked butter until creamy and pale after the butter was softened, added the powdered white granulated sugar and mixed evenly, then pure milk and salt were added and mixed with electric mixer at speed 1 for 3 minutes to make all the materials smooth, added the low protein flour in batches to make a soft dough. The soft dough was wrapped with baking paper and shaped into a cuboid with a width of 6 cm and a height of 4 cm with a mold. And then the dough was hardened in a refrigerator at -18°C for 1 hour. The frozen cuboid dough was cut into 0.7 cm thick slices with a knife and all the slices were put into the prepared baking pan. All these samples were baked in a preheated electric oven with both the upper and lower heat of 170°C for 17 minutes. After baking, the cookies were removed out from the oven, left to cool for 30 min at room temperature, and packed into hermetically sealed plastic bags to prevent moisture absorption. All quality measurements were performed in 1 hour after baking.

### **5.1.2 Preparation of fresh noodles**

The basic formulation of noodles was consisted of wheat flour (the protein content was  $10.0\% \pm 1.0\%$ , 12% moisture content) 100 g, salt 0.48 g and water 48 g. Wheat flour was substituted with HMTS or MWS at the levels of 10%, 20%, 30%, 40% and 50%, and named as HMT-10a, HMT-20a, HMT-30a, HMT-40a and HMT-50a, MW-10a, MW-20a, MW-30a, MW-40a and MW-50a, respectively. When the content of HMTS or MWS was more than 50%, the dough with strong network structure could not be formed due to too little gluten, thus the maximum substitution of HMTS and MWS was set as 50%. Wheat flour without HMTS or MWS was used as control. The recipe of fresh noodles was showed as Table 5.2.

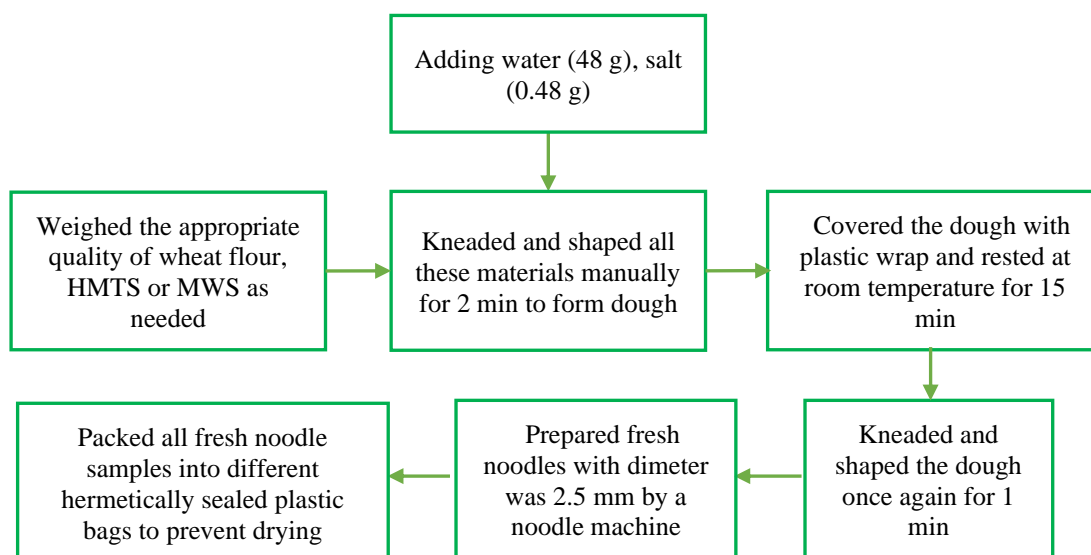
Dough was formed by mixing wheat flour, HMTS or MWS, water was kneaded and shaped manually for 2 min. The obtained dough was divided into two parts, one for the determination of textural and tensile properties of dough, and the other one for the preparation of fresh noodles. The dough for preparation of fresh noodles was covered with plastic wrap and rested at room temperature for 15 min. After resting, the dough was kneaded again and then passed through

a small noodle machine ((Joyoung, M6-L18, Joyoung Company Limited, Jinan, Shandong, China). The diameter of fresh noodles was 2.5 mm. The manufacturing process of fresh noodles as shown in Fig.5.2.

**Table 5.2**

The recipe of fresh noodles

Fresh noodle samples	Type of raw material				
	Wheat flour (g)	HMTS (g)	MWS (g)	Water (g)	Salt (g)
Control	100	0	0	48	0.48
HMT-10a	90	10	0	48	0.48
HMT-20a	80	20	0	48	0.48
HMT-30a	70	30	0	48	0.48
HMT-40a	60	40	0	48	0.48
HMT-50a	50	50	0	48	0.48
MW-10a	90	0	10	48	0.48
MW-20a	80	0	20	48	0.48
MW-30a	70	0	30	48	0.48
MW-40a	60	0	40	48	0.48
MW-50a	50	0	50	48	0.48



**Fig.5.2** The manufacturing process of fresh noodles

### 5.1.3 Preparation of steamed bread



The procedures for steamed bread manufacture were mixing, resting, sheeting, dividing, moulding, proofing, and steaming. The basic recipe of control of steamed bread was 100 g wheat flour (the protein content was  $10.0\% \pm 1.0\%$ , 12% moisture content), 55 g water, 1.0 g yeast and 1.0 g salt. Wheat flour was substituted with HMTS or MWS at the levels of 10%, 20%, 30%, 40% and 50%, and named as HMT-10b, HMT-20b, HMT-30b, HMT-40b and HMT-50b, MW-10b, MW-20b, MW-30b, MW-40b and MW-50b, respectively. When the content of HMTS or MWS was more than 50%, the dough with strong network structure could not be formed due to too little gluten, thus the maximum substitution of HMTS and MWS was set as 50%. Wheat flour without HMTS or MWS was used as control. The recipe of steamed breads was showed as Table 5.3.

Dough was formed by mixing wheat flour, HMTS or MWS, water, salt and yeast and was kneaded and shaped manually for 2 min. Then the dough samples were fermented in a fermenting box at 35°C and 65% relative humidity for 60 min. After fermentation, the dough samples were kneaded one more time by adding 5 g mixed flour (different substitution of wheat flour with HMTS/ or MWS), and then divided into small pieces ( $50 \pm 0.5$  g), rounded and shaped into buns, proofed for 10 min at 35°C and 65% relative humidity and then steamed at 100°C for 20 min at atmospheric pressure. The steamed bread buns were cooled at room temperature for 1 h prior to all analyses. The manufacturing process of steamed breads as shown in Fig.5.3.

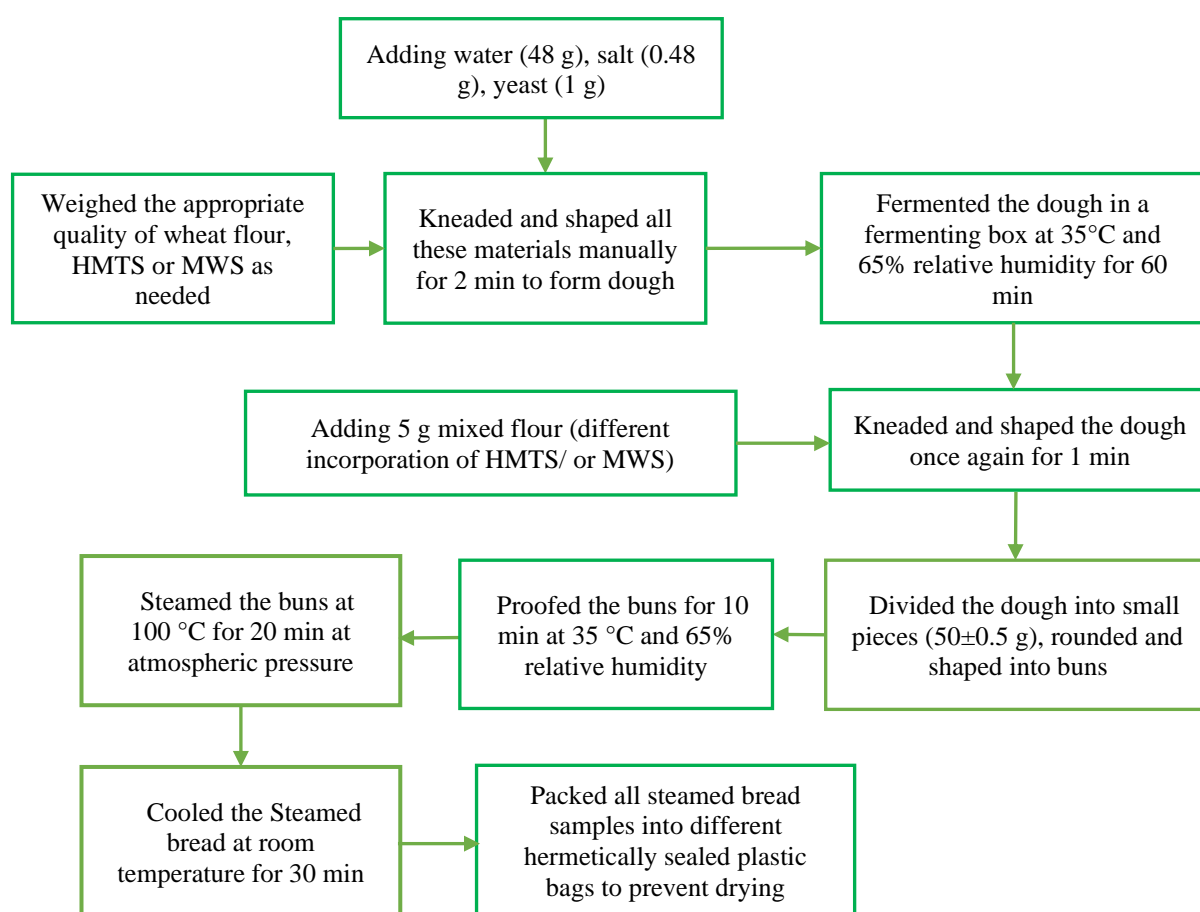
**Table 5.3**

The recipe of steamed breads

Steamed bread samples	Type of raw material					
	Wheat flour (g)	HMTS (g)	MWS (g)	Water (g)	Salt (g)	Yeast (g)
Control	100	0	0	55	1	1
HMT-10b	90	10	0	55	1	1
HMT-20b	80	20	0	55	1	1
HMT-30b	70	30	0	55	1	1
HMT-40b	60	40	0	55	1	1

Table 5.3 is continued

Steamed bread samples	Type of raw material					
	Wheat flour (g)	HMTS (g)	MWS (g)	Water (g)	Salt (g)	Yeast (g)
HMT-50b	50	50	0	55	1	1
MW-10b	90	0	10	55	1	1
MW-20b	80	0	20	55	1	1
MW-30b	70	0	30	55	1	1
MW-40b	60	0	40	55	1	1
MW-50b	50	0	50	55	1	1

**Fig.5.3** The manufacturing process of steamed breads

## 5.2 Substitution of wheat flour with modified potato starch affects the quality of cookies

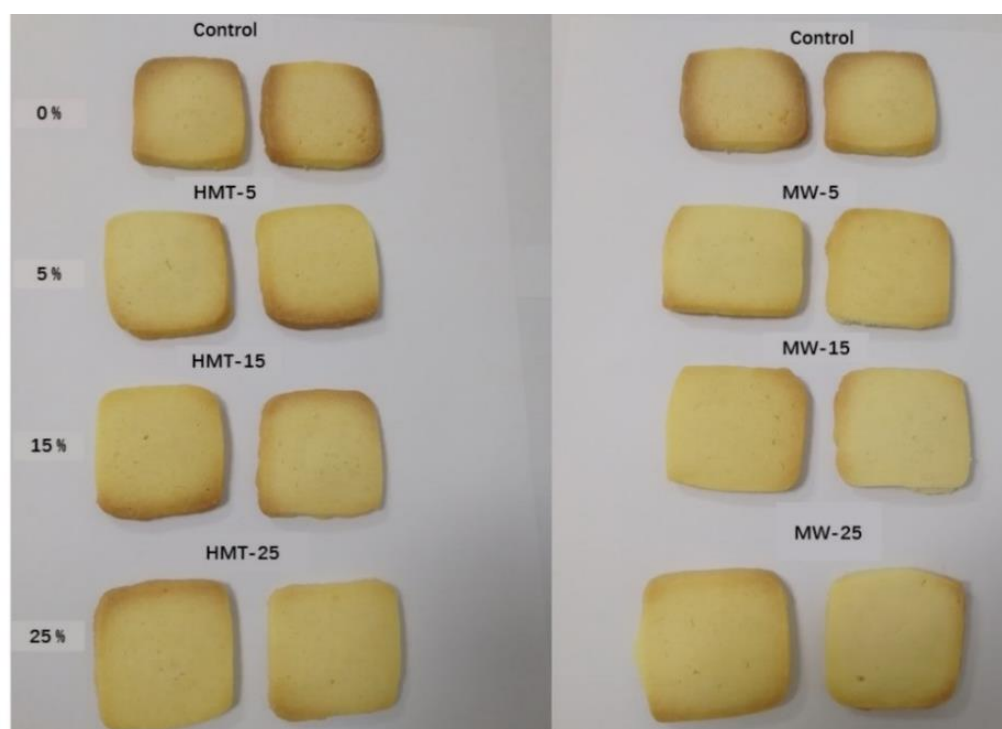
The basic recipe for cookies was as follows: 180 g low protein flour (the protein content was  $7.0\% \pm 1.5\%$ , 12% moisture content), 120 g unsalted butter, 50 g powdered white granulated sugar, 30 g pure milk, 1 g salt.

HMTS (modified starch prepared under the optimized conditions of HMT method) or MWS (modified starch prepared by MW (5min, 400W)) substituted

low protein flour with equal amounts of 5%, 15%, and 25% to make experimental cookies. According to the different substitution of HMTS and MWS, the experimental cookies were named as HMT-5, HMT-15, HMT-25, MW-5, MW-15 and MW-25 respectively. Cookies made entirely of low protein flour without HMTS and MWS were used as control.

### 5.2.1 Color analysis of cookies

Fig.5.4 showed examples of cookies produced with different substitution of low protein flour with HMTS or MWS. As can be seen from Fig.5.4, the edges of control cookies made entirely of low protein flour were more prone to scorch. Adding appropriate HMTS or MWS could effectively reduce the scorch phenomenon of cookies edges.



**Fig.5.4** Photos of cookies with different substitution of low protein flour with HMTS and MWS

Color is related to the physicochemical characteristics of ingredients and baking conditions. In this study, the center rather than the edge of every cookie was used as the test point for color determination. Table 5.4 presented the results of experiments evaluating the effect of partial substitution of low protein flour with HMTS or MWS on cookies color. Compared with the control cookies, all the

experimental cookies had lower  $a^*$  value, but higher  $L^*$  value and  $b^*$  value, indicating the cookies made of HMTS or MWS became brighter (higher  $L^*$ ), less reddish (lower  $a^*$ ), and more yellowish (higher  $b^*$ ). The  $L^*$  value and  $a^*$  value increased with the increase amount of MWS, while the increase amount of HMTS increased the  $L^*$  value but decreased  $a^*$  value. The color differences ( $\Delta E$ ) between experimental cookies and the control cookies was above 3, indicating that the differences in color between the control and experimental cookies were detectable by the human eye when the substitution amount of low protein flour with HMTS or MWS reached 5%. Although there was no significant difference between the experimental samples, the  $L^*$  value and  $\Delta E$  increased with the increase of substitution amount of HMTS or MWS. If the scorch phenomenon of cookies edges was not considered, the color observed by human eye was same as the result measured by colorimeter.

**Table 5.4**

Effect of HMT and MW modified potato starch on color of cookies

Cookies samples	$L^*$	$a^*$	$b^*$	$\Delta E$
Control	78.08±1.46 <sup>b</sup>	2.46±0.08 <sup>a</sup>	29.65±0.71 <sup>b</sup>	-
HMT-5	81.05±0.18 <sup>a</sup>	2.25±0.06 <sup>b</sup>	30.69±0.30 <sup>a</sup>	3.16±0.21 <sup>a</sup>
HMT-15	81.18±0.22 <sup>a</sup>	2.12±0.06 <sup>c</sup>	30.84±0.11 <sup>a</sup>	3.33±0.19 <sup>a</sup>
HMT-25	81.24±0.29 <sup>a</sup>	2.07±0.05 <sup>cd</sup>	30.78±0.49 <sup>a</sup>	3.41±0.30 <sup>a</sup>
MW-5	80.75±0.21 <sup>a</sup>	1.98±0.09 <sup>d</sup>	30.95±0.14 <sup>a</sup>	3.01±0.13 <sup>a</sup>
MW-15	80.95±0.46 <sup>a</sup>	2.28±0.06 <sup>b</sup>	31.35±0.18 <sup>a</sup>	3.34±0.44 <sup>a</sup>
MW-25	81.08±0.43 <sup>a</sup>	2.32±0.05 <sup>b</sup>	31.23±0.71 <sup>a</sup>	3.45±0.42 <sup>a</sup>

**Notes:** all values are the mean of at least triplicate determinations ± SD. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

### 5.2.2 Texture analysis of cookies

Texture analyzer is the main instrument used to objectively evaluate food quality, which mainly reflects the food texture properties related to mechanical properties. Cookies texture is an important index to evaluate the cookies quality, and the crispy value also can be used as sensory indicator of cookies. Too high crispy value would lead to dross, rough taste in cookies, which would reduce the

quality of cookies, while poor crispy value would lose the unique taste texture of cookies [223]. Texture properties of the control cookies and experimental cookies were investigated in 1 hour after baking according to the mentioned method, and the results were showed in Table 5.5. The hardness (included average hardness, surface hardness and max hardness) of cookies with HMTS or MWS was significantly lower than of control ( $P_{\text{value}} < 0.05$ ), but higher crispy value, indicating less work to be consumed when chewing [224]. The hardness of cookies decreased with the increase of substitution amount of HMTS or MWS, while crispy value increased with the increase of substitution amount of HMTS or MWS. When the substitution amount was same, the cookies made of HMTS had lower hardness and crispy value than of MWS, which indicated the HMTS had greater effect on the hardness of cookies while MWS had greater effect on crispy value. Moreover, the substitution of low protein flour with HMTS or MWS diluted the gluten in dough, which enhanced the dough plasticity and fat lubrication effect in the dough [225].

**Table 5.5**

Effect of HMT and MW modified potato starch on texture properties of cookies

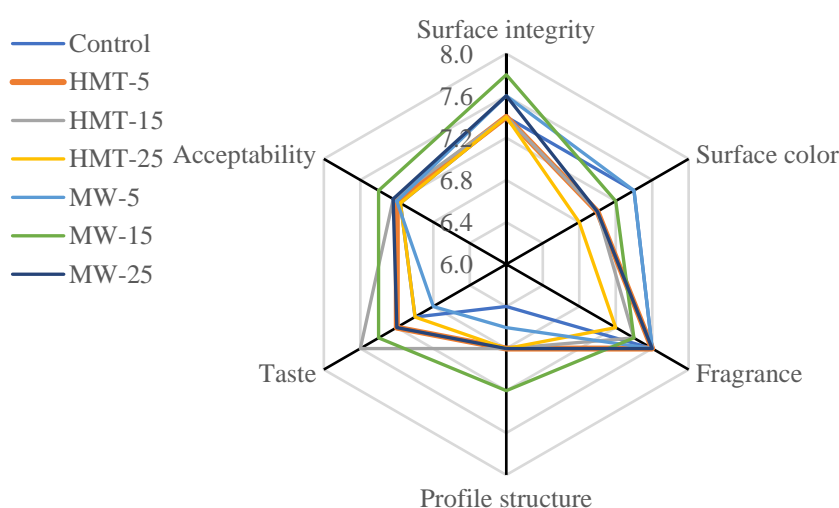
Cookies samples	Average hardness /g	Surface hardness /g	Max hardness /g	Crunch value	Crispy value
Control	543.55±11.30 <sup>a</sup>	545.66±12.96 <sup>a</sup>	725.79±14.02 <sup>a</sup>	1.40±0.55 <sup>a</sup>	2.40±0.55 <sup>e</sup>
HMT-5	425.42±18.03 <sup>bc</sup>	365.96±10.13 <sup>e</sup>	623.62±14.76 <sup>bc</sup>	1.00±0.00 <sup>a</sup>	2.60±0.55 <sup>e</sup>
HMT-15	412.15±13.17 <sup>cd</sup>	345.92±11.54 <sup>f</sup>	596.49±10.95 <sup>d</sup>	1.00±0.00 <sup>a</sup>	3.60±0.55 <sup>d</sup>
HMT-25	319.80±13.63 <sup>e</sup>	308.05±9.06 <sup>g</sup>	472.39±21.21 <sup>f</sup>	1.20±0.45 <sup>a</sup>	4.00±0.71 <sup>cd</sup>
MW-5	442.03±11.01 <sup>b</sup>	462.96±16.43 <sup>b</sup>	632.97±18.73 <sup>b</sup>	1.00±0.00 <sup>a</sup>	4.40±0.55 <sup>c</sup>
MW-15	424.48±14.27 <sup>bc</sup>	441.99±19.64 <sup>c</sup>	605.65±22.52 <sup>cd</sup>	1.25±0.46 <sup>a</sup>	6.38±0.52 <sup>b</sup>
MW-25	396.32±15.16 <sup>d</sup>	414.89±13.63 <sup>d</sup>	542.20±12.95 <sup>e</sup>	1.00±0.00 <sup>a</sup>	7.25±0.50 <sup>a</sup>

**Notes:** all values are the mean of at least triplicate determinations ± SD. The means within the same column with different letters are significantly different ( $P < 0.05$ ).

### 5.2.3 Sensory evaluation of cookies

A comprehensive assessment of cookies with HMTS or MWS was showed in Fig. 5.5. As can be seen from Fig. 5.5, there were no significant difference in

sensory indexes of all samples. Some cookies with HMTS or MWS had higher scores than the control cookies with intact shape and uniform color, while some cookies had lower scores. Due to the influence of MWS on the uniformity and texture distribution of cookies, appropriate addition of MWS could improve the score of surface integrity and profile structure. Although adding appropriate HMTS or MWS can effectively reduce the scorch phenomenon of cookies edges, the color scores of HMTS cookies and MWS cookies were lower than the control cookies, which was consistent with the results measured by colorimeter and the results observed with naked eye. However, the acceptability score of cookies added with HMTS or MWS was not lower than that of the control sample and even slightly higher. Which may be related to the improvement of crispy value of cookies by adding HMTS or MWS. In general, adding 15% HMTS or 15% MWS could had the highest acceptability score of cookies. Therefore, the addition of HMTS or MWS to cookies would not reduce the acceptability, but also could improve the quality of products. Similar results were obtained when evaluating the effect of heat moisture treated corn starch and heat moisture combined with microwave treated cassava starch on the quality of cookies [226].



**Fig. 5.5** Effect of HMT and MW modified potato starch on sensory scores of cookies

#### 5.2.4 Postprandial blood glucose levels of cookies

Dietary carbohydrates are the main components that affect the postprandial

blood glucose levels. Normal fasting blood glucose in adults is 3.9–6.1 mmol/L. The postprandial blood glucose levels of participants at different times after eating biscuits were showed in Table 5.6. The fasting blood glucose levels (Table 5.6, 0 min) of all participants were within the normal range, and there was no significant difference between them ( $P_{\text{value}} > 0.05$ ). The change trends of postprandial blood glucose levels before and after eating cookies were the same, with an increase followed by a decrease. After eating the control cookies, the blood glucose levels increased quickly and reached its peak value (8.41 mmol/L) at 30 min, and then the blood glucose levels decreased rapidly. The postprandial blood glucose levels fluctuated greatly. Eating cookies with the incorporation of 5% HMTS (Table 5.6, HMT-5) or 5% MWS (Table 5.6, MW-5) had the same postprandial blood glucose levels fluctuation changes as that of the control cookies, and reached the peak values at 30 min. The rates of increase in postprandial blood glucose after eating cookies with more than 15% incorporation of HMTS or MWS were slower than that of the control cookies. The postprandial blood glucose reached their peak values at 45 min after eating, and the peak values were significantly lower than that of the control cookies, and then the blood glucose levels decreased slowly. Moreover, postprandial blood glucose levels of participants at different times after eating cookies with incorporation of HMTS or MWS were lower than that of the control cookies. All these results indicated that cookies with incorporation of HMTS or MWS were more suitable for diabetics or the elderly, especially when the incorporation of HMTS or MWS exceeded 15%.

**Table 5.6**

Postprandial blood glucose levels of participants at different times after eating cookies

Cookie samples	Postprandial blood glucose levels (mmol/L)						
	0 min	15 min	30 min	45 min	60 min	90 min	120 min
Control	4.64±0.07 <sup>a</sup>	6.54±0.03 <sup>a</sup>	8.41±0.06 <sup>a</sup>	7.84±0.02 <sup>a</sup>	7.04±0.03 <sup>a</sup>	6.26±0.06 <sup>a</sup>	5.45±0.06 <sup>a</sup>
HMT-5	4.70 ±0.10 <sup>a</sup>	6.21±0.03 <sup>b</sup>	7.83±0.02 <sup>b</sup>	7.67±0.04 <sup>b</sup>	6.90±0.03 <sup>b</sup>	5.96±0.04 <sup>b</sup>	5.42±0.03 <sup>ab</sup>



Table 5.6 is continued

Cookie samples	Postprandial blood glucose levels (mmol/L)						
	0 min	15 min	30 min	45 min	60 min	90 min	120 min
HMT-15	4.76± 0.11 <sup>a</sup>	5.80±0.07 <sup>d</sup>	7.10±0.09 <sup>c</sup>	7.32±0.04 <sup>c</sup>	6.52±0.04 <sup>c</sup>	5.70±0.04 <sup>c</sup>	5.33±0.04 <sup>bc</sup>
HMT-25	4.67±0.02 <sup>a</sup>	5.64±0.01 <sup>ef</sup>	6.97±0.05 <sup>d</sup>	7.08±0.05 <sup>e</sup>	6.28±0.04 <sup>d</sup>	5.55±0.07 <sup>de</sup>	5.28±0.04 <sup>cd</sup>
MW-5	4.77±0.09 <sup>a</sup>	6.13±0.07 <sup>c</sup>	7.81±0.02 <sup>b</sup>	7.70±0.06 <sup>b</sup>	6.86±0.06 <sup>b</sup>	5.99±0.02 <sup>b</sup>	5.40±0.04 <sup>ab</sup>
MW-15	4.73±0.05 <sup>a</sup>	5.72±0.03 <sup>c</sup>	7.18±0.03 <sup>c</sup>	7.31±0.04 <sup>c</sup>	6.56±0.03 <sup>c</sup>	5.60±0.04 <sup>d</sup>	5.19±0.05 <sup>de</sup>
MW-25	4.66±0.03 <sup>a</sup>	5.61±0.04 <sup>f</sup>	6.99±0.07 <sup>d</sup>	7.18±0.04 <sup>c</sup>	6.30±0.06 <sup>d</sup>	5.48±0.06 <sup>e</sup>	5.13±0.12 <sup>e</sup>

**Notes:** all values are the mean of triplicate determinations ± SD. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

Based on the results of color analysis, texture analysis and sensory evaluation of cookies, it was concluded that the optimal substitution amount of wheat flour with HMTS or MWS was 15%. Therefore, the optimal recipe of cookies made with incorporation of HMTS or MWS was as follow: 153 g low protein flour (the protein content was  $7.0\% \pm 1.5\%$ , 12% moisture content), 27 g HMTS or MWS (12% moisture content), 120 g unsalted butter, 50 g powdered white granulated sugar, 30 g pure milk, 1 g salt, 1 g sodium bicarbonate.

### 5.3 Substitution of wheat flour with modified potato starch affects the quality of fresh noodles

The basic formulation of noodles was consisted of wheat flour (the protein content was  $10.0\% \pm 1.0\%$ , 12% moisture content) 100 g, salt 0.48 g and water 48 g. Wheat flour was substituted with HMTS or MWS at the levels of 10%, 20%, 30%, 40% and 50%, and named as HMT-10a, HMT-20a, HMT-30a, HMT-40a and HMT-50a, MW-10a, MW-20a, MW-30a, MW-40a and MW-50a, respectively. When the content of HMTS or MWS was more than 50%, the dough with strong network structure could not be formed due to too little gluten, thus the maximum substitution of HMTS and MWS was set as 50%.

#### 5.3.1 Texture and tensile properties of dough

Dough texture properties reflect the combination of water and gluten, which are important indicators to measure the quality characteristics of dough within a



certain range [227]. The texture and tensile properties of dough were determined by TPA compressive test and TPA tensile test in this study, and the results were showed in Table 5.7. With the increase of HMTS substitution, the dough hardness decreased first and then increased, while the dough hardness increased with the increase incorporation of MWS. The different effects of HMTS and MWS on dough hardness were related to the different starch structure properties of HMTS and MWS. Adding proper amount of HMTS would make the dough become soft, and the hardness value would be reduced compared with the control dough. However, incorporating MWS required absorbing more water, and excessive MWS would make starch particles filled in the gluten network, which would reduce elasticity of the dough and hindered the formation of gluten network structure, thus resulting in the reduction of pores and the increase of the hardness of dough. The cohesiveness, gumminess, chewiness and resilience were decreased with the increasing incorporation of HMTS or MWS. The dough springiness has no significant change with the increasing incorporation of HMTS except the sample HMT-50, but the dough springiness reduced with the increasing incorporation of MWS. These results might be related to the fact that the incorporation of starch reduced the gluten content in dough, leading to the deterioration of the dough network structure.

**Table 5.7**

Effect of HMT and MW modified potato starch on texture and tensile properties of dough

Dough samples	Dough TPA compressive test						Dough TPA tensile test	
	Hardness (g)	Springiness (mm)	Cohesiveness (–)	Gumminess (g)	Chewiness (g·mm)	Resilience (–)	Resistance to extension (g)	Extensibility (mm)
Control	5249±266 <sup>cd</sup>	0.903±0.035 <sup>a</sup>	0.636±0.060 <sup>b</sup>	3328±296 <sup>a</sup>	3012±345 <sup>a</sup>	0.045±0.002 <sup>a</sup>	60.24±3.21 <sup>a</sup>	25.44±0.77 <sup>a</sup>
HMT-10a	4801±300 <sup>def</sup>	0.919±0.020 <sup>a</sup>	0.597±0.054 <sup>bc</sup>	2859±212 <sup>b</sup>	2624±135 <sup>b</sup>	0.042±0.004 <sup>b</sup>	47.03±1.44 <sup>c</sup>	23.06±1.99 <sup>b</sup>
HMT-20a	4624±313 <sup>ef</sup>	0.923±0.012 <sup>a</sup>	0.568±0.063 <sup>dcd</sup>	26349±401 <sup>bc</sup>	2429±349 <sup>bc</sup>	0.038±0.003 <sup>cde</sup>	38.28±1.68 <sup>d</sup>	21.51±0.74 <sup>cd</sup>
HMT-30a	4513±257 <sup>f</sup>	0.914±0.028 <sup>a</sup>	0.532±0.033 <sup>cde</sup>	2403±215 <sup>c</sup>	2195±201 <sup>c</sup>	0.035±0.002 <sup>efg</sup>	33.85±2.18 <sup>e</sup>	21.34±2.84 <sup>cd</sup>
HMT-40a	4723±135 <sup>ef</sup>	0.879±0.053 <sup>ab</sup>	0.575±0.055 <sup>bcd</sup>	2713±284 <sup>bc</sup>	2415±316 <sup>bc</sup>	0.033±0.001 <sup>g</sup>	29.89±2.95 <sup>f</sup>	20.96±1.80 <sup>cd</sup>

Table 5.7 is continued

Dough samples	Dough TPA compressive test						Dough TPA tensile test	
	Hardness (g)	Springiness (mm)	Cohesiveness (–)	Gumminess (g)	Chewiness (g·mm)	Resilience (–)	Resistance to extension (g)	Extensibility (mm)
HMT-50a	4939±240 <sup>def</sup>	0.837±0.108 <sup>b</sup>	0.495±0.090 <sup>de</sup>	24283±366 <sup>e</sup>	2093±477 <sup>e</sup>	0.032±0.002 <sup>g</sup>	29.02±3.19 <sup>f</sup>	18.46±1.41 <sup>e</sup>
MW-10a	4691±284 <sup>ef</sup>	0.923±0.012 <sup>a</sup>	0.760±0.054 <sup>a</sup>	35433±157 <sup>a</sup>	3270±160 <sup>a</sup>	0.040±0.002 <sup>bc</sup>	52.38±2.29 <sup>b</sup>	21.86±1.41 <sup>bc</sup>
MW-20a	5003±256 <sup>de</sup>	0.875±0.055 <sup>ab</sup>	0.490±0.050 <sup>e</sup>	2476±304 <sup>bc</sup>	2175±397 <sup>e</sup>	0.040±0.001 <sup>bc</sup>	47.55±1.87 <sup>c</sup>	20.07±0.99 <sup>d</sup>
MW-30a	5551±293 <sup>c</sup>	0.607±0.013 <sup>c</sup>	0.508±0.037 <sup>de</sup>	2815±197 <sup>bc</sup>	1710±153 <sup>d</sup>	0.038±0.002 <sup>cd</sup>	38.66±2.51 <sup>d</sup>	18.68±1.02 <sup>e</sup>
MW-40a	6177±435 <sup>b</sup>	0.483±0.025 <sup>d</sup>	0.307±0.034 <sup>f</sup>	1890±164 <sup>d</sup>	912±85 <sup>e</sup>	0.037±0.002 <sup>def</sup>	34.57±1.82 <sup>e</sup>	18.38±0.96 <sup>e</sup>
MW-50a	6605±455 <sup>a</sup>	0.162±0.029 <sup>e</sup>	0.178±0.018 <sup>g</sup>	1171±105 <sup>e</sup>	192±53 <sup>f</sup>	0.034±0.002 <sup>fg</sup>	29.94±0.92 <sup>f</sup>	15.86±0.85 <sup>f</sup>

**Notes:** all values are the mean of at least triplicate determinations ± SD. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

The tensile properties reflect the strength and extensibility of dough. The resistance to extension reflects the longitudinal elasticity of the dough, and the greater resistance to extension reflects the stronger longitudinal elasticity of the dough. The extensibility reflects the transverse extensibility of the dough, and the greater extensibility reflects the better ductility [228]. It was observed that resistance to extension and extensibility of dough significantly decreased with the increase incorporation amount of HMTS or MWS (Table 5.7), which might due to incorporation of HMTS or MWS diluted the gluten protein in the mixed flour. At the same time, the filling of starch particles in dough hindered the formation of gluten network, which would weaken the combination between the components, thus reducing the energy required for tensile process. Therefore, incorporation of HMTS or MWS would significantly weaken the tensile characteristics (resistance to extension and extensibility) of the dough.

### 5.3.2 Cooking properties analysis of fresh noodles

The network structure of fresh and wet noodles and the degree of crosslinking of starch inside noodles directly affect the cooking properties of noodles. If the gluten network inside the noodles is poor and the crosslinking of starch is not tightly combined, it will cause the noodles to be lost during cooking process, or even the noodles will be broken [187]. Starch gelatinization and gluten

swelling have a certain effect on the dry matter water absorption of noodles, which mainly reflects the degree of hydration of protein and starch in noodles [229].

Table 5.8 showed that substitution wheat flour with HMT and MW modified potato starch (HMTS and MWS) significantly decreased the optimal cooking time of fresh noodles ( $P_{\text{value}} < 0.05$ ), moreover, the optimal cooking time of noodles with HMTS was lower than that of noodles with MWS when the substitution amount was same. HMTS and MWS weakened the binding strength between protein and starch, resulting in the formation of sparse structures in the noodles, making it easier for water to enter the interior of the molecules during cooking process, thereby reducing the optimal cooking time of noodles [151]. The dry matter water absorption rate is referred to the total amount of water that noodles can be absorb and the loss rate of dry matter is related to the resistance to disintegration during cooking process [230]. The dry matter water absorption rate and loss rate of dry matter significantly increased with the increase of substitution amount of HMTS and MWS. Moreover, both of the dry matter water absorption rate and loss rate of dry matter of noodles with HMTS were lower than that of noodles with MWS when the substitution amount was same, indicating noodles with HMTS had a better cooking quality than that of noodles with MWS in optimal cooking time condition. The increase in dry matter water absorption rate may due to the disruption of the double helical structure of amylopectin and the dissolution of amylose during starch gelatinization [231]. The added modified potato starch weakened the network structure in noodles, which might lead to the dissolution of water-soluble substances, thus increasing the loss rate of dry matter of noodles. Starch granules in noodles were over-expanded or even damaged during the cooking process, resulting in the increase of starch granules leaching, affecting the loss rate of dry matter, and even leading to the breakage of noodles. When the incorporation amount of HMTS was less than 30% and the incorporation amount of MWS was less than 20%, the noodles could remain intact without breaking.

**Table 5.8**

Effect of HMT and MW modified potato starch on cooking properties analysis  
of fresh noodles

Noodle Samples	Optimal cooking time (s)	Dry matter water absorption rate (%)	Loss rate of dry matter (%)	Cooking breakage rate (%)
Control	133.3±1.2 <sup>a</sup>	120.6±1.3 <sup>g</sup>	1.94±0.05 <sup>j</sup>	0.00±0.00 <sup>b</sup>
HMT-10a	121.5±2.5 <sup>b</sup>	122.5±0.3 <sup>f</sup>	2.98±0.06 <sup>i</sup>	0.00±0.00 <sup>b</sup>
HMT-20a	106.3±2.1 <sup>d</sup>	124.1±0.2 <sup>e</sup>	3.38±0.02 <sup>h</sup>	0.00±0.00 <sup>b</sup>
HMT-30a	87.3±0.6 <sup>f</sup>	125.3±0.3 <sup>e</sup>	4.39±0.04 <sup>g</sup>	0.00±0.00
HMT-40a	77.0±2.0 <sup>g</sup>	127.5±0.0 <sup>d</sup>	5.43±0.03 <sup>d</sup>	1.67±1.44 <sup>b</sup>
HMT-50a	75.0±1.0 <sup>g</sup>	135.5±1.9 <sup>b</sup>	6.00±0.05 <sup>c</sup>	1.67±1.44 <sup>b</sup>
MW-10a	124.3±2.1 <sup>b</sup>	122.7±1.2 <sup>f</sup>	3.40±0.10 <sup>h</sup>	0.00±0.00 <sup>b</sup>
MW-20a	118.3±0.6 <sup>c</sup>	127.2±0.6 <sup>d</sup>	4.51±0.06 <sup>f</sup>	0.00±0.00 <sup>b</sup>
MW-30a	103.7±1.5 <sup>d</sup>	128.4±0.9 <sup>d</sup>	5.00±0.10 <sup>e</sup>	1.67±1.44 <sup>b</sup>
MW-40a	97.3±2.5 <sup>e</sup>	130.6±0.4 <sup>c</sup>	7.13±0.05 <sup>b</sup>	1.67±1.44 <sup>b</sup>
MW-50a	97.7±2.5 <sup>e</sup>	147.4±0.7 <sup>a</sup>	8.18±0.04 <sup>a</sup>	5.00±0.00 <sup>a</sup>

**Notes:** all values are the mean of at least triplicate determinations ± SD. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

### 5.3.3 Texture and tensile analysis of fresh noodles

Texture of food is a key quality characteristic that determines its edible quality and consumer's acceptance. Textural properties of cooked fresh noodles were determined by TPA compressive test and tensile test in this study. The results (Table 5.9) showed that the control noodles made by 100% wheat flour had the best texture than that of noodles made by partial substitution of wheat flour with modified potato starch, which due to the gluten protein content of the starch-added flour was lower than that of original wheat flour. The incorporation of HMT and MW modified potato starch into noodles had some effects on the textural properties of cooked fresh noodles. The hardness and gumminess of cooked fresh noodles increased first and then decreased with the increase substitution amount of HMTS or MWS, the noodles with modified potato starch had the highest value of hardness and gumminess when the substitution amount of HMTS or MWS was 30% (HMT-30a and MW-30a, Table 5.9). Our previous research had confirmed

that appropriate MW and HMT modification could increase gel hardness of potato starch, therefore the increased hardness of noodles with certain amount of HMTS or MWS could attributed to the improved rigidity of starch gel. However, excessive addition of modified potato starch would reduce the gluten protein content in wheat flour, and would weaken the network of flour dough, resulting in poor texture properties of the noodles.

It was observed that tensile strength and elasticity of cooked fresh noodles decreased with the increase of the increase substitution amount of HMTS or MWS (Table 5.9), which due to the decrease of gluten protein content in wheat flour after adding modified potato starch. When the gluten protein content decreased, the stability of the gluten network formed by mixing and stirring wheat flour and water would be weakened, resulting in lower tensile strength and shorter stretching distance (lower elasticity value) [152].

**Table 5.9**

Effect of HMT and MW modified potato starch on texture and tensile properties of fresh noodles

Noodle Samples	TPA compressive test						TPA tensile test	
	Hardness (g)	Springiness (mm)	Cohesiveness (–)	Gumminess (g)	Chewiness (g·mm)	Resilience (–)	Tensile strength (g)	Elasticity (mm)
Control	4092±280 <sup>ab</sup>	0.90±0.03 <sup>ab</sup>	0.55±0.03 <sup>a</sup>	2267±218 <sup>a</sup>	2038±186 <sup>a</sup>	0.20±0.02 <sup>ab</sup>	21.63±1.50 <sup>a</sup>	46.34±2.49 <sup>a</sup>
HMT-10a	3157±297 <sup>d</sup>	0.92±0.03 <sup>a</sup>	0.55±0.04 <sup>a</sup>	1731±195 <sup>c</sup>	1591±191 <sup>c</sup>	0.20±0.02 <sup>ab</sup>	18.67±2.02 <sup>b</sup>	43.99±2.42 <sup>a</sup>
HMT-20a	3721±2511 <sup>c</sup>	0.92±0.03 <sup>a</sup>	0.54±0.05 <sup>ab</sup>	2017±255 <sup>b</sup>	1869±288 <sup>bc</sup>	0.21±0.02 <sup>a</sup>	17.78±0.68 <sup>bcd</sup>	43.36±2.03 <sup>a</sup>
HMT-30a	4272±565 <sup>a</sup>	0.89±0.03 <sup>abc</sup>	0.51±0.05 <sup>bcd</sup>	2175±158 <sup>a</sup>	1941±170 <sup>ab</sup>	0.19±0.02 <sup>abc</sup>	17.43±0.81 <sup>bcd</sup>	37.90±5.53 <sup>b</sup>
HMT-40a	4092±177 <sup>b</sup>	0.86±0.03 <sup>cd</sup>	0.50±0.03 <sup>def</sup>	2018±158 <sup>b</sup>	1735±163 <sup>d</sup>	0.18±0.03 <sup>abc</sup>	17.04±0.86 <sup>cd</sup>	33.05±2.61 <sup>de</sup>
HMT-50a	4001±149 <sup>b</sup>	0.84±0.05 <sup>de</sup>	0.49±0.03 <sup>def</sup>	1997±100 <sup>b</sup>	1687±130 <sup>de</sup>	0.17±0.04 <sup>c</sup>	15.45±1.01 <sup>ef</sup>	28.72±3.88 <sup>f</sup>
MW-10a	3667±225 <sup>c</sup>	0.88±0.02 <sup>bc</sup>	0.54±0.03 <sup>abc</sup>	1958±97 <sup>b</sup>	1729±101 <sup>d</sup>	0.20±0.03 <sup>ab</sup>	18.43±1.70 <sup>bc</sup>	36.82±2.25 <sup>bc</sup>
MW-20a	3988±187 <sup>b</sup>	0.88±0.04 <sup>bc</sup>	0.51±0.03 <sup>cdef</sup>	2020±100 <sup>b</sup>	1778±122 <sup>cd</sup>	0.19±0.02 <sup>abc</sup>	18.46±1.34 <sup>bc</sup>	34.54±2.95 <sup>cd</sup>
MW-30a	4294±312 <sup>a</sup>	0.82±0.04 <sup>c</sup>	0.48±0.04 <sup>f</sup>	2038±87 <sup>b</sup>	1672±126 <sup>de</sup>	0.18±0.02 <sup>bc</sup>	17.57±1.80 <sup>bcd</sup>	32.21±2.42 <sup>de</sup>
MW-40a	3737±235 <sup>c</sup>	0.90±0.02 <sup>ab</sup>	0.52±0.03 <sup>bcd</sup>	1934±128 <sup>b</sup>	1749±120 <sup>cd</sup>	0.20±0.04 <sup>ab</sup>	16.67±0.77 <sup>de</sup>	30.76±1.72 <sup>ef</sup>
MW-50a	3347±181 <sup>d</sup>	0.88±0.06 <sup>bc</sup>	0.49±0.02 <sup>df</sup>	1630±104 <sup>c</sup>	1436±127 <sup>f</sup>	0.17±0.03 <sup>c</sup>	14.71±1.36 <sup>f</sup>	25.27±4.88 <sup>g</sup>

**Notes:** all values are the mean of at least triplicate determinations ± SD. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

### **5.3.4 Correlation analysis between dough texture properties and noodles texture properties**

The correlation analysis between the TPA compressive texture properties of dough and noodles was investigated, and the results were showed in Table 5.10. It can be seen from Table 5.10 that there was strong correlation between the texture property indexes of dough. The dough hardness index had a significant or extremely significant negative correlation with the springiness, cohesiveness, gumminess and chewiness of the dough. The springiness, cohesiveness, gumminess and chewiness of dough were extremely significant positive correlated with each other. The gumminess and chewiness of cooked fresh noodles were extremely significant or significant positive correlated with the hardness of cooked fresh noodles, while the chewiness was extremely significant positive correlated with the gumminess. The resilience of cooked fresh noodles was significant positive correlated with the springiness and cohesiveness. However, except for the weak significant positive correlation between the springiness of dough and the gumminess and chewiness of cooked fresh noodles, the weak significant positive correlation between the chewiness of dough and the cohesiveness of cooked fresh noodles, the weak significant positive correlation between the resilience of dough and cohesiveness and resilience of cooked fresh noodles, there were no significant correlation between the dough and the cooked fresh noodles of the other texture properties indicators. These results might be related to the incorporation of HMTS or MWS. The incorporation of HMTS or MWS diluted the gluten protein in flour, making the dough unable to form good gluten network structure. Moreover, the noodles made with HMTS or MWS absorbed more water than the control noodles (Table 5.8, dry matter water absorption rate) in the cooking process, and the starch granules were easy to dissolve (Table 5.8, loss rate of dry matter), which might cause the texture properties of cooked fresh noodles to be different from that of the control noodles (Table 5.9), which might also be the reason that there was no obvious correlation

between the texture property indicators of cooked fresh noodles and that of the dough.

### **5.3.5 Correlation analysis between dough tensile properties and noodles tensile properties**

Although there was no obvious correlation between the texture property indicators of cooked fresh noodles and that of the dough, the dough tensile properties of resistance to extension and extensibility were extremely significant positive correlated with the cooked fresh noodles tensile properties of tensile strength and elasticity (Table 5.11). The results of correlation analysis between the TPA tensile properties of dough and noodles indicated that the tensile properties of dough could be used to evaluate the tensile properties of cooked fresh noodles and could also reflect the quality of the cooked fresh noodles.

**Table 5.10****Correlation analysis between dough tensile properties and noodles tensile properties**

Correlation coefficients		TPA compressive test of dough						TPA compressive test of noodles					
		Hardness (g)	Springiness (mm)	Cohesiveness (–)	Gumminess (g)	Chewiness (g·mm)	Resilience (–)	Hardness (g)	Springiness (mm)	Cohesiveness (–)	Gumminess (g)	Chewiness (g·mm)	Resilience (–)
TPA compressive test of dough	Hardness (g)	1											
	Springiness (mm)	-0.95**	1										
	Cohesiveness (–)	-0.84**	0.89**	1									
	Gumminess (g)	-0.70*	0.80**	0.97**	1								
	Chewiness (g·mm)	-0.86**	0.93**	0.98**	0.94**	1							
	Resilience (–)	-0.11	0.32	0.46	0.59*	0.5	1						
TPA compressive test of noodles	Hardness (g)	-0.27	0.28	0.21	0.25	0.19	-0.17	1					
	Springiness (mm)	-0.10	0.18	0.04	-0.02	0.14	0.42	-0.56	1				
	Cohesiveness (–)	-0.40	0.55	0.55	0.54	0.63*	0.70*	-0.35	0.81**	1			
	Gumminess (g)	-0.48	0.58*	0.50	0.54	0.53	0.22	0.85**	-0.12	0.2	1		
	Chewiness (g·mm)	-0.50	0.63*	0.50	0.51	0.57	0.36	0.61*	0.27	0.5	0.92**	1	
	Resilience (–)	-0.27	0.42	0.40	0.43	0.44	0.67*	-0.22	0.74**	0.87**	0.25	0.52	1

Notes: \* indicates correlation is significant at  $P_{\text{value}} < 0.05$ ; \*\* indicates correlation is significant at  $P_{\text{value}} < 0.01$ .



**Table 5.11** Correlation analysis between dough tensile properties and noodles tensile properties

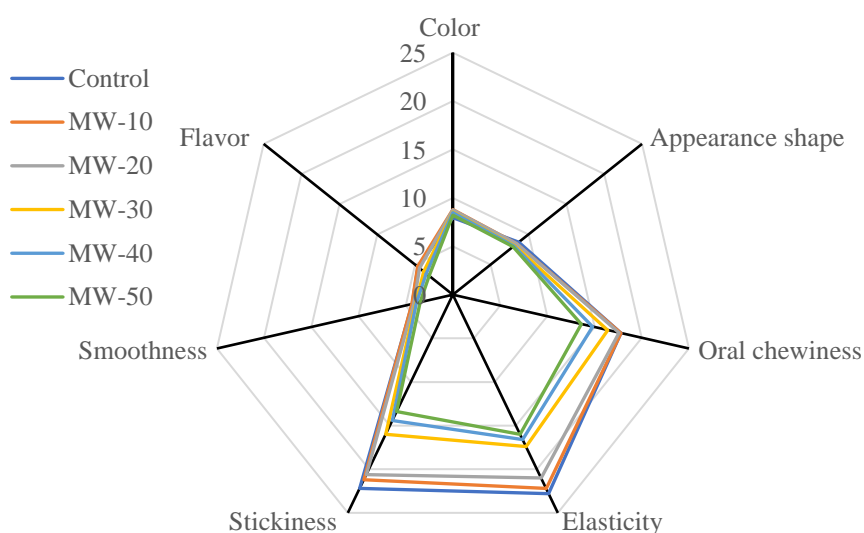
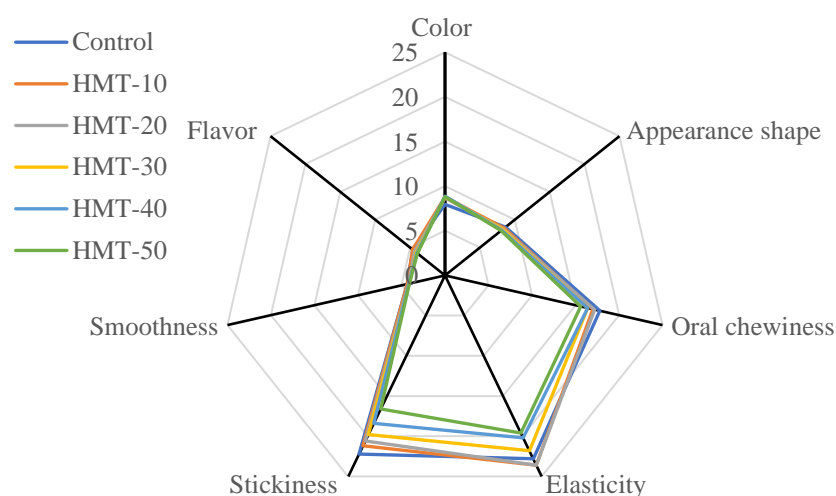
Correlation coefficients	Resistance to extension	to Extensibility	Tensile strength	Elasticity
Resistance to extension	1			
Extensibility	0.75**	1		
Tensile strength	0.91**	0.90**	1	
Elasticity	0.71**	0.93**	0.85**	1

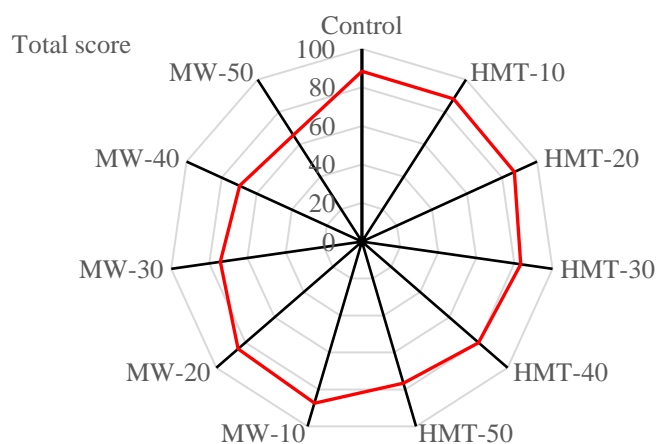
Notes: \*\* indicates correlation is significant at  $P_{\text{value}} < 0.01$ .

### 5.3.6 Sensory evaluation of fresh noodles

The sensory evaluation of fresh noodles consisted seven components- color, appearance shape, oral chewiness, elasticity, stickiness, smoothness and flavor, and the individual scores were added together to obtain the total score (Fig. 5.6). The incorporation of HMTS or MWS had different effects on the sensory evaluation indicators of noodles. Incorporation of HMTS or MWS increased the color score of noodles, while the appearance shape had lower score. According to the scoring method for sensory evaluation of fresh noodles (Table 2.1) and the results of sensory evaluation (Fig. 5.6), the appearance shape scores of all the noodles with modified potato starch were higher than 8.0 except the sample of MW-50a, indicating that the appearance shape of noodles after incorporating of HMTS or MWS was still acceptable for consumers. Incorporation of small amount of HMTS ( $\leq 20\%$ ) or MWS ( $\leq 10\%$ ) would not significantly change the score of oral chewiness, elasticity, smoothness and total score of noodles. The other sensory evaluation indicators, including oral chewiness, elasticity, stickiness, smoothness and flavor, decreased with the increase substitution amount of HMTS ( $>30\%$ ) or MWS ( $>20\%$ ), and similarly, the total score of sensory evaluation also decreased. Increasing HMTS incorporation level decreased the total score of fresh noodles from 88.06 to 76.68, increasing MWS incorporation level decreased the total scores of fresh noodles from 87.46 to 65.78, whereas the control fresh noodles had total score of 88.38. In terms of general acceptability, there was no significant difference between the samples of HMTS-10a, HMT-20a, MWS-10a

and the control fresh noodle, the sample MW-50a was the least acceptable. For all sensory attributes, total score of 80 was considered as the limit of acceptability. Thus, the maximum incorporation of HMTS should not be exceeded 40%, and incorporation of MWS should not be exceeded 20%. According to the results of sensory evaluation and cooking properties analysis (Table 5.8, cooking breakage rate) of fresh noodles, the optimal incorporation of HMTS and MWS was 30% and 20%, respectively. The noodles made with 30 % HMTS or 20% MWS could remain intact without breaking and had good acceptability.





**Fig.5.6** Effect of HMT and MW modified potato starch on sensory scores of fresh noodles

### 5.3.7 Postprandial blood glucose levels of fresh noodles

The postprandial blood glucose levels of participants at different times after eating fresh noodles were showed in Table 5.12. The fasting blood glucose levels (Table 5.12, 0 min) of all participants were within the normal range, and there was no significant difference between them ( $P_{\text{value}} > 0.05$ ). The change trends of postprandial blood glucose levels before and after eating fresh noodles were the same, with an increase followed by a decrease. After eating the control fresh noodles, the blood glucose levels increased quickly and reached its peak value (7.41 mmol/L) at 30 min, and then the blood glucose levels decreased rapidly. The postprandial blood glucose levels fluctuated greatly. The rates of increase in postprandial blood glucose after eating fresh noodles with more than 10% incorporation of HMTS or MWS were slower than that of the control fresh noodles. The postprandial blood glucose reached their peak values at 45 min after eating, and the peak values were significantly lower than that of the control fresh noodles, and then the blood glucose levels decreased slowly. Moreover, postprandial blood glucose levels of participants at different times after eating fresh noodles with incorporation of HMTS or MWS were lower than that of the control fresh noodles. All these results indicated that fresh noodles with incorporation of HMTS or MWS were more suitable for diabetics or the elderly.

Postprandial blood glucose levels of participants at different times after eating  
fresh noodles

Noodle Samples	Postprandial blood glucose levels (mmol/L)						
	0 min	15 min	30 min	45 min	60 min	90 min	120 min
Control	4.75±0.07 <sup>a</sup>	6.36±0.02 <sup>a</sup>	7.41±0.06 <sup>a</sup>	7.28±0.07 <sup>a</sup>	6.77±0.05 <sup>a</sup>	6.40±0.06 <sup>a</sup>	5.39±0.07 <sup>a</sup>
HMT-10a	4.67±0.05 <sup>a</sup>	6.26±0.02 <sup>b</sup>	6.83±0.02 <sup>c</sup>	7.06±0.03 <sup>c</sup>	6.61±0.03 <sup>b</sup>	6.09±0.07 <sup>c</sup>	5.29±0.03 <sup>b</sup>
HMT-20a	4.68±0.04 <sup>a</sup>	5.73±0.04 <sup>d</sup>	6.58±0.06 <sup>c</sup>	6.72±0.04 <sup>ef</sup>	6.38±0.03 <sup>c</sup>	5.83±0.05 <sup>c</sup>	5.26±0.02 <sup>bc</sup>
HMT-30a	4.67±0.05 <sup>a</sup>	5.42±0.11 <sup>ef</sup>	6.42±0.04 <sup>f</sup>	6.63±0.05 <sup>gh</sup>	6.29±0.02 <sup>cd</sup>	5.73±0.05 <sup>f</sup>	5.21±0.03 <sup>cde</sup>
HMT-40a	4.74±0.06 <sup>a</sup>	5.19±0.05 <sup>g</sup>	6.30±0.07 <sup>g</sup>	6.59±0.05 <sup>hi</sup>	6.24±0.02 <sup>de</sup>	5.42±0.03 <sup>h</sup>	5.18±0.04 <sup>de</sup>
HMT-50a	4.69±0.04 <sup>a</sup>	5.08±0.03 <sup>h</sup>	6.21±0.03 <sup>h</sup>	6.53±0.04 <sup>i</sup>	6.16±0.03 <sup>ef</sup>	5.30±0.04 <sup>i</sup>	5.01±0.06 <sup>fg</sup>
MW-10a	4.68±0.01 <sup>a</sup>	6.15±0.02 <sup>c</sup>	6.91±0.02 <sup>b</sup>	7.13±0.03 <sup>b</sup>	6.57±0.16 <sup>b</sup>	6.22±0.01 <sup>b</sup>	5.24±0.02 <sup>bcd</sup>
MW-20a	4.70±0.06 <sup>a</sup>	5.46±0.03 <sup>c</sup>	6.75±0.06 <sup>d</sup>	6.88±0.01 <sup>d</sup>	6.54±0.07 <sup>b</sup>	5.94±0.04 <sup>d</sup>	5.15±0.03 <sup>c</sup>
MW-30a	4.69±0.04 <sup>a</sup>	5.37±0.05 <sup>f</sup>	6.62±0.04 <sup>e</sup>	6.73±0.04 <sup>e</sup>	6.28±0.04 <sup>cd</sup>	5.84±0.04 <sup>c</sup>	5.04±0.02 <sup>f</sup>
MW-40a	4.73±0.03 <sup>a</sup>	5.25±0.03 <sup>g</sup>	6.42±0.03 <sup>f</sup>	6.67±0.04 <sup>efg</sup>	6.10±0.06 <sup>fg</sup>	5.60±0.04 <sup>g</sup>	5.05±0.01 <sup>f</sup>
MW-50a	4.68±0.04 <sup>a</sup>	5.11±0.06 <sup>h</sup>	6.25±0.03 <sup>gh</sup>	6.65±0.03 <sup>fgh</sup>	6.01±0.02 <sup>g</sup>	5.40±0.04 <sup>h</sup>	4.95±0.06 <sup>g</sup>

**Notes:** all values are the mean of at least triplicate determinations ± SD. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

Based on the results of cooking properties analysis, texture and tensile analysis and sensory evaluation of fresh noodles, it was concluded that the optimal substitution amount of wheat flour with HMTS or MWS was 30%, 20 %, respectively. Therefore, the optimal recipe of fresh noodles made with incorporation of HMTS was as follow: 70 g wheat flour (the protein content was 10.0%±1.0%, 12% moisture content), 30 g HMTS (12% moisture content), salt 0.48 g and water 48 g; the optimal recipe of fresh noodles made with incorporation of MWS was as follow: 80 wheat flour (the protein content was 10.0%±1.0%, 12% moisture content), 20g MWS (12% moisture content), salt 0.48 g and water 48 g.

#### 5.4 Substitution of wheat flour with modified potato starch affects the quality of steamed bread

The basic recipe of control of steamed bread was 100 g wheat flour (the protein content was 10.0%±1.0%, 12% moisture content), 55 g water, 1.0 g yeast and 1.0 g salt. Wheat flour was replaced by HMTS or MWS at the levels of 10%,

20%, 30%, 40% and 50%. When the content of HMTS or MWS was more than 50%, the dough with strong network structure could not be formed due to too little gluten, thus the maximum substitution of HMTS and MWS was set as 50%.

According to the different substitution of HMTS (10%, 20%, 30%, 40% and 50%), the experimental steamed bread buns were named as HMT-10b, HMT-20b, HMT-30b, HMT-40b and HMT-50b. Similarly, according to the different substitution of MWS (10%, 20%, 30%, 40% and 50%), the experimental steamed bread buns were named as MW-10b, MW-20b, MW-30b, MW-40b and MW-50b. steamed bread made entirely of wheat flour without HMTS and MWS were used as control.

#### **5.4.1 Color analysis of steamed bread**

Table 5.13 presented the results of experiments evaluating the effect of partial substitution of wheat flour with HMTS or MWS on the crust and core colors of steamed bread. The results showed that for both crust and core, as the amount of added HMTS and MWS increased, the lightness value ( $L^*$ ) and the red-green value ( $a^*$ ) increased, the yellow-blue value ( $b^*$ ) decreased, which indicated that as the lightness of steamed bread increased, the red value increased, and the transparent color became lighter. As an intuitive indicator of food, color is one of the most important quality indicators steamed bread and plays a decisive role in consumption and popularity [232]. A higher  $L^*$  value is generally as an indicator of better acceptance and quality [233]. In this study, although the  $L^*$  value of both crust and core of steamed bread increased with more incorporation of HMTS or MWS, and there was no significantly difference color score of sensory evaluation between wheat flour steamed bread and all steamed bread containing modified potato starch (HMTS or MWS) except with 50% substitution (HMT-50b and MW-50b), which indicated that consumers pay more attention to their health than food color preference.

In general, MWS had greater effect on the crust and core colors of the steamed bread than that of HMTS. The color differences ( $\Delta E$ ) between each experimental steamed bread and the control steamed bread increased with the

increase of substitution levels of HMTS or MWS. Moreover, when the substitution levels of HMTS were higher than 30% or MWS was higher than 20%, the color differences ( $\Delta E$ ) between each experimental steamed bread and the control steamed bread was above 3, indicating that the differences in color between the control and experimental steamed bread were detectable by the human eye.

**Table 5.13**

Effect of various levels of HMTS and MWS on color of the steamed bread

Steamed bread Samples	Crust				Core			
	L*	a*	b*	$\Delta E$	L*	a*	b*	$\Delta E$
Control	82.63±0.63 <sup>c</sup>	2.05±0.07 <sup>d</sup>	11.84±0.45 <sup>a</sup>	-	79.02±0.42 <sup>g</sup>	2.02±0.07 <sup>f</sup>	12.81±0.62 <sup>a</sup>	-
HMT-10b	83.00±0.86 <sup>c</sup>	2.07±0.04 <sup>d</sup>	10.62±0.24 <sup>b</sup>	1.49±0.05 <sup>h</sup>	79.27±0.38 <sup>ig</sup>	2.05±0.07 <sup>ef</sup>	12.26±0.55 <sup>b</sup>	0.65±0.62 <sup>i</sup>
HMT-20b	83.10±0.59 <sup>c</sup>	2.10±0.03 <sup>d</sup>	10.40±0.41 <sup>b</sup>	1.57±0.51 <sup>gh</sup>	79.65±0.43 <sup>f</sup>	2.08±0.04 <sup>ef</sup>	11.70±0.24 <sup>cd</sup>	1.35±0.11 <sup>h</sup>
HMT-30b	84.99±0.37 <sup>cd</sup>	2.17±0.08 <sup>cd</sup>	10.13±0.54 <sup>bc</sup>	2.97±0.12 <sup>d</sup>	80.72±0.48 <sup>e</sup>	2.13±0.03 <sup>de</sup>	11.26±0.23 <sup>d</sup>	2.34±0.19 <sup>f</sup>
HMT-40b	84.48±0.25 <sup>d</sup>	2.26±0.06 <sup>bc</sup>	9.48±0.18 <sup>d</sup>	3.01±0.21 <sup>d</sup>	82.03±0.08 <sup>d</sup>	2.35±0.03 <sup>c</sup>	10.23±0.33 <sup>ef</sup>	3.98±0.18 <sup>e</sup>
HMT-50b	85.27±0.26 <sup>c</sup>	2.44±0.07 <sup>a</sup>	9.37±0.16 <sup>d</sup>	3.65±0.11 <sup>c</sup>	83.76±0.28 <sup>b</sup>	2.46±0.04 <sup>b</sup>	9.24±0.18 <sup>e</sup>	5.95±0.27 <sup>b</sup>
MW-10b	84.38±0.22 <sup>d</sup>	2.08±0.08 <sup>d</sup>	11.53±0.27 <sup>a</sup>	1.80±0.19 <sup>g</sup>	80.68±0.28 <sup>e</sup>	2.07±0.03 <sup>ef</sup>	12.29±0.36 <sup>b</sup>	1.79±0.14 <sup>g</sup>
MW-20b	84.51±0.08 <sup>d</sup>	2.12±0.03 <sup>d</sup>	11.30±0.44 <sup>a</sup>	1.99±0.14 <sup>f</sup>	81.16±0.27 <sup>c</sup>	2.16±0.09 <sup>d</sup>	11.81±0.45 <sup>bc</sup>	2.40±0.19 <sup>f</sup>
MW-30b	84.36±1.09 <sup>d</sup>	2.16±0.08 <sup>cd</sup>	10.31±0.66 <sup>b</sup>	2.58±0.11 <sup>e</sup>	83.22±0.16 <sup>c</sup>	2.32±0.04 <sup>c</sup>	10.43±0.38 <sup>c</sup>	4.85±0.19 <sup>d</sup>
MW-40b	86.54±0.21 <sup>b</sup>	2.35±0.07 <sup>ab</sup>	9.68±0.55 <sup>cd</sup>	4.50±0.10 <sup>b</sup>	83.47±0.25 <sup>bc</sup>	2.32±0.09 <sup>c</sup>	9.89±0.37 <sup>f</sup>	5.34±0.11 <sup>c</sup>
MW-50b	87.35±0.27 <sup>a</sup>	2.41±0.25 <sup>a</sup>	9.26±0.45 <sup>d</sup>	5.41±0.26 <sup>a</sup>	86.07±0.48 <sup>a</sup>	2.54±0.05 <sup>a</sup>	8.92±0.34 <sup>g</sup>	8.08±0.31 <sup>a</sup>

**Notes:** all values are the mean of at least triplicate determinations  $\pm$  SD. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

#### 5.4.2 Specific volume analysis of steamed bread

The specific volume of steamed bread is an important quality parameter and is related to the gas capacity and elasticity of steamed bread. A high steamed bread specific volume represents a better product appearance [234]. Moreover, it has been reported increased gel surface area and porosity would support the more action sites for amylase, therefore high specific volume implies high starch digestibility [235].

The specific volume and colors of the steamed bread are shown in Table 5.14. The specific volume of steamed bread decreased with the increase of substitution

levels of HMTS and MWS, which was significantly lower than that of the control steamed bread. All the steamed bread containing modified potato starch (HMTS or MWS) demonstrated an observably lower specific volume compared with the wheat flour steamed bread, indicating that steamed bread made with modified potato starch had lower digestibility and was more suitable for diabetics or obese people. This speculation was consistent with our previous research results that HMTS and MWS contained more SDS and RS. The specific volume of control steamed bread (Table 5.14, Control) was 2.82 mL/g, while the lowest specific volume of steamed bread was 1.85 mL/g (Table 5.14, MW-50b). The smaller specific volume indicated the denser tighter crumb structure, and the higher hardness values were observed in the subsequent research of TPA compressive test (Table 5.14). Compared with HMTS, MWS had greater effect on the specific volume of steamed bread, and the specific volume of MW- steamed bread was lower than that of HMT- steamed bread at the same substitution level. The specific volume of steamed bread is positively related to the amount of gluten and gas hold capacity during fermentation [155]. Strong gluten network structure is essential for the gas cell in dough to provide expansion strength during fermentation and steaming. Substitution of wheat flour with HMTS and MWS reduced the amount of gluten in mixed flour and diluted gluten network of the dough, the mixed dough cannot form strong network structure needed for gas retention. The dilution also affected the formation of an elastic network during steaming, resulting in easy weaken of the gluten network and lower specific volume of steamed bread [149].

#### **5.4.3 Texture analysis of steamed bread**

The texture properties of food mainly refer to its tissue characteristics and this quality is related to the sensory and edible properties of food [232]. The hardness, gumminess and chewiness are negatively correlated with the quality of steamed bread, while springiness, cohesiveness and resilience are positively correlated with the quality of steamed bread. The texture results of steamed bread are shown in Table 5.14.

Effect of various levels of HMTS and MWS on the specific volume and textural properties of steamed bread.

Sample	Specific volume (mL/g)	Hardness (g)	Springiness (mm)	Cohesiveness (-)	Gumminess (g)	Chewiness (g·mm)	Resilience (-)
Control	2.82±0.14 <sup>a</sup>	367±19 <sup>j</sup>	0.97±0.01 <sup>a</sup>	0.86±0.01 <sup>a</sup>	314±17 <sup>j</sup>	303±15 <sup>h</sup>	0.48±0.01 <sup>ab</sup>
HMT-10b	2.64±0.05 <sup>b</sup>	416±15 <sup>i</sup>	0.97±0.01 <sup>a</sup>	0.84±0.02 <sup>a</sup>	348±17 <sup>ij</sup>	338±17 <sup>gh</sup>	0.48±0.01 <sup>ab</sup>
HMT-20b	2.47±0.04 <sup>c</sup>	547±28 <sup>h</sup>	0.96±0.01 <sup>a</sup>	0.80±0.02 <sup>b</sup>	437±16 <sup>h</sup>	418±15 <sup>f</sup>	0.46±0.02 <sup>bc</sup>
HMT-30b	2.38±0.07 <sup>cd</sup>	688±22 <sup>f</sup>	0.96±0.01 <sup>a</sup>	0.79±0.01 <sup>bc</sup>	542±20 <sup>f</sup>	518±18 <sup>e</sup>	0.45±0.00 <sup>c</sup>
HMT-40b	2.30±0.07 <sup>de</sup>	1375±15 <sup>c</sup>	0.91±0.02 <sup>c</sup>	0.70±0.01 <sup>d</sup>	967±14 <sup>c</sup>	881±24 <sup>b</sup>	0.39±0.01 <sup>e</sup>
HMT-50b	1.99±0.10 <sup>f</sup>	1685±29 <sup>a</sup>	0.88±0.03 <sup>d</sup>	0.67±0.02 <sup>e</sup>	1129±51 <sup>a</sup>	999±59 <sup>a</sup>	0.38±0.01 <sup>e</sup>
MW-10b	2.61±0.11 <sup>b</sup>	442±21 <sup>i</sup>	0.96±0.01 <sup>a</sup>	0.84±0.01 <sup>a</sup>	372±15 <sup>i</sup>	359±15 <sup>g</sup>	0.48±0.01 <sup>a</sup>
MW-20b	2.46±0.10 <sup>c</sup>	620±25 <sup>g</sup>	0.93±0.01 <sup>b</sup>	0.79±0.01 <sup>bc</sup>	487±21 <sup>g</sup>	455±17 <sup>f</sup>	0.42±0.01 <sup>d</sup>
MW-30b	2.27±0.03 <sup>de</sup>	840±43 <sup>e</sup>	0.93±0.02 <sup>b</sup>	0.76±0.04 <sup>c</sup>	639±22 <sup>e</sup>	596±24 <sup>d</sup>	0.42±0.03 <sup>d</sup>
MW-40b	2.22±0.07 <sup>e</sup>	995±44 <sup>d</sup>	0.92±0.01 <sup>bc</sup>	0.73±0.03 <sup>d</sup>	722±21 <sup>d</sup>	664±21 <sup>c</sup>	0.41±0.02 <sup>d</sup>
MW-50b	1.85±0.04 <sup>g</sup>	1634±58 <sup>b</sup>	0.89±0.02 <sup>d</sup>	0.66±0.03 <sup>e</sup>	1085±58 <sup>b</sup>	963±55 <sup>a</sup>	0.37±0.02 <sup>e</sup>

**Notes:** all values are the mean of at least triplicate determinations ± SD. The means within the same column with different letters are significantly different ( $P_{\text{value}} < 0.05$ ).

The results showed that the hardness, gumminess and chewiness increased significantly with the increase of substitution levels of HMTS or MWS. As a major indicator of textural properties, hardness is the force required to resist deformation, a small value of hardness implies a fluffy texture of steamed bread. The incorporation of HMTS from 10% to 50% led to the increase of hardness from 416 to 1685 g, and the incorporation of MWS from 10% to 50% led to the increase of hardness from 442 to 1634g, whereas the hardness of control steamed bread was 367 g (Table 5.14). The increased hardness implied a firm texture of steamed bread, which could be largely attributed to the gluten dilution effect from the incorporation of HMTS or MWS. Chewiness refers to the energy required to break down food into small pieces by mastication [236]. In this study, the chewiness of steamed bread with HMTS incorporation increased from 338 to 999 and the chewiness of steamed bread with MWS incorporation increased from 359 to 963, whereas the chewiness of control steamed bread was 303. The increased chewiness reflected the denser structure of steamed bread with HMTS or MWS



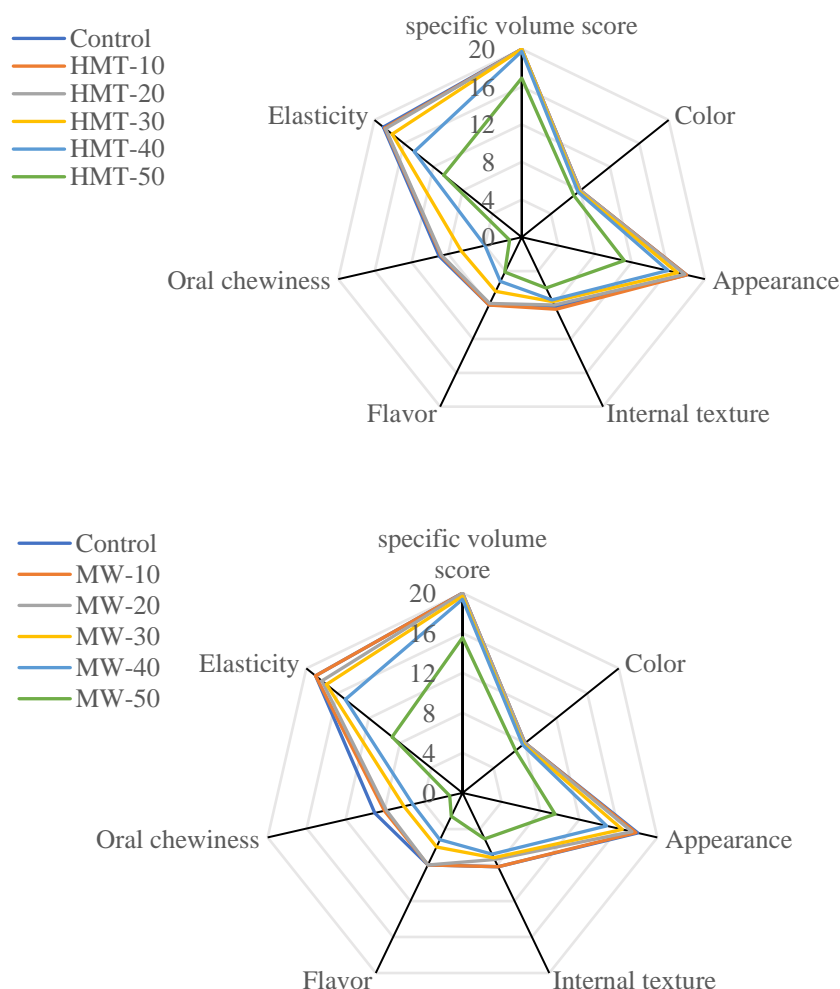
incorporation that required more energy and longer time for oral processing before swallowing. The reduced cohesiveness of HMT- steamed bread (from 0.84 to 0.67) or MW- steamed bread (from 0.84-0.66) indicated the disruption of steamed bread microstructure due to the diluted gluten matrix by the HMTS or MWS incorporation. The reduction in springiness of HMT- steamed bread (from 0.97 to 0.88) or MW- steamed bread (0.96 to 0.89) characterized the loss of elasticity, which could due to the reduction amounts of gluten effect form the HMTS or MWS incorporation and low leavening property, leading to the structure breaking of dough.

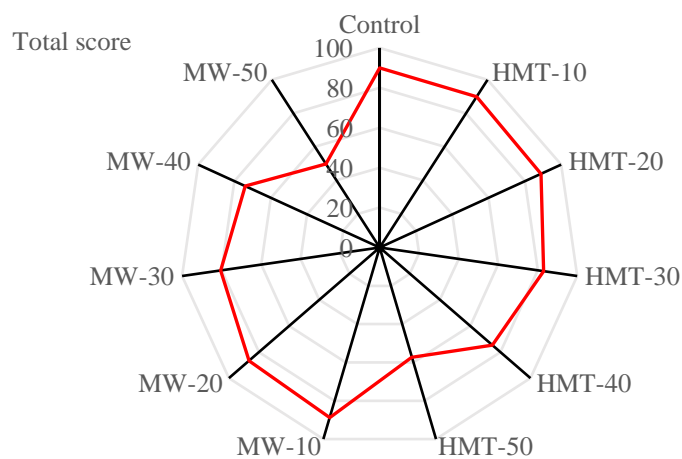
#### **5.4.4 Sensory evaluation of steamed bread**

Sensory evaluation directly reflects the acceptability of food. HMTS or MWS replacement affected different sensory quality attributes of steamed bread to different degrees (Fig. 5.7). Increasing HMTS incorporation level decreased the total score of steamed bread from 89.9 to 52.7, increasing MWS incorporation level decreased the total score of steamed bread from 88.8 to 49.8. The total score of steamed bread with 10% (HMT-10) and 20% (HMT-20) HMTS was 89.9 and 88.8 respectively, which was similar to that of the control sample (90.0), whereas steamed bread with 10% MWS (MW-10) was 88.8. The steamed bread with 50% MWS had the lowest total score of 49.8, while the total score of steamed bread with 50% HMTS was 57.2 (Fig. 5.7). For all sensory attributes, total score of 80 was considered as the limit of acceptability. From Fig. 5.7, We could conclude that steamed bread can be accepted by consumers when the substitution level of wheat flour with HMTS or MWS was no more than 30%. When the replacement levels of HMTS or MWS exceed 40 %, the oral sensation of steamed bread deteriorated sharply, and the internal structure of steamed bread become too hard and too firm, making it difficult to swallow. As the overall acceptability of products was a subjective testing, a large number of sensory panelists should be used in the future for commercial applications.

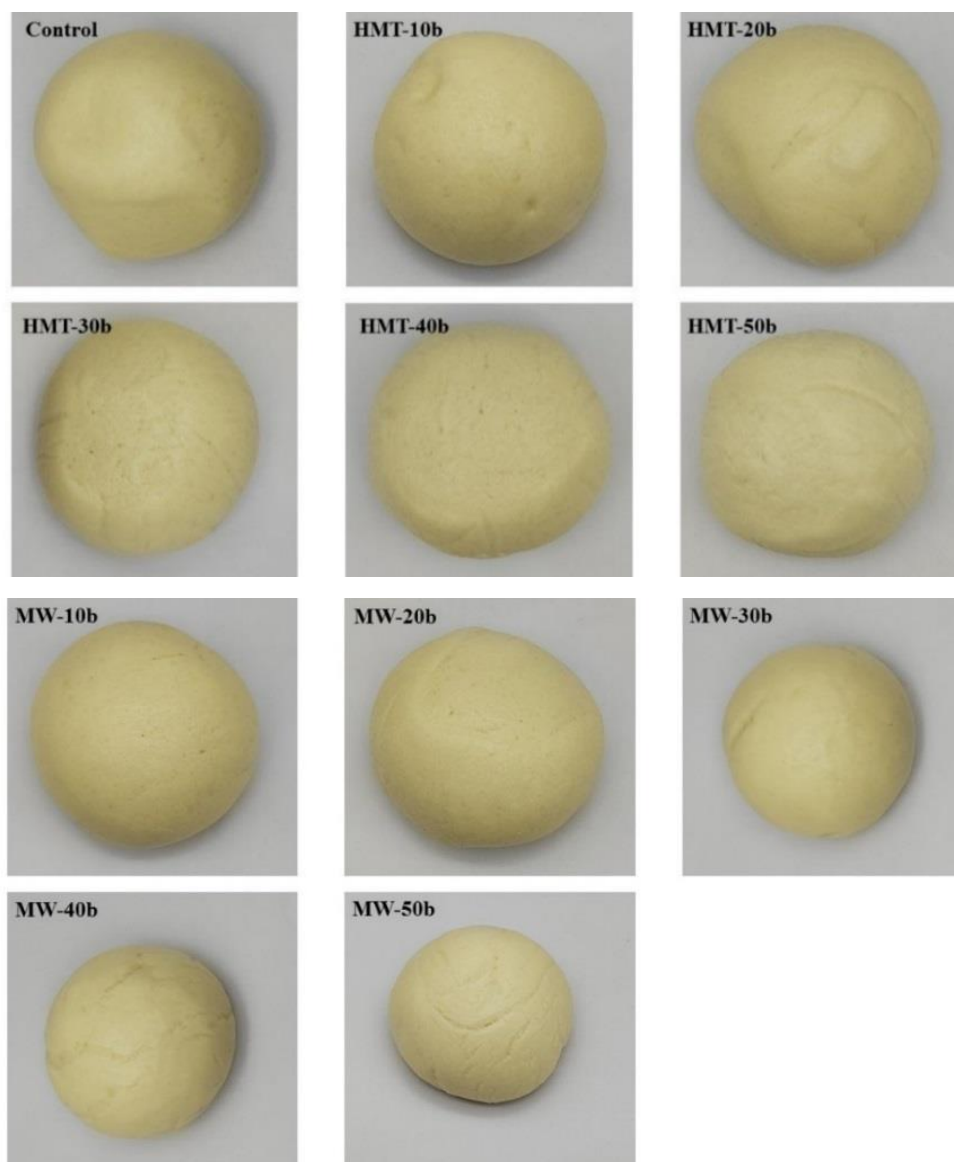
It is worth noting that there is a great diversity in steamed bread texture by Chinese consumers preference [221, 236]. The altered texture of steamed bread

due to HMTS or MWS incorporation (e.g. increased hardness, or decreased springiness, Table 5.14) may be a textural advantage to some consumers as indicated by sensory evaluation. The decreased appearance scores of HMT-steamed bread and MW-steamed bread could be attributed to the increased occurrence of cracks and surface roughness (Fig. 5.8). Moreover, the incorporation of HMTS or MWS reduced gluten content and diluted gluten network of the dough, which affected the formation of elastic network during steaming and reduced oral chewiness and elasticity of steamed bread during mastication, eventually resulting in low oral chewiness and elasticity scores. This agreed with the results of textural profile analysis as described in section 5.4.3 above. In addition, incorporation of HMTS or MWS made the surfaces of steamed bread become rougher, and even appeared a few specks or bubbles on the surface. The appearance scores of the sensory evaluation were consistent with the results shown in the photos (Fig. 5.8).





**Fig.5.7** Effect of HMT and MW modified potato starch on sensory scores of steamed breads



**Fig. 5.8** Photos of steamed bread with incorporation of HMTS or MWS at different substitution levels

#### 5.4.5 Postprandial blood glucose levels of steamed bread

The postprandial blood glucose levels of participants at different times after eating steamed breads were showed in Table 5.15. The fasting blood glucose levels (Table 5.15, 0 min) of all participants were within the normal range, and there was no significant difference between them ( $P_{\text{value}} > 0.05$ ). The change trends of postprandial blood glucose levels before and after eating steamed bread were the same, with an increase followed by a decrease. After eating the control steamed bread, the blood glucose levels increased quickly and reached its peak value (7.54 mmol/L) at 30 min, and then the blood glucose levels decreased rapidly. The postprandial blood glucose levels fluctuated greatly. The rates of increase in postprandial blood glucose after eating steamed bread with more than 10% incorporation of HMTS or MWS were slower than that of the control steamed breads. The postprandial blood glucose reached their peak values at 45 min after eating, and the peak values were significantly lower than that of the control steamed breads, and then the blood glucose levels decreased slowly. Moreover, postprandial blood glucose levels of participants at different times after eating steamed bread with incorporation of HMTS or MWS were lower than that of the control steamed bread. All these results indicated that steamed breads with incorporation of HMTS or MWS were more suitable for diabetics or the elderly.

**Table 5.15**

Postprandial blood glucose levels of participants at different times after eating steamed bread

Steamed bread samples	Postprandial blood glucose levels (mmol/L)						
	0 min	15 min	30 min	45 min	60 min	90 min	120 min
Control	4.65±0.03 <sup>a</sup>	6.24±0.03 <sup>a</sup>	7.54±0.03 <sup>a</sup>	7.32±0.01 <sup>a</sup>	6.84±0.03 <sup>a</sup>	6.43±0.02 <sup>a</sup>	5.29±0.03 <sup>a</sup>
HMT-10b	4.65±0.04 <sup>a</sup>	6.13±0.08 <sup>b</sup>	6.82±0.10 <sup>bc</sup>	7.03±0.03 <sup>b</sup>	6.66±0.07 <sup>b</sup>	6.32±0.01 <sup>b</sup>	5.23±0.02 <sup>c</sup>
HMT-20b	4.67 ±0.02 <sup>a</sup>	5.56±0.03 <sup>c</sup>	6.77±0.03 <sup>cd</sup>	6.85±0.04 <sup>c</sup>	6.57±0.01 <sup>c</sup>	5.96±0.02 <sup>c</sup>	5.19±0.02 <sup>c</sup>
HMT-30b	4.64±0.01 <sup>a</sup>	5.48±0.00 <sup>d</sup>	6.63±0.04 <sup>c</sup>	6.75±0.02 <sup>d</sup>	6.42±0.04 <sup>de</sup>	5.86±0.02 <sup>d</sup>	5.15±0.01 <sup>c</sup>
HMT-40b	4.63±0.03 <sup>a</sup>	5.37±0.03 <sup>e</sup>	6.50±0.02 <sup>f</sup>	6.66±0.04 <sup>e</sup>	6.34±0.05 <sup>f</sup>	5.71±0.04 <sup>f</sup>	5.09±0.01 <sup>fg</sup>
HMT-50a	4.66±0.03 <sup>a</sup>	5.24±0.03 <sup>f</sup>	6.43±0.02 <sup>f</sup>	6.57±0.09 <sup>f</sup>	6.23±0.02 <sup>g</sup>	5.43±0.03 <sup>i</sup>	5.07±0.01 <sup>gh</sup>
MW-10b	4.66±0.01 <sup>a</sup>	6.15±0.02 <sup>b</sup>	6.87±0.02 <sup>b</sup>	7.05±0.02 <sup>b</sup>	6.65±0.03 <sup>b</sup>	6.29±0.01 <sup>b</sup>	5.22±0.01 <sup>b</sup>
MW-20b	4.64±0.04 <sup>a</sup>	5.50±0.03 <sup>cd</sup>	6.72±0.03 <sup>d</sup>	6.89±0.03 <sup>c</sup>	6.58±0.03 <sup>c</sup>	5.88±0.02 <sup>d</sup>	5.17±0.01 <sup>d</sup>
MW-30b	4.66±0.02 <sup>a</sup>	5.37±0.05 <sup>e</sup>	6.62±0.03 <sup>c</sup>	6.76±0.03 <sup>d</sup>	6.46±0.03 <sup>d</sup>	5.79±0.01 <sup>c</sup>	5.11±0.01 <sup>f</sup>

Steamed bread samples	Postprandial blood glucose levels (mmol/L)						
	0 min	15 min	30 min	45 min	60 min	90 min	120 min
MW-40b	4.66±0.03 <sup>a</sup>	5.25±0.03 <sup>f</sup>	6.47±0.03 <sup>f</sup>	6.67±0.05 <sup>e</sup>	6.37±0.01 <sup>ef</sup>	5.61±0.01 <sup>g</sup>	5.06±0.01 <sup>h</sup>
MW-50b	4.67±0.02 <sup>a</sup>	5.18±0.06 <sup>g</sup>	6.36±0.04 <sup>g</sup>	6.55±0.03 <sup>f</sup>	6.36±0.02 <sup>f</sup>	5.54±0.04 <sup>h</sup>	4.99±0.02 <sup>i</sup>

**Notes:** all values are the mean of at least triplicate determinations  $\pm$  SD. The means within the same column with different letters are significantly different (P value<0.05).

Based on the results of color analysis, specific volume analysis, texture analysis and sensory evaluation of steamed bread, it was concluded that the optimal substitution amount of wheat flour with HMTS or MWS was 30%. Therefore, the optimal recipe of steamed bread made with incorporation of HMTS or MWS was as follow: 70 g wheat flour (the protein content was 10.0%  $\pm$  1.0%, 12% moisture content), 30 g HMTS or MWS (12% moisture content), 55 g water, 1.0 g yeast and 1.0 g salt.

## Conclusions to section 5

1. The substitution of low protein flour with HMTS or MWS in quantity above 5% made cookies brighter, yellower, and less reddish. The differences in color between the control and experimental cookies were detectable by the human eye when the substitution amount of low protein flour with HMTS or MWS reached 5%. The hardness (included average hardness, surface hardness and max hardness) of cookies with HMTS or MWS was significantly lower than of control ( $P_{\text{value}} < 0.05$ ), but higher crispy value, indicating less work to be consumed when chewing. The addition of appropriate amount of HMTS or MWS to cookies could improve the appearance. Cookies with 15% HMTS or 15% MWS had the highest acceptability score. Good quality cookies can thus be prepared from low protein flour with substitution of HMTS and MWS. The present research might help to enlarge the application of modified potato starch in bakeries.

2. Substitution of with heat-moisture treatment modified potato starch (HMTS) or with microwave treatment modified potato starch (MWS) altered the texture and tensile properties of dough. Through correlation analysis, it has been concluded that the dough tensile properties of resistance to extension and extensibility were extremely significant positive correlated with the cooked fresh noodles tensile properties of tensile strength and elasticity. Incorporation of HMTS and MWS significantly decreased the optimal cooking time of fresh noodles ( $P_{\text{value}} < 0.05$ ), moreover, the optimal cooking time of noodles with HMTS was lower than that of noodles with MWS when the substitution amount was same. The dry matter water absorption rate and loss rate of dry matter significantly increased with the increase of substitution amount of HMTS and MWS. Moreover, both of the dry matter water absorption rate and loss rate of dry matter of noodles with HMTS were lower than that of noodles with MWS when the substitution amount was same, indicating noodles with HMTS had a better cooking quality than that of noodles with MWS in optimal cooking time condition. When the incorporation amount of HMTS was less than 30% and the incorporation amount

of MWS was less than 20%, the noodles could remain intact without breaking. The present research might help to enlarge the application of modified potato starch in cooking noodle-like food.

3. The specific volume of steamed bread decreased with more incorporation of HMTS or MWS. The specific volume of steamed bread buns made by incorporating MWS was lower than that of steamed bread made by incorporating HMTS, which indicated that MWS had greater impact on the specific volume of steamed bread than HMTS. The experimental steamed bread showed higher  $L^*$  and  $a^*$ , but lower  $b^*$  values than those of the control steamed bread, and the color changes was more obvious with the increase of substitution level of HMTS or MWS, indicating that steamed bread with more incorporation of HMTS or MWS displays lighter transparent color. Moreover, when the substitution level of HMTS or MWS was higher than 30%, 20%, respectively, the differences in color ( $\Delta E > 3$ ) between the control and experimental steamed bread can be detectable by the human eye. In terms of texture properties, the results of this study showed that hardness, gumminess and chewiness of all the experimental steamed bread were significantly higher than those of the control steamed bread, while springiness, cohesiveness and resilience of experimental steamed bread were lower than those of the control steamed bread. These results indicated that the incorporation of HMTS or MWS led to firmer and denser structure of steamed bread. The total sensory score of was higher than 80 when the substitution level of wheat flour with HMTS or MWS was less than 30%, indicating the produced steamed bread can be accepted by consumers. The appropriated amount of substitution of wheat flour with HMTS or MWS can not only maintain the quality of steamed bread, but also increase the nutrition of steamed bread. Based on the above research results of steamed bread quality, the optimal substitution of wheat flour with HMTS or MWS was 30%. This research can provide valuable guidance for further application of HMT and MW modified potato starch in wheat-based products, and it is also of great significance for promoting potato as staple food.

4. Postprandial blood glucose levels of participants at different times after

eating cookies, fresh noodles or steamed breads with incorporation of HMTS or MWS were lower than that of the control. All these results indicated that cookies, fresh noodles and steamed breads with incorporation of HMTS or MWS were more suitable for diabetics or the elderly.



## GENERAL CONCLUSIONS

In this research, we not only investigated the effects of heat-moisture treatment (HMT) conditions the morphological, physicochemical and *in vitro* digestion properties of potato starch and its process optimization, but also evaluated the effects of heat-moisture treatment assisted by microwave (MW) pre- and post-treatment on the morphological, physicochemical and *in vitro* digestion properties of potato starch. This research presented a comprehensive understanding of the effects of HMT conditions and HMT and MW bi-directional modifications on functional and digestibility properties of starch, as well as the related mechanism, which would provide a useful theoretical basis for further studies on improving the application of hydrothermal treatment technology and microwave treatment technology in starch modification. Besides, we also investigated the effects of substitution of wheat flour with potato starch modified by heat-moisture treatment and microwave treatment on the quality characteristics of three typical food products including cookies, fresh noodles and steamed bread, which would provide a beneficial theoretical basis for further research on the application of HMT and MW modified starch in food. Moreover, we also analyzed the socio-economic efficiency of cookies, fresh noodles and steamed bread with the studied raw materials. The main work and general conclusions of this research can be summarized as follows:

1. The transparency and retrogradation stability of potato starch after HMT were reduced, solubility and swelling power varied with the gelatinization temperature. HMT can significantly affect the textural properties of potato starch and the hardness, gumminess, chewiness and resilience of HMT starch gels first increased significantly and then decreased with the extension of treatment time. Short heating time (<1.5 h), relatively low heating temperature (<100°C) and low moisture content (<25%) of HMT can significantly enhance the texture properties of HMT starch gels.

2. HMT led to the rupture, adhesion and partial gelatinization and agglomeration of the granules, which surface became rougher, thereby increasing the particle size and result in the hollow structure located at the hilum of potato starch granules. XRD results showed an increased relative crystallinity and transformed crystalline structure from B-type to C-type with the extension heat moisture treatment. FTIR spectroscopy results indicated, that the HMT may lead to the increase of the number of carbonyl group and hydroxyl group, and it is possible to increase the new C-H bonds. HMT significantly decreased the peak viscosity, hold viscosity and breakdown viscosity of HMT starch, while the gelatinization temperature increased. The total content of SDS and RS, and tHMT1, THMT100 and CHMT15 had the highest content of SDS and RS (more than 71.6%), and there was no significant difference between the three samples.

3. The results of response surface methodology showed the optimal parameters of HMT were that moisture content of potato starch was 23.56%, heat-moisture treatment temperature was 90°C, and heat-moisture treatment time was 1.5 h. Under such conditions, setback viscosity of heat-moisture treatment modified potato starch (HMTS) paste was 3677 cP, which was higher than native starch (496 cP) obviously. The results of retrogradation was consistent with the viscosity properties, all of which indicating that potato starch modified by heat-moisture treatment was more prone to retrogradation. TPA tests demonstrated that HMT can enhance the textural properties of starch gel. Compared with native starch gel, HMTS starch gel had better functional properties. The optimized HMT potato starch (HMTS) had higher SDS content and RS content than that of native potato starch (NS), but lower RDS content.

4. Heat-moisture treatment combined with microwave pre- and post-treatment decreased the swelling power of potato starch when the test temperature was 65–85°C, but increased the solubility when test temperature was 75–95°C. Single MW, short-time single HMT and short-time HMT combined with MW pretreatment can enhance the repeated freeze-thaw stability of potato starch pastes, while long- time HMT (>4h) could weaken the freeze-thaw stability of potato

starch. Dual modification of HMT and MW had greater effects on starch retrogradation and transparency than that of single HMT or single MW, and the subsequence of dual modification of HMT and MW would also affect the starch retrogradation and transparency. The hardness, cohesiveness, gumminess and chewiness of all the HMT modified potato starch gel (including single HMT, HMT combined with MW) decreased with the extension of heating time. The HMT potato starch pretreated by MW had higher hardness value than that of HMT potato starch post-treated by MW.

5. In general, HMT caused a slight increase of lightness ( $L^*$  values), while single MW caused a slight decrease of lightness, indicating that the color of all the HMT treated samples became brighter and the color of the single MW treated sample (MWS) became darker. The color of MW- HMT samples became more reddish, while MWS, HMT and HMT-MW samples became more greenish. The results of particle size distribution showed that  $D_{50}$ ,  $D(4,3)$  and  $D(3,2)$  of all treated starch were higher than NS, while the value of S.S.A. was significantly decreased by MW and HMT, indicating that MW and HMT treatments can caused expansion, partial gelatinization and agglomeration of starch granules, resulting in large particle size of starch granules. MW and HMT treatments of potato starch caused the  $T_{21}$  shifted toward faster relaxation times compared with native starch (NS). The results of water distribution indicated the MW treated starch had three different state water, while NS and single HMT treated starch only had two different state water. Furthermore, MW and HMT treatments could change the water distribution and improve the interaction between starch and water.

6. Dual starch modification via MW and HMT made the surface of its granules rougher and caused more serious depressions or scallops than single modification with MW or HMT, especially in the case of HMT combined with post-MW. FT-IR and XRD spectra implied that MW and HMT destroyed the double helices and crystalline structure of potato starch. The relative crystallinity of modified starch granules (15.17–18.17%) was lower than that of native starch (19.39%). In the case of physicochemical properties, the modified starches had

higher pasting temperature (68.8–93.0°C) and setback viscosity (807–3168 cP), but lower peak viscosity (1315–3662 cP) and breakdown viscosity (17.3–78.3 cP) than that of native potato starch, which were 68.5°C, 496 cP, 6598 cP and 2526 cP, respectively. The HMT and MW modifications significantly increased the content of slowly digestible starch and resistant starch. The resistant starch content of starch obtained by HMT combined with MW post-treatment was significantly higher than that of starch obtained by HMT combined with MW pre-treatment and single HMT. These results may promote good understanding of the effects of HMT combined with MW pre- and post-treatment on physicochemical properties and digestibility of potato starch, and wide utilization of microwave and heat-moisture techniques in starch modification.

7. The incorporation of HMTS or MWS powder had significant effects on the color, texture properties of cookies compared with the control. Although there were no significant effects on the sensorial properties of cookies, cookies with addition of HMTS or MWS powder in the amount of 15% not only had crispy taste, but also had the highest acceptability score and yellowest color. Good quality cookies can thus be prepared from low protein flour with substitution of HMTS and MWS. The results of postprandial blood glucose levels of participants at different times after eating cookies indicated that cookies with incorporation of HMTS or MWS were more suitable for diabetics or the elderly. The present results might help to enlarge the application of modified potato starch in bakeries.

8. Substitution wheat flour with HMT and MW modified potato starch (HMTS and MWS) significantly decreased the optimal cooking time of fresh noodles ( $P_{\text{value}} < 0.05$ ). The dry matter water absorption rate and loss rate of dry matter significantly increased with the increase of substitution amount of HMTS and MWS. Both of the dry matter water absorption rate and loss rate of dry matter of noodles with HMTS were lower than that of noodles with MWS when the substitution amount was same, indicating noodles with HMTS had a better cooking quality than that of noodles with MWS in optimal cooking time condition. When the incorporation amount of HMTS was less than 30% and the

incorporation amount of MWS was less than 20%, the noodles could remain intact without breaking. The results of postprandial blood glucose levels of participants at different times after eating fresh noodles indicated that fresh noodles with incorporation of HMTS or MWS were more suitable for diabetics or the elderly. The present results might help to enlarge the application of modified potato starch in cooking noodle-like food.

9. Substitution of modified potato starch (HMTS and MWS) decreased the specific volume of steamed bread. The differences in color ( $\Delta E > 3$ ) between the control and experimental steamed bread were detectable by the human eye when the substitution level of HMTS was higher than 30% or the substitution level of MWS was higher than 20%. Texture properties, including hardness, gumminess, chewiness, springiness, cohesiveness and resilience of steamed bread were affected with substitution due to the disruption of dough structure, and the incorporation of HMTS or MWS led to firmer and denser structure of steamed bread. The total sensory evaluation scores of steamed breads decreased with more incorporation of HMTS or MWS. Based on the above research results of steamed bread quality, the optimal substitution of wheat flour with HMTS or MWS was 30 %. The results of postprandial blood glucose levels of participants at different times after eating steamed breads indicated that steamed breads with incorporation of HMTS or MWS were more suitable for diabetics or the elderly. This research can provide valuable guidance for further application of HMT and MW modified potato starch in wheat-based products, and it is also of great significance for promoting potato as staple food.

10. According to the optimal recipe of cookies made with incorporation of 15% HMTS or 15% MWS, the profit of production of cookies in the pastry shop with a capacity of 60 kg per shift will be UAH 1351.14 and the profitability of production will be 11.26%. The profit of production of fresh noodles made with the optimal recipe of fresh noodles made with incorporation of 30 % HMTS or 20 % MWS in the pastry shop with a capacity of 100 kg per shift will be UAH 495.76 and the profitability of production will be 12.39%. The profit of production

of steamed bread made with incorporation of 30% HMTS or 30% MWS in the pastry shop with a capacity of 100 kg per shift will be UAH 285.66 and the profitability of production will be 7.14%. It is expedient and profitable of introducing the production of cookies, fresh noodles and steamed bread made with incorporation of HMTS or MWS in the pastry shop of the supermarket.

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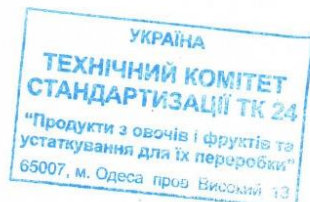
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## APPENDICES

## APPENDIX A



ЗАТВЕРДЖУЮ

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10.05.2023 року

**МОДИФІКОВАНИЙ КАРТОПЛЯНИЙ КРОХМАЛЬ**  
(фізична модифікація)**ТЕХНІЧНІ УМОВИ**

ТУ У 00383403.001:2023

Уведено вперше  
дата надання чинності 10.05.2023**РОЗРОБЛЕНО**

Сумський національний аграрний університет

Кафедра технології харчування

Аспірант кафедри технології харчування

Deng Chunli Денг Чунлі10 травня 2023 року

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Мельник О.Ю. Мельник10 травня 2023 року

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## 1. СФЕРА ЗАСТОСУВАННЯ

1.1. Цей стандарт поширюється на модифікований крохмаль — крохмаль, одержаний внаслідок фізичного (волого-термічне, мікрохвильове оброблення, комбіноване) оброблення нативного крохмалю для зміни його властивостей.

Модифікований крохмаль застосовують в різних галузях харчової промисловості: кондитерській, хлібопекарській, пивоварній, м'ясо-молочній, харчоконцентратній, для реалізації через торговельну мережу і використання в системі громадського харчування та для технічних цілей (в текстильній, паперовій, для буріння свердловин).

Вимоги щодо безпечності модифікованого крохмалю викладено в 3.3.4 та у розділах 4 і 5.

## 2. НОРМАТИВНІ ПОСИЛАННЯ

2.1. Цей стандарт містить посилання на подані нижче нормативні документи:

ДСТУ 2211-93 Крохмаль та крохмалепродукти. Терміни та визначення

ДСТУ 3976-2000 Крохмаль кукурудзяний сухий. Технічні умови

ДСТУ 4286:2004 Крохмаль картопляний. Технічні умови

ГОСТ 12.1.004-91 ССБП. Пожежна безпека. Загальні вимоги

ГОСТ 12.1.005-88 ССБП. Загальні санітарно-гігієнічні вимоги до повітря робочої зони

ГОСТ 12.2.124-90 ССБП. Устаткування продовольче. Загальні вимоги щодо безпеки

ГОСТ 12.3.002-75 ССБП. Процеси виробничі. Загальні вимоги щодо безпеки

ГОСТ 17.2.3.02-78 Охорона природи. Атмосфера. Правила встановлювання допустимих викидів шкідливих речовин промисловими підприємствами

ГОСТ 1770-74 Посуд мірний лабораторний скляний. Циліндри, мензурки, колби, пробірки. Технічні умови

Видання офіційне

ГОСТ 4328-77 Натрію гідроксид. Технічні умови

ГОСТ 6434-74 Декстрини. Технічні умови

ГОСТ 6709-72 Вода дистильована. Технічні умови

- ГОСТ 7698-93 Крохмаль. Правила приймання і методи аналізування
- ГОСТ 9070-75 Віскозиметри для визначання умовної в'язкості лакофарбових матеріалів
- ГОСТ 10444.12-88 Продукти харчові. Метод визначання дріжджів і плісневих грибів
- ГОСТ 10444.15-94 Продукти харчові. Методи визначання кількості мезофільних аеробних і факультативно-анаеробних мікроорганізмів
- ГОСТ 12026-76 Папір фільтрувальний лабораторний. Технічні умови
- ГОСТ 14919-83 Електроплити, електроплитки та жарильні електрошафи. Загальні технічні умови
- ГОСТ 24104-2001 Ваги лабораторні. Загальні технічні вимоги
- ГОСТ 24297-87 Вхідний контроль продукції. Основні положення
- ГОСТ 25336-82 Посуд та устаткування лабораторні скляні. Типи, основні параметри та розміри
- ГОСТ 26668-85 Продукти харчові та смакові. Методи відбирання проб для мікробіологічних аналізів
- ГОСТ 26669-85 Продукти харчові та смакові. Підготовка проб для мікробіологічних аналізів
- ГОСТ 26927-86 Сировина та продукти харчові. Методи визначення ртуті
- ГОСТ 26929-94 Сировина та продукти харчові. Готування проб. Мінералізація для визначання вмісту токсичних елементів
- ГОСТ 26930-86 Сировина та продукти харчові. Метод визначання миш'яку
- ГОСТ 26931-86 Сировина та продукти харчові. Методи визначання міді
- ГОСТ 26932-86 Сировина та продукти харчові. Методи визначання свинцю
- ГОСТ 26933-86 Сировина та продукти харчові. Методи визначання кадмію
- ГОСТ 26934-86 Сировина та продукти харчові. Метод визначання цинку
- ГОСТ 28498-90 Термометри рідинні скляні. Загальні технічні вимоги. Методи випробовування
- ГОСТ 29228-91 (ИСО 835-2-81) Посуд лабораторний скляний. Піпетки розградувані. Частина 2. Піпетки розградувані без встановленого часу

очікування

ГОСТ 30178-96 Сировина та продукти харчові. Атомно-абсорбційний метод визначення токсичних елементів

### 3. ЗАГАЛЬНІ ТЕХНІЧНІ ВИМОГИ

3.1 Модифікований крохмаль повинен відповідати вимогам цього стандарту, чинним нормативним документам на даний вид модифікованого крохмалю і його треба виробляти згідно з технологічною інструкцією по виробництву даного виду модифікованого крохмалю, затвердженою у встановленому порядку, з дотриманням санітарних норм і правил, затверджених центральним органом виконавчої влади у сфері охорони здоров'я.

3.2 Для виготовлення модифікованого крохмалю використовують нативний картопляний крохмаль – згідно ДСТУ 4286:2004 Крохмаль картопляний. Технічні умови.

Для виготовлення модифікованого крохмалю можуть використовуватись інші види вітчизняної та імпортої сировини - згідно з чинним нормативним документом або імпортої виробництва за наявності висновку санітарно-епідеміологічної експертизи центрального органу виконавчої влади, що забезпечує формування політики у сфері охорони здоров'я.

#### 3.3 Характеристики

3.3.1 За органолептичними показниками модифікований крохмаль повинен відповідати вимогам, зазначеним у таблиці 1.

*Таблиця 1 – Органолептичні показники*

Найменування показника	Характеристика
Зовнішній вигляд	Однорідний порошок
Колір	Білий або із злегка жовтим або сіруватим відтінком
Запах	Властивий крохмалю, без стороннього запаху

3.3.2 За фізико-хімічними показниками модифікований крохмаль повинен відповідати вимогам, зазначеним в таблиці 2.

*Таблиця 2 – Фізико-хімічні показники*

Назва показника	Норма для групи модифікованого крохмалю
Масова частка вологи, % для картопляного	≤18
Масова частка золи, %, для картопляного	≤0,5
Кислотність — витрати розчину гідроксиду натрію молярної концентрації NaOH = = 0,1 моль/дм <sup>3</sup> на нейтралізування 100 г сухої речовини, см <sup>3</sup> , не більше ніж	3
Умовна в'язкість водного крохмального клейстеру з масовою часткою крохмалю, с: 8 % для картопляного	≥900
Здатність набухати, см <sup>3</sup> /г	≥2,3
Ступінь помелу — проходження через сито з вічками 1,0 мм × 1,0 мм, %, не менше ніж	99,0
Кількість краплень на 1 дм <sup>2</sup> видимих неозброєним оком, шт., не більше ніж	500
Масова частка сірчистого ангідриду (SO <sub>2</sub> ), %, не більше ніж	0,005
Примітка 1. Норми фізико-хімічних показників уточнюють в нормативних документах на конкретний вид модифікованого крохмалю залежно від сировини і способу виготовлення	

3.3.3 За мікробіологічними показниками модифіковані крохмалі повинні відповідати вимогам, наведених в таблиці 3.

Таблиця 3 – Мікробіологічні показники

Назва показника	Норма
Загальна кількість мезофільних аеробних та факультативно-анаеробних мікроорганізмів, КУО в 1 г, не більше ніж	1,0×10 <sup>4</sup>
Плісєневі гриби, КУО в 1 г, не більше ніж	5,0×10
Дріжджі, КУО в 1 г, не більше ніж	1,0×10
Бактерії групи кишкових паличок (коліформи), в 1 г	Не допустимі
Патогенні мікроорганізми, зокрема бактерії роду Salmonella, в 25 г	Не допустимі

3.3.4 Вміст токсичних елементів у модифікованому крохмалі, який використовують для харчових цілей, не повинен перевищувати рівнів, встановлених МБТ № 5061 і вказаних в таблиці 4.

Таблиця 4 – Допустимі рівні вмісту токсичних елементів

Назва показника	Допустимий рівень вмісту, мг/кг, не більше ніж
Ртуть	0,02
Миш'як	0,1
Мідь	10,0
Свинець	0,5
Кадмій	0,1
Цинк	30,0

3.3.5 Вміст радіонуклідів у модифікованому крохмалі згідно з ДР-97 (Допустимі рівні вмісту радіонуклідів  $^{137}\text{Cs}$  і  $^{90}\text{Sr}$  в продуктах харчування та питній воді, затверджені МОЗ України від 25.06.97 р.):  $^{90}\text{Sr}$  — не більше ніж 200 Бк/кг,  $^{137}\text{Cs}$  — не більше ніж 600 Бк/кг.

#### 4. ВИМОГИ ЩОДО БЕЗПЕКИ

4.1 Процес виробництва модифікованого крохмалю здійснюють відповідно до загальних вимог щодо безпеки згідно з ГОСТ 12.3.002, вимог до виробничого устаткування, згідно з ГОСТ 12.2.124, та повинен відповідати вимогам, що викладені у ДНАОП 18.10-1.27 Правила безпеки при виробництві крохмале-патокової продукції, затверджені Держнаглядохоронпраці, № 306 від 15.12.97 р.

4.2 Вимоги до природного та штучного освітлювання під час виробництва модифікованого крохмалю повинні відповідати СНиП 11-4 (Природне та штучне освітлення).

4.3 Вимоги до опалення, вентиляції і кондиціювання під час виробництва модифікованого крохмалю повинні відповідати СНиП 2.04.05 (Опалення, вентиляція та кондиціювання).

4.4 Вимоги до повітря робочої зони під час виробництва модифікованого крохмалю повинні відповідати ГОСТ 12.1.005.

4.5 Пожежну безпеку здійснюють згідно з ГОСТ 12.1.004.

#### 5. ВИМОГИ ЩОДО ОХОРОНИ ДОВКІЛЛЯ

5.1 Стічні води під час виробництва модифікованого крохмалю піддають очищенню згідно з вимогами СанПіН 4630 Санітарні правила та норми щодо охорони поверхневих вод від забруднювання.



5.2 Контролюють викиди шкідливих речовин в атмосферу згідно з ГОСТ 17.2.3.02 та ДСП 201 [7].

## **6. ПАКУВАННЯ**

6.1 Пакують крохмаль модифікований згідно з ДСТУ 3976 та ДСТУ 4286.

## **7. МАРКУВАННЯ**

7.1 Маркують крохмаль модифікований згідно з ДСТУ 3976 та ДСТУ 4286.

## **8. ПРАВИЛА ТРАНСПОРТУВАННЯ ТА ЗБЕРІГАННЯ**

8.1 Правила транспортування та зберігання здійснюють згідно з ДСТУ 3976 та ДСТУ 4286.

## **9. МЕТОДИ КОНТРОЛЮВАННЯ**

### **9.1 Відбирання проб**

9.1.1 Відбирають проби для визначання органолептичних, фізико-хімічних показників та показників токсичних елементів згідно з ГОСТ 7698, для мікробіологічних показників — згідно з ГОСТ 26668 та ГОСТ 26669, готують пробу для визначання токсичних елементів — згідно з ГОСТ 26929.

9.2 Визначають органолептичні та фізико-хімічні показники: масову частку вологи, масову частку золи, кислотність, масову частку загального фосфору, кількість краплень, масову частку сірчистого ангідриду згідно з ГОСТ 7698.

### **9.3 Визначання умовної в'язкості водного крохмального клейстеру**

9.3.1 Визначання умовної в'язкості водного крохмального клейстеру на віскозиметрі Брукфільда RVDV-E, або аналогічному

Метод полягає у вимірюванні в'язкості водного клейстеру крохмалю на ротаційному віскозиметрі за температури 50 °C.

#### **9.3.1.1 Засоби контролювання та допоміжні пристрої**

Ваги лабораторні загального призначення 4-го класу точності з найбільшою границею зважування 500 г — згідно з ГОСТ 24104.

Ротаційний віскозиметр для вимірювання динамічної в'язкості — згідно з чинною нормативною документацією.

Термометр рідинний скляний з ціною поділки шкали 1 °C і діапазоном вимірювання від 0 °C до 100 °C — згідно з ГОСТ 28498.

Секундомір чи годинник наручний чи кишеньковий механічний — згідно з ГОСТ 10733 чи електронний — згідно з ГОСТ 23350.

Стакан В(Н)-1(2)-1000 ТС — згідно з ГОСТ 25336

Циліндр 1(3)-250 — згідно з ГОСТ 1770

Паличка скляна або лінійка пластмасова

Мішалка лопатева (з валом, здатним від'єднуватись від приводу) — згідно з чинною нормативною документацією

Баня водяна

Вода дистильована — згідно з ГОСТ 6709

Допустиме застосування інших засобів контролювання та допоміжних пристроїв з технічними і метрологічними характеристиками не нижче зазначених.

#### 9.3.1.2 Готування крохмальної суспензії масовою часткою 10 %

В стакан зважують 50 г крохмалю і циліндром додають таку кількість дистильованої води, щоб загальна маса крохмалю і дистильованої води становила 500 г. Вміст стакана ретельно перемішують. Кладуть вал мішалки в стакан і зважують загальну їх масу.

#### 9.3.1.3 Проведення випробовування

Стакан ставлять в киплячу водяну баню, яку щільно закривають накривкою з прорізом для мішалки. Вал з'єднують з мішалкою і суспензію перемішують з постійною швидкістю 250 об/хв (швидкість обертання повинна бути постійна). Через 15 хв мішалку зупиняють і продовжують нагрівання ще 15 хв.

Потім від'єднують вал від мішалки, але залишають його в стакані. Стакан виймають з водяної бані, зважують і доливають стільки води, на скільки зменшилась загальна маса суспензії, компенсуючи кількість води, яка випарувалась.

Перемішуючи (швидкість обертання 100 об/хв), клейстер охолоджують на водяній бані до 50 °С і відразу вимірюють в'язкість ротаційним віскозиметром.

За кінцевий результат випробовування беруть середнє арифметичне двох паралельних визначень.

Результати округлюють до першого десяткового знаку з подальшим

заокругленням до цілого числа.

#### 9.4 Визначання здатності набухати

Метод полягає у визначанні об'єму крохмалю, набухлого у воді.

##### 9.4.1 Засоби контролювання та допоміжні пристрої

Ваги лабораторні загального призначення 4-го класу точності з найбільшою границею зважування 500 г — згідно з ГОСТ 24104

Циліндр 1(3)-100-2 — згідно з ГОСТ 1770

Вода дистильована — згідно з ГОСТ 6709

Допустиме застосування інших засобів контролювання та допоміжних пристроїв з технічними і метрологічними характеристиками не нижче зазначених.

##### 9.4.2 Проведення випробовування

На аркуші паперу зважують 5 г крохмалю, переносять у циліндр, доливають дистильовану воду кімнатної температури, ретельно перемішують, а потім вміст циліндра доводять дистильованою водою до 100 см<sup>3</sup>. Суспензію ще раз ретельно перемішують і залишають у стані спокою за кімнатної температури на одну годину, після чого відмічають межу розділу двох фаз. У разі повного набухання спостерігається рівномірний розподіл крохмалю за об'ємом.

У разі неповного набухання крохмалю в циліндрі утворюються дві фази:

- верхня — рідка, прозора, яка майже не містить крохмалю;
- нижня — непрозора, з набухлим крохмалем.

##### 9.4.3 Опрацювання результатів

Здатність набухати крохмалю  $H$  в сантиметрах кубічних на грам розраховують за формулою:

$$H = V/m,$$

де  $V$  — об'єм крохмалю в циліндрі після відстоювання, см<sup>3</sup>;  $m$  — маса наважки крохмалю, г.

За кінцевий результат беруть середнє арифметичне двох паралельних визначень, за умови, коли:

- результати визначень, виконаних в одній лабораторії, не повинні відрізнятись більш ніж на 0,01 см<sup>3</sup>/г від їх середнього арифметичного;

— результати визначень, виконаних двома різними лабораторіями, не повинні відрізнятись більш ніж на  $0,02 \text{ см}^3/\text{г}$  від їх середнього арифметичного.

Результати округлюють до першого десяткового знаку.

#### 9.5 Визначання ступеня помелу

Метод полягає у визначанні маси проходження крохмалю через сито з проволочною сіткою з розміром вічок  $0,1 \text{ мм}$ .

##### 9.5.1 Засоби контролювання та допоміжні пристрої

Ваги лабораторні загального призначення 4-го класу точності з найбільшою границею зважування  $500 \text{ г}$  — згідно з ГОСТ 24104

Металеve сито з вічками розміром  $0,1 \text{ мм}$

Розсівник лабораторний (з кількістю обертів від  $180 \text{ об/хв}$  до  $200 \text{ об/хв}$ )

Годинник наручний чи кишеньковий механічний — згідно з ГОСТ 10733, чи електронний — згідно з ГОСТ 23350

Стакан В(Н)-2-150 — згідно з ГОСТ 25336

Допустиме застосування інших засобів контролювання та допоміжних пристроїв з технічними і метрологічними характеристиками не нижче зазначених.

##### 9.5.2 Проведення випробовування

Визначання ступеня помелу проводять на лабораторному розсівнику. Сито встановлюють на піддон. В стакані зважують  $100 \text{ г}$  крохмалю, висипають на сито, закривають сито накривкою і закріплюють на платформі розсівник, після цього вмикають пристрій. Через  $8 \text{ хв}$  просіювання припиняють, злегка постукують по сити і знову продовжують просіювання протягом  $2 \text{ хв}$ . По закінченні просіювання крохмаль на піддоні зважують.

Під час ручного просіювання час струшування сита з крохмалем повинен становити  $15 \text{ хв}$ .

##### 9.5.3 Опрацювання результатів

Ступінь помелу у відсотках чисельно дорівнює масі крохмалю, який пройшов через сито.

9.6 Вміст токсичних елементів в модифікованому крохмалі визначають: ртуть — згідно з ГОСТ 26927, миш'як — згідно з ГОСТ 26930, мідь — згідно з ГОСТ

26931 чи ГОСТ 30178, свинець — згідно з ГОСТ 26932 чи ГОСТ 30178, кадмій — згідно з ГОСТ 26933 чи ГОСТ 30178, цинк — згідно з ГОСТ 26934 чи ГОСТ 30178.

Допустиме застосування інших методів визначання токсичних елементів, які мають свідоцтво про метрологічну атестацію і узгоджені з центральним органом виконавчої влади у сфері охорони здоров'я.

9.7 Мікробіологічні показники модифікованого крохмалю визначають згідно з ГОСТ 10444.12, ГОСТ 10444.15 та СанПиН 42-123-4940 Мікробіологічні нормативи й методи аналізування продуктів дитячого, лікарського й дієтичного харчування та їх компонентів.

Аналіз на патогенні мікроорганізми проводять відомчі чи інші лабораторії, що мають відповідний дозвіл органів Держсаннагляду, у порядку Державного санітарного нагляду санітарно-епідеміологічними станціями за затвердженими методами.

9.8 Визначання вмісту радіонуклідів проводять згідно з Методичними вказівками, затвердженими Міністерством охорони здоров'я України: 90Sr — № 5778 Методичні вказівки. «Стронцій-90. Визначання в харчових продуктах», № 5778-91, М., —1991.Свідоцтво МА МВМ ИБФ № 14/1-89, 137Cs — № 5779 Методичні вказівки «Цезій-137. Визначення в харчових продуктах», № 5779-91, М., —1991.Свідоцтво МА МВМ ИБФ № 15/1-89.

## **10 ПРАВИЛА ПРИЙМАННЯ**

10.1 Правила приймання — згідно з ГОСТ 7698.

10.2 Періодичність визначання токсичних елементів, мікробіологічних показників і радіонуклідів у модифікованому крохмалі встановлюють відповідно до Методичних рекомендацій. Визначення токсичних елементів і мікробіологічних показників у модифікованому крохмалі проводять один раз у квартал. Показник золи контролюють періодично, але не рідше одного разу в 10 днів.

10.3 Контролюють якість сировини та матеріалів, що поступають, згідно з ГОСТ 24297.

## **11 ГАРАНТІЇ ВИРОБНИКА**

11.1 Виробник повинен гарантувати відповідність якості модифікованого

крохмалю вимогам цього стандарту за умови дотримування споживачем умов транспортування і зберігання.

11.2 Термін придатності до споживання модифікованого крохмалю — 2 роки від дати виготовлення.

## РЕЦЕПТУРА І ТЕХНОЛОГІЧНА ІНСТРУКЦІЯ на печиво «МоКа»

Підстава: ДСТУ 3781:2014 «Печиво. Загальні технічні умови».

### 1. ТЕХНІЧНІ ВИМОГИ.

Печиво «МоКа» виготовляють відповідно до вимог цих технічних інструкцій та вимог чинного законодавства України щодо безпечності та окремих показників якості харчових продуктів.

Печиво «МоКа» – здобне печиво з борошна вищого сорту з додаванням картопляного крохмалю фізичної модифікації. Має квадратну форму.

Для виготовлення печива «МоКа» використовують такі основні види сировини:

борошно пшеничне вищого сорту з низьким вмістом клейковини (7,0 %) – ДСТУ 46.004-99

модифікований крохмаль – ТУ У 00383403.001:2023

цукрова пудра – ДСТУ 4623:2006

молоко питне – ДСТУ 2661:2010

сіль - ДСТУ 3583:2015

В 1 кг міститься не менше 80 штук.

Вологість  $10,0 \pm 1,5\%$ .

Для виготовлення печива «МоКа» можуть використовуватись інші види вітчизняної та імпоротної сировини - згідно з чинним нормативним документом або імпортного виробництва за наявності висновку санітарно-епідеміологічної експертизи центрального органу виконавчої влади, що забезпечує формування політики у сфері охорони здоров'я.

За органолептичними показниками печиво «МоКа» має відповідати вимогам, зазначеним у таблиці 1.

*Таблиця 1*

Найменування показника	Характеристика
Форма	Квадратна, без пошкоджень, краї рівні без вм'ятин. Допускається – не більше 5,0%

	виробів надломлених від маси нетто паковальної одиниці
Поверхня	Непідгоріла, без вкраплень крихт, без здутих, пухирців, що лопнули
Колір	Рівномірний, від світло-кремового до жовтого
Смак і запах	Властиві печиву, без стороннього присмаку і запаху
Вид в розломі	Пропечене печиво з рівномірною пористістю без пустот і слідів непромісу

**За фізико-хімічними показниками печиво «МоКа» повинно відповідати вимогам, зазначеним в таблиці 2.**

Таблиця 2

Найменування показників	Норма	Метод аналізу
Вологість, %	$10,0 \pm 0,5$	ДСТУ 4910
Масова частка загального цукру (за сахарозою) в перерахунку на суху речовину, %	$16,0 \pm 2,5$	ДСТУ 5059
Масова частка жиру в перерахунку на суху речовину, %	$30,0 \pm 1,0$	ДСТУ 5060
Лужність, град не більше	2,0	ДСТУ 4672
Масова частка золи, нерозчинної в розчині соляної кислоти з м.ч. 10%, %, не більше	0,1	ДСТУ 4672
Намочуваність, %, не менше	110	ДСТУ 5023

**Вміст токсичних елементів в печиві** не повинен перевищувати гранично допустимих концентрацій, передбачених Сан ПіН 42-123-4089 та приведених в таблиці 3.

Таблиця 3

Найменування токсичного елементу	Гранично допустима концентрація, мг/кг, не більше	Метод аналізу
Ртуть	0,02	ГОСТ 26927
Миш'як	0,3	ГОСТ 26930
Свинець	0,5	ГОСТ 26932
Кадмій	0,1	ГОСТ 26933
Мідь	10,0	ГОСТ 26931
Цинк	30,0	ГОСТ 26934

**Вміст мікотоксинів в печиві** не повинен перевищувати рівні, передбачені «Медико-біологічними вимогами та санітарними нормами



якості продовольчої сировини та харчових продуктів» №5061, а **вміст пестицидів** не повинен перевищуватит рівнів, зазначених у ДСанПіН 8.8.1.2.3.4.-000 і їх регламенту у сировині.

**За мікробіологічними показниками печиво** повинно відповідати вимогам, наведених в таблиці 4.

Таблиця 4

Найменування показників	Норма
Кількість мезофільних аеробних і факультативно-анаеробних мікроорганізмів, КУО в 1,0 г продукту, не більше	$1 \times 10^4$
Бактерії групи кишкових паличок (коліформи), в 0,1 г продукту	не допускаються
Патогенні мікроорганізми, в т.ч. бактерії роду Сальмонела, в 25 г	не допускаються
Плісеневі гриби, КУО в 1 г, не більше ніж	-

## 2. Вимоги до сировини

Сировина, що застосовується при виготовленні печива, повинна бути дозволена до застосування МОЗ України. Якість її повинна відповідати вимогам чинної нормативної документації; за вмістом токсичних елементів, мікотоксинів, пестицидів, нітратів має відповідати «Медико-біологічним вимогам і санітарним нормам якості продовольчої сировини і харчових продуктів № 5061».

**Рецептура на печиво «МоКа»** наведена в таблиці 5.

Таблиця 5

Сировина	Вміст сухих речовин, %	Витрата сировини на 100,0 кг печива,	
		в натурі	в сухих речовинах
Борошно пшеничне з низьким вмістом клейковини	88,0	49,36	43,44
Модифікований крохмаль	88,0	8,71	7,66
Масло вершкове	80,0	38,71	30,97
Цукрова пудра	99,85	16,13	16,11
Молоко	12,5	9,68	1,21
Сіль	96,5	0,32	0,31
Натрію бікарбонат	99,0	0,32	0,32
Вода	0,00	0	0
Разом		122,94	100,02
Вихід			

## **Технологічна інструкція з приготування печива «МоКа»**

Приготування печива складається з наступних стадій:

- підготовка сировини до виробництва;
- приготування тіста;
- формування тіста;
- випікання та охолодження;
- упаковка, маркування, транспортування і зберігання.

### **Підготовка сировини до виробництва**

Підготовка сировини до виробництва здійснюється відповідно до Технологічних інструкцій для виробництва борошняних кондитерських виробів. Допускається взаємозамінність сировини відповідно до Вказівок до рецептур на печиво.

### **Приготування тіста**

Розм'якшене масло збивають до кремоподібного та блілого кольору, додають цукрову пудру та рівномірно перемішують, додають молоко та сіль і перемішують електричним міксером протягом 3 хвилин на 1 швидкості до утворення однорідної маси, додають суміш пшеничного борошна з низьким вмістом глютену та модифікованого крохмалю та замішують м'яке тісто.

### **Формування тіста, випікання**

Перед формуванням тісто загортають в пекарський папір і за допомогою форми формують прямокутник шириною 6 см і висотою 4 см. Тістову заготовку залишають в холодильнику при температурі -18 °С протягом 1 години. Заморожене кубоподібне тісто нарізають ножом на тістові заготовки товщиною 0,7 см і викладають у підготовлену форму для випікання. Випікання тістових заготовок проводять при температурі 170 °С протягом 17 хвилин.

Випечене печиво надходить на стрічковий транспортер, де під обдувом холодного повітря охолоджується до температури 18-20 °С.

### **Упаковка, маркування, транспортування і зберігання**

Упаковка, маркування, транспортування і зберігання печива проводяться відповідно до вимог ДСТУ 3781:2014.

Допустимі відхилення маси нетто пакувальної одиниці печива складають, не більше:

мінус 10,0 % — до 50 г включно,

мінус 5,0 % - понад 50 до 400 г,

мінус 2,5 % - понад 400 до 500 г,

мінус 1,5 % - понад 500 до 1000 г,

мінус 1,0 % - понад 1000 г.

Відхилення маси нетто за верхньою межею не обмежується.

Печиво зберігають у сухих, чистих, добре вентильованих приміщеннях, що не мають стороннього запаху, не заражених шкідниками хлібних запасів, при температурі  $(18 \pm 5) ^\circ\text{C}$  і відносній вологості повітря не більше 75%.

**Строк придатності печива «МоКа»** вагового з подальшим обтягуванням короба полімерною плівкою — 4 міс.

**Харчова (поживна) та енергетична цінність (калорійність) 100 г печива «МоКа»**

Білки, г	Жири, г	Вуглеводи, г	Калорійність, ккал
<b>3,8</b>	<b>30,9</b>	<b>54,5</b>	<b>511,9</b>

**РОЗРОБЛЕНО:**

аспірантка кафедри технології харчування Сумського національного аграрного університету

Денг Чунлі

## РЕЦЕПТУРА І ТЕХНОЛОГІЧНА ІНСТРУКЦІЯ на хлібці «Парові»

Підстава: ДСТУ 4582:2006 «Хліб та хлібобулочні вироби».

### 1. Технічні вимоги.

Хлібці «Парові» виготовляють відповідно до вимог цих технічних інструкцій або нормативних документів виробника (ТУ, ТУУ, СОУ тощо, які не суперечать вимогам відповідного ДСТУ), затвердженим у встановленому законодавством України порядку.

Хлібці «Парові» – хлібці з борошна пшеничного з пониженим вмістом білку (10,0 %  $\pm$  1,0%) з додаванням картопляного крохмалю фізичної модифікації. Мають круглу форму.

Для виготовлення хлібців «Парових» використовують такі основні види сировини:

борошно пшеничне вищого сорту з низьким вмістом клейковини (10,0 %  $\pm$  1,0%) – ДСТУ 46.004-99

модифікований крохмаль – ДСТУ 4380:2005

дріжджі – ДСТУ 4812:2007

сіль – ДСТУ 4307:2004

вода - ДСТУ 7525:2014

В 1 кг міститься не менше 20 штук.

Вологість 38,0 $\pm$ 0,5%.

Для виготовлення хлібців «Парових» можуть використовуватись інші види вітчизняної та імпоротної сировини - згідно з чинним нормативним документом або імпортного виробництва за наявності висновку санітарно-епідеміологічної експертизи центрального органу виконавчої влади, що забезпечує формування політики у сфері охорони здоров'я.

За органолептичними показниками хлібці «Парові» мають відповідати вимогам, зазначеним у таблиці 1.

*Таблиця 1*

Найменування показника	Характеристика
Зовнішній вигляд: форма	Кругла, правильної форми, без бокових впливів
поверхня	Без краплень, без здутин, пухирців, гладка. Відшарування скоринки від м'якушки не дозволено.

колір	Рівномірний, світло-кремовий
Стан м'якушки	Пористість добре розвинена, рівномірна, середній розмір пор
Смак і запах	Запах та смак властивий пшеничному хлібу без сторонніх запахів та присмаків

**За фізико-хімічними показниками хлібці «Парові» повинні відповідати вимогам, зазначеним в таблиці 2.**

Таблиця 2

Найменування показників	Норма	Метод аналізу
Вологість, %, не більше	45,0	Згідно з ДСТУ 7045
Кислотність, град, не більше	3,5	Згідно з ДСТУ 7045
Пористість, %, не менше	65,0	Згідно з ДСТУ 7045

**Вміст токсичних елементів в хлібцях «Парових» не повинен перевищувати гранично допустимих концентрацій, передбачених СанПіН 42-123-4089 та приведених в таблиці 3.**

Таблиця 3

Найменування токсичного елементу	Гранично допустима концентрація, мг/кг, не більше	Метод аналізу
свинець	0,3	Згідно з ГОСТ 26932, ГОСТ 30178
кадмій	0,05	Згідно з ГОСТ 26933, ГОСТ 30178
миш'як	0,1	Згідно ГОСТ 26930
ртуть	0,01	Згідно ГОСТ 26927
мідь	5,0	Згідно з ГОСТ 26931, ГОСТ 30178
цинк	25,0	Згідно з ГОСТ 26934, ГОСТ 30178
Мікотоксини: афлатоксин В <sub>1</sub>	0,005	Згідно з МР № 2273 [1], МР № 4082 [2], ДСТУ EN 12955
дезоксиніваленол	0,5	Згідно з МР № 3940 [3], МУ № 5177 [4]
зеараленон	1,0	Згідно з МР № 2964 [5], МУ № 5177 [4]

**Вміст мікотоксинів в хлібцях** не повинен перевищувати рівні, передбачені «Медико-біологічними вимогами та санітарними нормами якості продовольчої сировини та харчових продуктів» №5061, а **вміст пестицидів** не повинен перевищуватит рівнів, зазначених у ДСанПіН 8.8.1.2.3.4.-000 і їх регламенту у сировині.

**За мікробіологічними показниками хлібці** мають відповідати вимогам, наведених в таблиці 4.

Таблиця 4

Найменування показників	Норма
Кількість мезофільних аеробних і факультативно-анаеробних мікроорганізмів, КУО в 1,0 г продукту, не більше	$1 \times 10^3$
Плісєневі гриби, КУО в 1 г, не більше ніж	Не допускається

## 2. Вимоги до сировини.

Сировина, що застосовується при виготовленні хлібців, повинна бути дозволена до застосування МОЗ України. Якість її повинна відповідати вимогам чинної нормативної документації; за вмістом токсичних елементів, мікотоксинів, пестицидів, нітратів має відповідати «Медико-біологічним вимогам і санітарним нормам якості продовольчої сировини і харчових продуктів № 5061».

**Рецептура на хлібці «Парові»** наведена в таблиці 5.

Таблиця 5

Сировина	Масова частка вологи, %	Витрати борошна, кг	
		1	100
Борошно пшеничне з низьким вмістом клейковини	12,0	0,70	70,0
Модифікований крохмаль	12,0	0,30	30,0
Дріжджі пресовані	15,0	0,01	1,0
Сіль	3,0	0,01	1,0
Вода	100,0	0,55	55,0
Всього тіста	-	1,57	157,0
Кількість тістових заготовок	-	31	3140

## Технологічна інструкція з приготування хліба «Паровий»

Приготування хліба складається з наступних стадій:

- підготовка сировини до виробництва;
- приготування тіста;
- бродіння тіста;

- формування тіста;
- випікання та охолодження;
- упаковка, маркування, транспортування і зберігання.

### **Підготовка сировини до виробництва**

Підготовка сировини до виробництва здійснюється відповідно до Технологічних інструкцій для виробництва хлібних виробів. Допускається взаємозамінність сировини відповідно до Вказівок до рецептур на хліб.

### **Приготування тіста**

Тісто формували шляхом змішування пшеничного борошна, модифікованого крохмалю (НМТS або МWS), солі, дріжджів та води і замішування тіста протягом 2 хв. Потім тісто залишали на бродіння в камері при 35°C і відносній вологості 65% протягом 60 хв. Після бродіння тісто обминали та подавали на формування.

### **Формування тіста, пропарювання**

Виброджене тісто поділяли на шматки масою ( $50 \pm 0,5$  г), округляли та формували хлібці, вистоювали протягом 10 хвилин при 35 °C і відносній вологості 65%, а потім пропарювали при  $100 \pm 5^\circ\text{C}$  протягом 20 хвилин при атмосферному тиску. Приготовані на пару хлібці охолоджували до температури 18-20 °C протягом 1 години.

### **Упаковка, маркування, транспортування і зберігання**

Упаковка, маркування, транспортування і зберігання хлібців проводяться відповідно до вимог маркування товару - згідно Закону України «Про інформацію для споживачів щодо харчових продуктів» від 06.12.2018 № 2639-VIII та вимог ДСТУ 7046:2009 «Вироби хлібобулочні. Укладання, зберігання і транспортування».

Відхилення маси в бік зменшення - не повинно перевищувати 3,0 %, в бік збільшення - не обмежено.

Хлібці зберігають у сухих, чистих, добре вентильованих приміщеннях, що не мають стороннього запаху, не заражених шкідниками хлібних запасів, при температурі ( $18 \pm 5$ ) °C і відносній вологості повітря не більше 75%.

**Строк придатності хлібців «Парових»** складає – 24 год без пакування, запакованих у пакети з полімерної плівки — 48 год.

### **Харчова (поживна) та енергетична цінність (калорійність) 100 г хлібців «Парових»**

Білки, г	Жири, г	Вуглеводи, г	Калорійність, ккал
<b>4,3</b>	<b>0,3</b>	<b>48,5</b>	<b>201,0</b>

### **РОЗРОБЛЕНО:**

аспірантка кафедри технології харчування Сумського національного аграрного університету

Денг Чунлі

## APPENDIX D

## РЕЦЕПТУРА І ТЕХНОЛОГІЧНА ІНСТРУКЦІЯ на локшину «Легка»

Підстава: ДСТУ 7043:2009 «Вироби макаронні. Загальні технічні умови».

### 1. Технічні вимоги.

Локшину «Легка» виготовляють відповідно до вимог цих технічних інструкцій або нормативних документів виробника (ТУ, ТУУ, СОУ тощо, які не суперечать вимогам відповідного ДСТУ), затвердженим у встановленому законодавством України порядку.

Локшина «Легка» – це стрічкоподібні макаронні вироби з гладкою поверхнею та прямими краями з додаванням картопляного крохмалю фізичної модифікації. Короткорізані.

Для виготовлення локшини «Легкої» використовують такі основні види сировини:

борошно пшеничне вищого сорту з низьким вмістом клейковини (10,0 %±1,0%) – ДСТУ 46.004-99

модифікований крохмаль – ТУ У 00383403.001:2023

сіль – ДСТУ 4307:2004

вода - ДСТУ 7525:2014

Вологість 12,0±0,5%.

Для виготовлення локшини «Легкої» можуть використовуватись інші види вітчизняної та імпортової сировини - згідно з чинним нормативним документом або імпортового виробництва за наявності висновку санітарно-епідеміологічної експертизи центрального органу виконавчої влади, що забезпечує формування політики у сфері охорони здоров'я.

За органолептичними показниками локшина «Легка» має відповідати вимогам, зазначеним у таблиці 1.

*Таблиця 1*

Найменування показника	Характеристика
Зовнішній вигляд: колір поверхня форма	Однотонний з кремовим відтінком, без слідів непромісу Гладенька Стрічки з прямими краями довжиною не більше 100,0 мм, ширина до 5,0 мм, товщина – не більше 2,0 мм
Смак і запах	Запах та смак властивий локшині без сторонніх запахів та присмаків
Стан виробів після варіння	Зварені до готовності вироби зберігають форму, не злипаються, не утворюють грудочок



**За фізико-хімічними показниками локшина «Легка» повинна відповідати вимогам, зазначеним в таблиці 2.**

Таблиця 2

Найменування показників	Норма	Метод аналізу
Вологість, %, не більше ніж	$12,0 \pm 0,5$	ГОСТ 14849
Кислотність, град, не більше ніж	$4,0 \pm 0,5$	ГОСТ 14849
Масова частка лому, %, не більше ніж	10,0	ГОСТ 14849
Масова частка деформованих виробів, %, не більше ніж	5,0	ГОСТ 14849
Масова частка крихти, %, не більше ніж	8,0	ГОСТ 14849
Металомагнітні домішки, мг на 1 кг продукту, не більше ніж	3,0 – якщо розмір окремих часток не більше, ніж 0,3 мм у найбільшому лінійному вимірі	ГОСТ 14849
Наявність шкідників хлібних запасів	Не допускається	ГОСТ 14849

**Вміст токсичних елементів і мікотоксинів в локшині «Легкій»** не повинен перевищувати гранично допустимих концентрацій, передбачених МБТиСН 5961 та приведених в таблиці 3.

Таблиця 3

Назва елемента	Гранично допустимі рівні, мг/кг, не більше ніж	Метод контролювання
Токсичні елементи: свинець	0,5	ГОСТ 26932, ГОСТ 30178
кадмій	0,1	ГОСТ 26933, ГОСТ 30178
миш'як	0,2	ГОСТ 26930
ртуть	0,02	ГОСТ 26927
мідь	10,0	ГОСТ 26931, ГОСТ 30178
цинк	50,0	ГОСТ 26934, ГОСТ 30178
Мікотоксини: афлатоксин В <sub>1</sub>	0,005	МР № 2273 (3), МР № 4082 (4), ДСТУ EN 12955
Т-2 токсин	0,1	МУ № 3184 (5)
дезоксиніваленол	0,5	МР № 3940 (6), МУ № 5177 (7)
заараленон	1,0	МР № 2964 (8), МУ № 5177 (7)

**Вміст залишкової кількості пестицидів у локшині** не повинен перевищувати допустимих рівнів, передбачених МБТиСН №5061 і ДСанПіН 8.8.1.2.3.4-000 і їх регламенту у сировині.

**Вміст радіонуклідів у локшині** не повинен перевищувати допустимих рівнів, передбачених ГН 6.6.1.1-130, зазначених у таблиці 4.

Таблиця 4

Назва елемента	Гранично допустимі рівні, Бк/кг, не більше ніж	Метод контролювання
$^{137}\text{Cs}$ (Цезій -137)	20,0	МУ № 5779 (11)
$^{90}\text{Sr}$ (Стронцій-90)	5,0	МУ 5778 (12)

## 2. Вимоги до сировини.

Сировина, що застосовується при виготовленні локшини, повинна бути дозволена до застосування МОЗ України. Якість її повинна відповідати вимогам чинної нормативної документації; за вмістом токсичних елементів, мікотоксинів, пестицидів, нітратів має відповідати «Медико-біологічним вимогам і санітарним нормам якості продовольчої сировини і харчових продуктів № 5061».

**Рецептура на локшину «Легку»** наведена в таблиці 5.

Таблиця 5

Сировина	Масова частка вологи, %	Витрати борошна, кг	
		1	100
Борошно пшеничне з низьким вмістом клейковини	12,0	0,70	70,0
Модифікований крохмаль	12,0	0,30	30,0
Вода	100,0	0,48	48,0
Всього	-	1,48	148,0

## Технологічна інструкція виробництва локшини «Легкої»

Приготування локшини складається з наступних стадій:

- підготовка сировини до виробництва;
- замішування тіста;
- формування і випресовування;
- нарізання на шматки необхідної довжини;
- сушіння та охолодження;
- упаковка, маркування, транспортування і зберігання.

### Підготовка сировини до виробництва

Підготовка сировини до виробництва здійснюється відповідно до Технологічних інструкцій для виробництва макаронних виробів. Допускається взаємозамінність сировини відповідно до Вказівок до рецептур на макаронні вироби.

### **Замішування та формування тіста**

Тісто формували шляхом змішування пшеничного борошна, модифікованого крохмалю (HMTS або MWS), додавали воду, замішували протягом 2 хв вручну. Отримане тісто накривали поліетиленовою плівкою і залишали на 15 хв при температурі 18-20 °С. Потім тісто знову замішували протягом 1 хв і направляли на формування шляхом випресовування.

### **Сушіння та охолодження локшини**

Сушіння локшини проводили у сушильній шафі при температурі 60-80 °С протягом 2,5-3,0 годин. Після сушіння локшину охолоджували до температури 18-20 °С протягом 1-2 годин.

### **Упаковка, маркування, транспортування і зберігання**

Упаковка, маркування, транспортування і зберігання локшини проводяться відповідно до вимог маркування товару - згідно Закону України «Про інформацію для споживачів щодо харчових продуктів» від 06.12.2018 № 2639-VIII та вимог ДСТУ 4518:2008 «Продукти харчові. Маркування для споживачів. Загальні правила», ДСТУ ISO 780-2001 «Пакування. Графічне маркування щодо поводження з товарами».

Локшину фасували у поліетиленові пакети масою 0,5 кг: відхилення маси нетто в меншу сторону від установленної маси нетто одиниці пакування не повинні перевищувати:

1,0 % - від середньої маси нетто 10 одиниць пакування,

2,0 % - від маси нетто одиниці пакування.

Відхилення маси нетто в більшу сторону від установленної маси нетто не обмежено.

Локшину зберігають у сухих, чистих, добре вентильованих приміщеннях, що не мають стороннього запаху, не заражених шкідниками хлібних запасів, при температурі (18±5) °С і відносній вологості повітря не більше 75%.

### **Харчова (поживна) та енергетична цінність (калорійність) 100 г локшини «Легкої»**

Білки, г	Жири, г	Вуглеводи, г	Калорійність, ккал
<b>7,9</b>	<b>0,5</b>	<b>77,6</b>	<b>346,5</b>

### **РОЗРОБЛЕНО:**

аспірантка кафедри технології харчування Сумського національного аграрного університету

Денг Чунлі

**APPENDIX E****CERTIFICATE OF PRODUCT APPLICATION**

Since June 10, 2022, Hezhou Xianhe Health Technology Co., Ltd. has conducted pilot tests on the product formula and processing technology of “Modified potato Starch Cookies” provided by Deng Chunli. The modified potato starch cookies products are crunchy and delicious, with good nutritional value, and they are in line with our company’s product needs. Furthermore, the company puts the product on the market in July 2022 for trial sale, which is deeply loved by consumers and has brought direct economic benefits to our company more than RMB 20,000 yuan.

Hezhou Xianhe Health Technology Co., Ltd.

October 20, 2022



## CERTIFICATE OF PRODUCT APPLICATION

Since August 10, 2022, Hezhou Xianhe Health Technology Co., Ltd. has conducted pilot tests on the product formula and processing technology of “Modified potato Starch Fresh Noodles” provided by Deng Chunli. The modified potato starch fresh noodles products have good cooking properties and taste with good nutritional value and economic value, and they are in line with the company’s product needs. Furthermore, the company puts the product on the market in October 2022 for trial sale, which is deeply loved by consumers and has brought direct economic benefits to our company more than RMB10,000 yuan.

Hezhou Xianhe Health Technology Co., Ltd.

December 15, 2022



## CERTIFICATE OF PRODUCT APPLICATION

Since November 10, 2022, Hezhou Xianhe Health Technology Co., Ltd. has conducted pilot tests on the product formula and processing technology of “Modified potato Starch Steamed Breads” provided by Deng Chunli. The modified potato starch steamed breads products have good specific volume and texture properties with good nutritional value and economic value, and they are in line with our company’s product needs. Furthermore, the company puts the product on the market in January 2023 for trial sale, which is deeply loved by consumers and has brought direct economic benefits to our company more than RMB15,000 yuan.

Hezhou Xianhe Health Technology Co., Ltd.

March 10, 2023

