

## RESEARCH IR SPECTRA PECTINS AND GELS ON THEIR BASIS

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**Abstract:** Studied the IR-spectra high-ester (CE = 62 - 66%) and of low-ester (CE = 31 - 36%) apple pectin in the solid phase. In order to detect differences in the structure formation of these pectins studied the IR spectra of films of dry gels (system pectin-sugar-citric acid). Comparison of the obtained IR spectra of dried gels from high-ester and low-ester apple pectins showed that for the first mentioned pectin formed a solid gel structure.

**Keywords:** pectin, degree of esterification, structure formation, film of gel, IR-spectra.

### 1. Introduction

In recent years, worldwide production of pectin increased almost twice. Unique properties of pectin as a food and biological additives resulted in his special role in the economy of developed countries. Pectin is widely used in various sectors of the food industry as gel former, stabilizer, thickener and emulsifier [1, 2, 3]. Accordingly, the actual development pectin industry in Ukraine is a complex of theoretical and experimental studies to the scientific basis and the development of modern, highly efficient technologies pectin and products on pectin basis [4].

During the acid extraction of fruits and vegetables (beets, apples, citrus) are excluded pectins, which constitute a group of commercial pectin. For these pectins are usually used marc citrus and apples - a byproduct in obtaining juice and beet pulp - waste of sugar from sugar beets. For apple and citrus pectins characterized by alternating linear 1, 4 - linked chains  $\alpha$ , D - galacturonan and extensive area, which contains most of the neutral polysaccharides. These pectins differ in molecular weight and content of rhamnose, arabinose, galactose and other monosaccharides.

Physicochemical properties of commercial pectins depend on their molecular weight and degree of esterification [5]. The degree of esterification is determined by the number of moles of methanol per 100 mol of galacturonic acid. Pectins with high degree of esterification containing more than 50% esterified galacturonan residues. Low-ester pectins contain less than 50% esterified residues. Both groups of pectin polysaccharides form a gels, but under different conditions: for low-ester pectin requires low values of pH or the presence of calcium ions, and

for high-ester pectin gel formation occurs by hydrogen bonds and hydrophobic interactions.

### 2. Experimental part

The aim of our work was to investigate the IR-spectra of dry films of pectin gels with different degrees of esterification and identifying differences in the structure formation of its pectins.

Terms of characteristic absorption bands of IR-spectra gel formers and their intensity determined by the nature of atomic groups that are part of macromolecules and their location relative to each other [6]. If the composition of the macromolecules are ionic group, while a characteristic absorption band depends on the nature and degree of oxidation of low molecular counterions. IR-spectra can provide additional information on possible interactions of macromolecules with low molecular weight additives to form associates. Spectra analysis also provides information on how these atomic groups of macromolecules involved in molecular interactions, if sufficiently strong effect on the oscillation frequency of connections.

Research pectins by IR-spectroscopy were started much later than other polysaccharides. The considered only the most intense and characteristic bands. It is possible to identify the IR spectra pectin in food products and to distinguish them from other gel formers. Later research began IR-spectra of pectin derivatives obtained by oxidation of pectin and stitching his chains [7, 8].

Compared to the numerous studies of polysaccharides works connect with infrared spectroscopy of pectins bit. This is due to the complexity of the allocation of these substances

in a state close to the native and imperfect methods of preparing samples for analysis.

The first phase of work we have obtained IR-spectra high-ester (CE = 62 - 66%) and of low-ester (CE = 31 - 36%) of apple pectin in crystalline state (solid phase). Samples of pectin are homogenized with optically pure dry potassium bromide. As a result, pressing the mixture obtained pills size  $22 \times 5$  mm and weighing 150 mg. Spectra were relatively potassium bromide in the region from  $4000 \text{ cm}^{-1}$  to  $400 \text{ cm}^{-1}$  on a spectrophotometer with Fourier transformation of TENSOR 27. In such spectrophotometers used polychromatic radiation

and calculated spectrum in a given frequency range by Fourier conversion output.

### 3. Results and discussion

The obtained IR-spectra of pectins are shown in Fig. 1 and Fig. 2.

Table 1 shows the results of the analysis obtained IR-spectra of high-ester and low-ester apple pectins in the solid phase.

The second phase of work, we investigated the IR-spectra of gels on the basis of high-ester and low-ester apple pectins, compounding the composition of which is given in Table. 2.

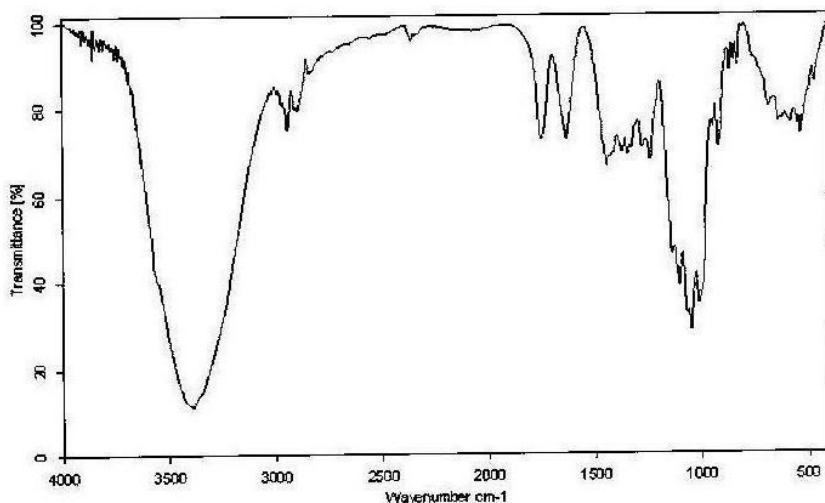


Figure 1. IR-spectra of dry high-ester apple pectin

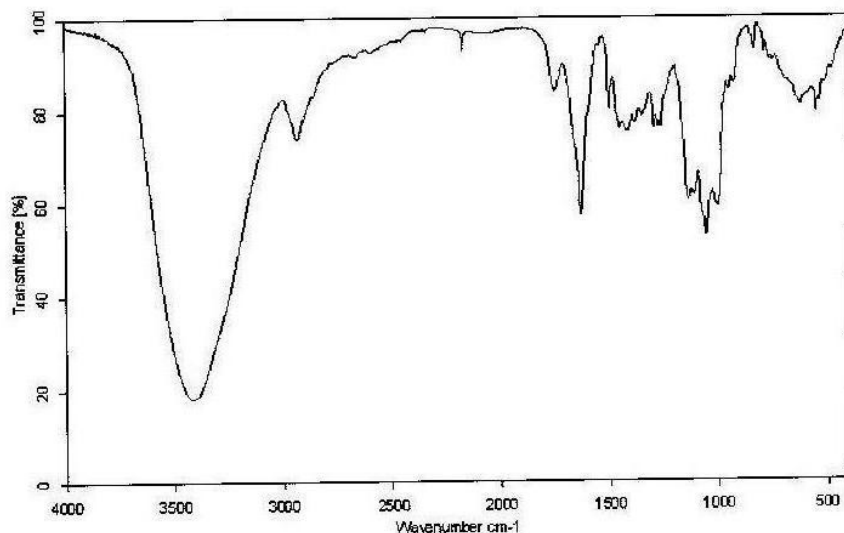


Figure 2. IR-spectra of dry low-ester apple pectin

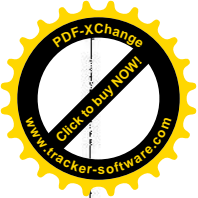


Table 1. The results of IR-spectra of high-ester and low-ester apple pectin in the solid phase

High-ester apple pectin		Low-ester apple pectin		The assignment of bands	Conclusions
Areas of peaks, cm <sup>-1</sup>	Skipping, %	Areas of peaks, cm <sup>-1</sup>	Skipping, %		
3389,37	10	3411,86	14	stretching vibrations of OH-related H-bond	strong broad peak, characteristic of carbohydrates
2942,21	72	2944,26	81	covalent bond stretching vibrations of C-H (condition C - sp <sup>3</sup> hybridization)	at methylation of carboxyl group absorption increases
2895,87	79	2897,62	83		
2837,00	84	2838,02	87		
1751,47	70	1754,99	85	stretching vibrations of carbonyl C = O ester groups	for high-ester pectin absorption increases due to more difficult-essential groups
1631,26	70	1628,56	57	stretching vibrations of COO <sup>-</sup>	for low-ester pectin absorption more due to dissociation is not methylated carboxyl groups
1442,18 – 1238,34	65	1494,72 – 1272,99	80 – 75	internal deformation vibrations of methyl group-CH <sub>3</sub> ; stretching vibrations of air the C-O-C.	for high-ester pectin absorption increases due to more difficult-essential groups
1104,46	39	1133,62	60	stretching vibrations of the C-C and C-O piranoznych cycles and C-O-C ether bridges.	for high-ester pectin absorption more due to more difficult-essential groups
1051,40	27	1052,55	50		
1012,56	33	999,4	55		
920,77	69	948,42	82	pendulum oscillations in hard-CH <sub>3</sub> ester groups	for high-ester pectin absorption more due to more difficult-essential groups

Table 2. Composition of pectin gels

raw	Mass fraction of dry substances, %	Total cost of raw materials taking into account losses in the technological process, 100 g	
		In nature	In solids
Apple pectin	92,00	1,00	0,92
Sugar	99,85	65,00	64,90
Citric	91,20	0,80	0,73
Water		33,20	-
Total		100,00	66,55



Difficulty applying IR-spectroscopy to study gels is that the presence of significant amounts of water leads to an overlap of nearly all the characteristic bands of macromolecules. Therefore, IR-spectroscopy gel formers preferred method of removing the spectra in the form of films [9]. This is due to the fact that the manufacture of polymer films can not be destruction, and in the spectra due to the homogeneity of the samples are usually not observed violations by the uneven distribution of compounds.

For film samples pectin gel thin layer deposited on a plastic base and dried at  $20 \pm 1,0 \text{ }^\circ\text{C}$  in a desiccators. The dried film is well shot with the basics. Spectra were relatively potassium bromide in the region from  $4000 \text{ cm}^{-1}$  to  $400 \text{ cm}^{-1}$  on a spectrophotometer with Fourier transformation of TENSOR 27. In Fig. 3 and Fig. 4 shows the IR-spectra of films of gels based on high-ester and low-ester apple pectin taken relatively pure pectin.

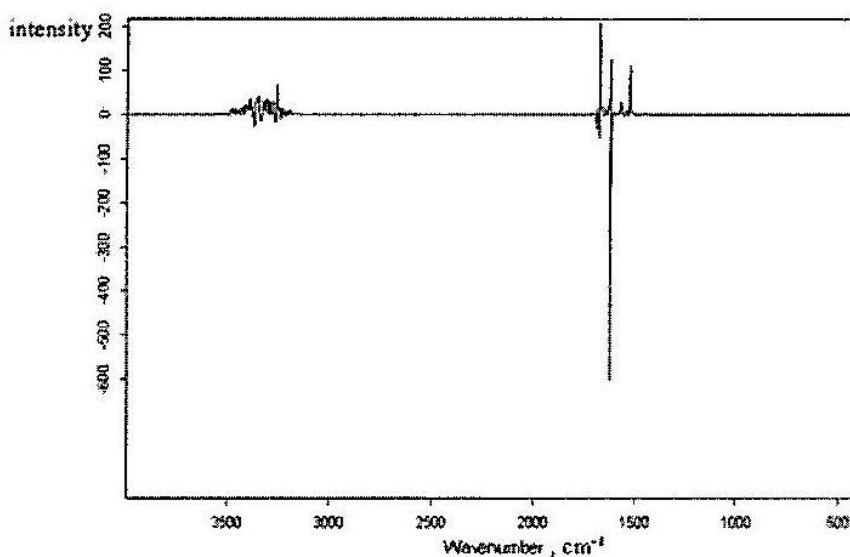


Figure 3. IR spectrum of the gel from high-ester apple pectin

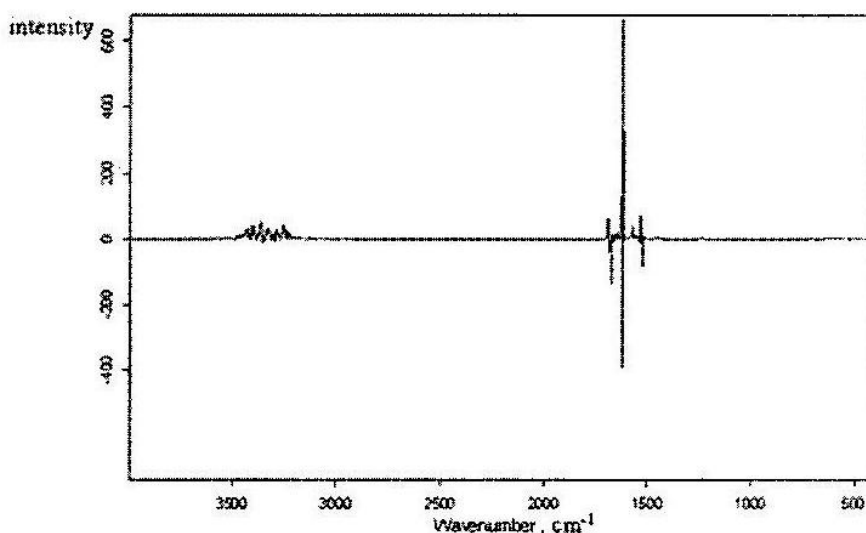


Figure 4. IR spectrum of the gel-based low-ester apple pectin



When comparing the absorption peaks within  $3366,81\text{ cm}^{-1} - 3240,12\text{ cm}^{-1}$  for high-ester pectin and  $3426,31\text{ cm}^{-1} - 3272,52\text{ cm}^{-1}$  - of low-ester pectin, it is clear that the first of these peaks shifted low-frequency region. These peaks correspond to vibrations of free hydroxyl groups. For both samples pectin compared with the frequency of oscillation of free hydroxyl groups, these peaks are shifted in the low-frequency region. This is due to the participation of hydroxyls in the system of H-bonds.

For high-ester pectin is greater absorption intensity and frequency lower. This suggests an increase in the strength of H-bonds in comparison with the model based of low-ester pectin.

The second area of peak absorption for high-ester pectin is between  $1689,70\text{ cm}^{-1} - 1528,42\text{ cm}^{-1}$ , and for low-ester pectin –  $1687,04\text{ cm}^{-1} - 1524,96\text{ cm}^{-1}$ . Maximum absorption falls to  $1618,06\text{ cm}^{-1}$  for the sample on the basis of high-ester pectin and  $1620,43\text{ cm}^{-1}$  – sample of low-ester pectin. Such pikes absorption corresponding fluctuations dissociated carboxyl groups  $\text{COO}^-$ . Acquisitions in the above areas for samples from both pectins close.

Comparison of IR-spectra obtained films gels (system pectin-sugar-citric acid) on the basis of high-ester and low-ester pectins showed that for the first mentioned pectin produced more robust gel structure.

### Conclusions

Studied the infrared spectra high-ester (CE = 62 - 66%) and low-ester (CE = 31 - 36%) of apple pectin in the solid phase.

Studied the infrared spectra of dry films jelly pectin with different degrees of esterification to detect differences in the structure formation of pectin.

Found that gel (pectin-sugar-citric acid) from high-ester apple pectin produced more robust structure compared with the gel-based of low apple pectin.

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