

# Enhancement of Mechanical, Electrical and Chemical Properties of Polycarbonate-Based Core-Shell Composites by Modification with Single-Walled Carbon Nanotubes

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## Abstract

The paper describes mechanical, electrical and chemical properties of modified polycarbonate obtained by hot compression moulding of core-shell composites consisting of a polymer core covered with a shell containing single-walled carbon nanotubes (SWCNTs). Such an approach in material design based on an ultralow modification with nanomaterials allows obtaining a segregated 3D network of SWCNTs, which can provide a functionalisation of material and a significant enhancement of mechanical, chemical and electrical properties. It was observed that impact strength of polycarbonate modified with SWCNTs was increased almost by 2.5 times even at ultra-low SWCNTs concentration of 0.0024 wt.% and reached more than 5 times increase at 0.01 wt.% of SWCNTs. Chemical resistance of the sample with 0.01 wt.% of SWCNTs was improved by 2.4 times when exposed to ammonia. At the same time, for other selected solvents any positive or negative effect of SWCNTs was not demonstrated, as well as the hardness of modified polycarbonate was not affected by SWCNTs. Electrical percolation threshold was defined at ultra-low level of 0.0010-0.0012 wt.% of SWCNTs.

**Key words:** carbon nanotubes, composites, segregated network, chemical resistance, impact strength, polycarbonate modification, thermoplastic reinforcement.

## 1. Introduction

To overcome limits of the mechanical and physical properties of the traditional materials a composite structure design is one of the best choices allowing a reinforcement of the materials (Pogrebnjak et al., 2012; Hasan et al., 2019; Gibson, 2010). On the other hand, last few decades have shown a growing potential of nanocarbon materials, especially carbon nanotubes (CNTs) in terms of their recognised outstanding mechanical and functional properties (Iijima, 1991; Kinloch et al., 2018; Harris, 1999; Ajayan et al., 2007). Nowadays a concept of using CNTs as fillers in reinforced polymer composites is a common approach to design advanced tough materials with additional

functionalisation (Mittal et al., 2015; Soni et al., 2020; Kim et al., 2009; Thomassin et al., 2013). As seen in

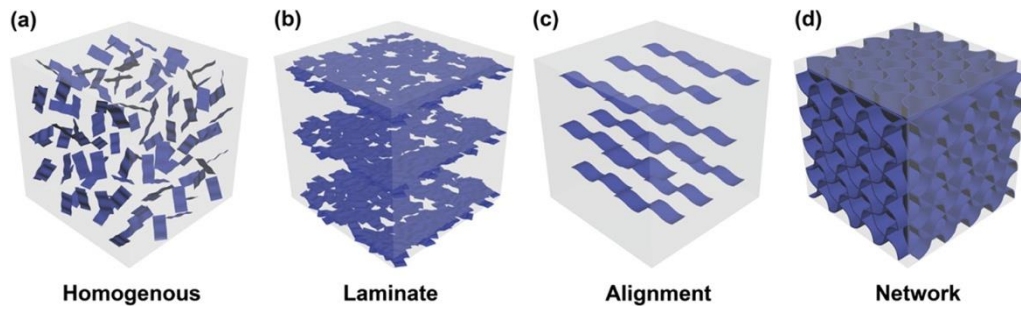


Figure 1, there are four main architectures of nanocarbon reinforced composites, where each one is obtained using specific fabrication strategies.

Segregated CNTs network is characterised by a continuous intergranular filler distribution. The strategy of the fabrication methods adopted for segregated network architecture is to encapsulate a matrix material (powder, granules, etc.) with nanocarbon material and then form a continuous network by employing densification processes (Zhang et al., 2020).

The main challenge in this approach is how to ensure a homogeneous dispersion of the nanocarbon material on the surface of the matrix components and improve the densification process of the composites.

The distribution of conductive fillers in the boundaries area between polymeric particles is the main geometric feature of segregated structure which allows reducing the percolation values of such composites by several orders of magnitude in comparison to those prepared by conventional melting methods (Zhang et al., 2020).

The network-like distribution of CNTs in the polymer matrix is a practical way to develop material with extraordinary isotropic mechanical properties at low CNTs contents (Xu et al., 2020). However, X. Zhang et al. in their review-paper (Zhang et al., 2020) concluded that a full potential of CNTs network architecture has not been explored and has not been reached yet due to the current imperfect preparation methods.

In terms of functionalisation of polymers by inducing electrical conductivity, polymer/CNTs composite network architecture is a facile and effective method. Different network architectures resulted from different fabrication techniques lead to different conducting properties of the composite and, usually, allow to reduce the percolation threshold and obtain a high electrical conductivity with minimum content of nanocarbon fillers (Zhang et al., 2020; Wu et al., 2021). Segregated conductive CNTs networks successfully demonstrated high electrical conductivity in polymer matrix (Wang et al., 2017; Shrivastava et al., 2011).

Since conductive fillers in the segregated network are distributed in the intervals between polymer components (granules, powder, etc.) and not throughout the entire volume of the composites, conductive pathways are formed from highly concentrated nanocarbon fillers, which

leads to better contact between fillers, denser conductive pathways and lower percolation thresholds. Conductive properties of polymer/CNTs composite segregated network were reported even at ultralow CNTs concentration and percolation thresholds (Wang et al., 2017; Li et al., 2015; Maiti et al., 2014; Cui et al., 2016). However, highly dispersed nanocarbon fillers are essential to avoid nanocarbon agglomerations which deteriorate conductive properties of segregated network polymer/CNTs composites (Alig et al., 2012). At the same time, many works also suggest that a presence of carbon nanotube agglomerations at some optimal level may improve conductivity (Tarlton et al., 2017; Watt et al., 2020).

In this paper the properties of polycarbonate/SWCNTs composite with segregated network obtained by advanced fabrication method ensuring highly dispersed CNTs with no agglomerations will be investigated. Since the details of the used fabrication method are a trade secret, the paper is focused on a study of SWCNTs segregated network effect on mechanical, physical and chemical properties of the modified polycarbonates, in particular, their hardness, impact strength, chemical resistance and electrical resistivity.

## **2. Experimental Details**

### **2.1. Samples Preparation**

Commercial polycarbonate Makrolon ET3117 (Material Data Center et al., 2021) was used as a polymer to be modified with SWCNTs. TUBALL SWCNTs with dimensions of  $1.6\pm 0.4$  nm in diameter and 5  $\mu$ m length (1017 nanotubes in 1 g) produced by OCSiAl were used for the polycarbonate granules' surface modification. Detailed method how the polycarbonate was modified with SWCNTs is a trade secret and cannot be disclosed within this paper.

Modified polycarbonate granules have a thick layer (up to 1  $\mu$ m) of material containing dispersed and deagglomerated nanotubes. The core of the granule is still a pure polycarbonate. Concentration of nanotubes on the surface depends on exposure time and applied technological parameters. Series of samples with different SWCNTs concentrations have been produced. Later in this paper the concentration of SWCNTs is assumed as an average concentration within the grain, considering their presence at the near-surface layer only.

Discs of the modified polycarbonate were obtained from granules by hot compression moulding using a manual screw press. The mould was preheated up to 220-245°C with the grains for 5 minutes. At this temperature the polycarbonate is in a viscous state. The pressing load was 450 N. As a result, segregated 3D network of nanotubes has been achieved. It was possible to dope the near-surface volume of the polycarbonate granules with an ultra-low concentration of nanotubes (from 0.001 to 0.01 wt.% within the polycarbonate grains).

### **2.2. Samples Characterisation**

Microstructural characterisation was performed by variable pressure field emission scanning electron microscope Zeiss SIGMA VP at accelerated voltage of 0.4-0.8 kV.

Raman spectroscopy analysis was performed using DXR2 Raman microscope produced by Thermo Fisher Scientific.

Mechanical properties were studied by Rockwell hardness test (scale E, steel sphere indenter with a diameter of 3.175 mm, 980.7 N load) and Charpy impact strength test (pendulum pile-driver with a mass of 1.09 kg, length 0.43 m, angle 90°, samples with 5 mm width and 4 mm thickness were cut from the hot moulded polycarbonate discs). To consider only a contribution of SWCNTs and avoid a role of the thermoplastic fabrication method, a relative Charpy impact strength is considered with normalisation of obtained values to the reference sample of pure polycarbonate (unmodified thermoplastic without SWCNTs).

Chemical resistance of modified polycarbonate samples was examined by immersion of samples in aggressive environments for two weeks: ethyl acetate, gasoline, ammonia, solvent 647 (butyl- or amyl acetate 29.8 vol.%, ethyl acetate 1.2 vol.%, butanol 7.7 vol.%, toluene or pyrobenzene 41.3 vol.%). The appearance of the samples was evaluated before and after the experiment. The samples were weighted before the experiment, immediately after extraction from the liquids and after the drying.

Basic electrical properties of polycarbonate modified with SWCNTs were studied by measuring surface resistance (two points method) using multi tester Vichy VC890+ with standard probes at distance of 40 and 5 mm.

### **3. Results and Discussion**

#### **3.1. Disc Samples of Polycarbonate Granules Modified with SWCNTs**

Polycarbonate Makrolon ET3117 granules modified with TUBALL SWCNTs have been obtained. Nanotubes concentration in modified granules varies from 0.0006 to 0.01 wt.%. Cylindrical granules are transparent, slightly mate, with some degree of a grey tint. It is possible to estimate a degree of modification with SWCNTs by the colour: the darker the sample, the higher the concentration of nanotubes. Disc samples of the SWCNTs-modified Makrolon ET3117 polycarbonate have been obtained by hot compressing moulding. Details about the samples selected for further study are shown in Table 1.

Polycarbonate discs obtained from the SWCNTs-modified granules by hot compression moulding are shown in Figure 2. Samples have diameter of 45 mm and thickness of 3.5-4.5 mm. The 3D network formed by modifier (SWCNTs) is clearly seen in the transparent medium of the sample.

#### **3.1. Microstructural Characterisation**

Microstructural characterisation has been performed to study real dimensions of the nanotubes, their distribution on the surface of polycarbonate granules, confirm or deny the presence of nanotubes agglomerations which could decrease the efficiency of nanotubes or even serve for stress concentration. Surface of the polycarbonate granule modified with SWCNTs is shown in Figure 3 (a) and demonstrates mostly homogeneous distribution of fibres. Slightly higher concentration of nanotubes can be observed in some areas. However, in the final product obtained by hot compression moulding a plastic redistribution of the material occurs and nanotubes relocate, which results in their more uniform distribution on the surface of granules. Due to the limited resolution of the microscope it was not possible to measure a separate nanotube, thus, a thread with a diameter of 35 nm was measured, as shown in Figure 3 (b).

It was observed that the length of the nanotubes is by tens orders higher than their diameter. Nanotubes are distributed as fibres and due to their fibre design can significantly improve a toughness of a core-material by nano-reinforcement as a fibrous filler. Moreover, the absence of nanotube agglomerations avoids a potential weakening of the material.

To identify carbon forms present in the polycarbonate modified with SWCNTs Raman spectroscopy was performed. Obtained Raman spectrum, typical for single-walled carbon nanotubes, is shown in Figure 4.

The highest double peak corresponds to the "G" band in SWCNTs: the stronger peak at  $1590\text{ cm}^{-1}$  is associated with vibrations of carbon atoms along the nanotube axis, and the smaller peak at  $1570\text{ cm}^{-1}$  is associated with vibrations of carbon atoms along the circumferential direction of SWCNTs. Another distinguishing and unique feature of SWCNTs is the lower frequency radial breathing mode (RBM) usually appearing with frequencies between  $120\text{-}350\text{ cm}^{-1}$  (Dresselhaus et al., 2005). Raman spectrum demonstrates low level of defects and high quality of SWCNTs as seen from the low intensity of "D" band peak around  $1340\text{ cm}^{-1}$ . Some contribution to the peak at  $2670\text{ cm}^{-1}$  is associated with 2D band can come from graphite and graphene since the declared purity of TUBALL SWCNTs is  $\geq 80\text{ wt.}\%$  (TUBALL, 2020).

### **3.2. Mechanical Properties**

Results of hardness measurements by Rockwell test are shown in Figure 5. As seen, modification of polycarbonate granules with SWCNTs doesn't cause changes in mechanical properties of the studied samples. This is explained by an ultra-low amount of SWCNTs used for the modification of polycarbonate grains. Thus, only the polycarbonate matrix is mainly considered as a one contributing to the hardness values. A slight difference in hardness of studied samples can be related to the difference in the moulding parameters (load, heating rate, temperature, time, etc.) or local defects on the surface of the fabricated samples.

Results of Charpy impact strength are shown in Figure 6. The experiment shown that even at ultra-low concentration, 0.0024 wt.% of SWCNTs, the impact strength of produced thermoplastic can be increased by almost 2.5 times. Further increase of SWCNTs concentration to 0.0064 wt.% leads to 4.2 times higher impact strength in comparison to the pure polycarbonate. The best value of impact strength was achieved for Sample 10 with 0.01 wt.% of SWCNTs. It has 5.2 times higher impact strength than Sample 1 made of unmodified granules. It is explained by the formation of 3D network, or matrix, made of carbon nanotubes. It may be assumed that the optimal fraction of SWCNTs on the surface of the modified polycarbonate grains can be higher than in the mentioned sample, however, the study on the samples with higher degree of polycarbonate modification by SWCNTs hasn't been performed within this research.

### **3.3. Chemical Resistance**

Chemical resistance of modified polycarbonate has been tested in various aggressive chemically active environments and results are shown in Figure 7.

Figure 7 (a) demonstrates results of pure polycarbonate and samples modified with SWCNTs immersion in gasoline for two weeks. As seen from the chart, no changes in mass were observed. Also, no effect of immersion in gasoline is concluded from the photos of the samples after the experiment. It means that polycarbonate is chemically stable in gasoline and nanotube network doesn't change its chemical resistance to gasoline.

Results of the experiments performed in ethyl acetate are shown in Figure 7 (b). An increase in a mass of all samples after the exposure to ethyl acetate indicates a chemical destruction that occurred in the samples, and it could not be suppressed by a presence of carbon nanotubes. Moreover, a cracking of samples was observed at the intergranular interfaces of fabricated thermoplastics. Thus, in case of ethyl acetate, nanotubes cannot be recommended to increase the chemical resistance of polycarbonate.

In case of solvent 647 results shown in Figure 7 (c) demonstrate a variation in samples' mass change. However, within the performed experiment and obtained data it was not possible to come to an evident conclusion in regard to the role of carbon nanotubes in the enhancement of chemical resistance of the polycarbonate materials exposed to the solvent. A much more significant effect of polycarbonate modification by SWCNTs is demonstrated in the following experiment reported below.

The results of immersion of polycarbonate-SWCNTs samples in ammonia for 2 weeks are shown in Figure 7 (d). As seen from the chart, a significant weight loss has occurred for all studied samples in such extreme conditions. As expected, polycarbonate is not resistant to ammonia and Sample 1 (pure polycarbonate) lost 61% of its weight after 2 weeks. However, carbon nanotubes which are chemically stable in ammonia solution can improve chemical resistance of the modified

material and reduce mass loss. At presence of 0.01 wt.% SWCNTs in Sample 10 the mass loss was only 25%, which means 2.4 times lower corrosion rate in comparison to unmodified polycarbonate. When polycarbonate granules are covered by carbon nanotubes shell, the diffusion of ammonia into the polycarbonate grain is impeded and, thus, its destruction is delayed. Therefore, chemical resistance of polycarbonate or polycarbonate-based composites can be significantly improved by the modification with an optimal amount of carbon nanotubes and appropriate fabrication processes.

### **3.4. Electrical Properties**

Results of surface resistance measurements are shown in Figure 8. It is well known that pure polycarbonate is a good electrical insulator. Also, it should be noted that for Sample 1 of unmodified polycarbonate and Samples 2 and 3 modified with 0.0006 and 0.0009 wt.% SWCNTs the values of measured surface resistance were out of a maximum range of the measuring device (more than 20 MOhm). Thus, it was assumed that Samples 1-3 are dielectrics.

As seen from Figure 8, polycarbonate modified with 0.0012 wt.% SWCNTs (Sample 4) demonstrates much lower surface resistivity. Since Sample 3 (0.0009 wt.% SWCNTs) is considered as a nonconductive material, one may conclude that a percolation threshold in the studied series of sample is in the range of 0.0010-0.0012 wt.% SWCNTs. It is seen that with a further increase of SWCNTs fraction to 0.0051-0.0069 wt.% surface resistance continues to decrease to 40-80 kOhm. It is important to note that values of surface resistivity obtained by two-probe method give qualitative comparison and trend rather than precise quantitative information since they highly depend on the quality of the samples and a local structure of the material near to the probes: polycarbonate surface and CNTs interface/edge demonstrate different local electrical properties.

## **4. Conclusions**

A low dose of SWCNTs in the surface layer of modified polycarbonate granules does not affect the hardness of the moulded polycarbonate.

3D matrix of SWCNTs may increase impact strength of hot compression moulded polycarbonate core-shell composites even at ultra-low concentrations. It was observed that at 0.0064 wt.% and 0.01 wt.% of SWCNTs the impact strength of the modified polycarbonate may be increased at least by up to 4.2 and 5.3 times, respectively.

Modification of polycarbonate with SWCNTs may improve chemical resistance of the material against aggressive environment such as ammonia solution. It was shown that the presence of 0.01 wt.% SWCNTs in the polycarbonate retards destruction by 2.4 times. Other aggressive environments, such as solvent 647 and ethyl acetate destroy polycarbonate by chemical

destruction, partial dissolution and partial swelling. The presence of CNTs doesn't change an excellent resistance of polycarbonate to gasoline.

The effect of SWCNTs on electrical properties of polycarbonate was observed at ultra-low degree of modification. A significant decrease of surface resistance was observed even at 0.0012 wt.% of SWCNTs with further enhancement of electrical conductivity at 0.0051-0.0069 wt.% of SWCNTs. Electrical percolation threshold was defined at ultra-low level of 0.0010-0.0012 wt.% of SWCNTs.

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Table 1. Samples of polycarbonate modified with SWCNTs and their concentration.

| Sample Number | SWCNTs concentration, wt.%   |
|---------------|------------------------------|
| Sample 1      | 0 (unmodified polycarbonate) |
| Sample 2      | 0.0006                       |
| Sample 3      | 0.0009                       |
| Sample 4      | 0.0012                       |
| Sample 5      | 0.0024                       |
| Sample 6      | 0.0038                       |
| Sample 7      | 0.0051                       |
| Sample 8      | 0.0064                       |
| Sample 9      | 0.0070                       |
| Sample 10     | 0.01                         |

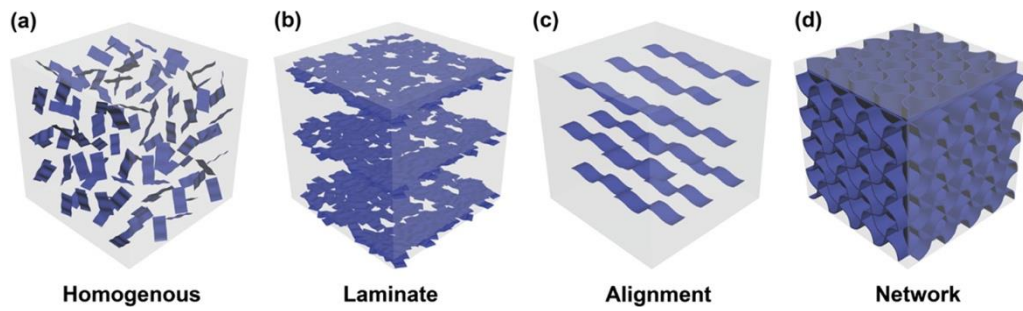


Figure 1. Schematic illustration of four types of nanocarbon architectures: (a) homogenous, (b) laminate, (c) alignment and (d) network. Reprinted from Progress in Materials Science, Vol. 113, Zhang, X., Zhao, N. and He, C., The Superior Mechanical and Physical Properties of Nanocarbon Reinforced Bulk Composites Achieved by Architecture Design – A Review, p. 100672, Copyright 2020, with permission from Elsevier.

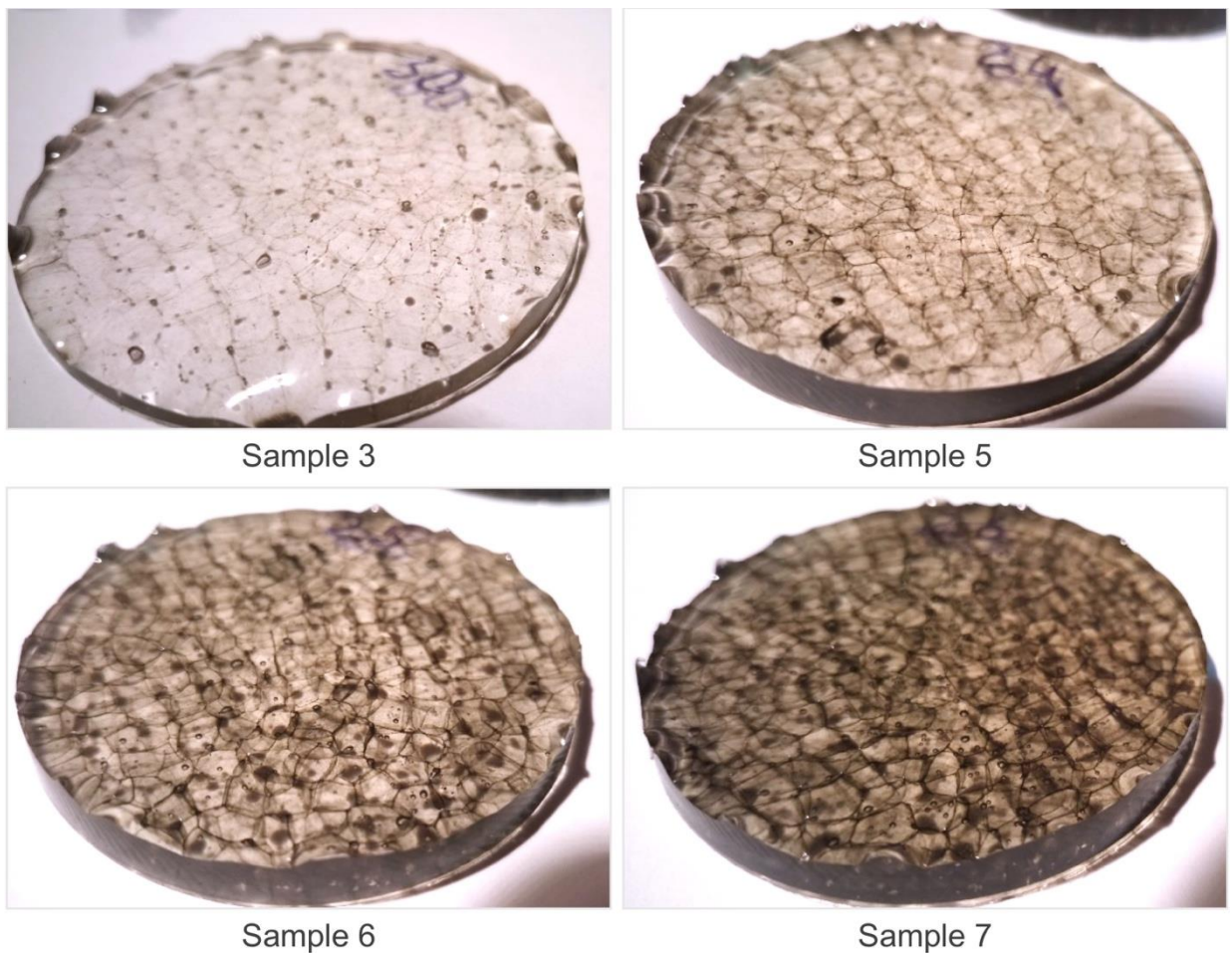


Figure 2. Segregated 3D network of SWCNTs in modified polycarbonate samples.

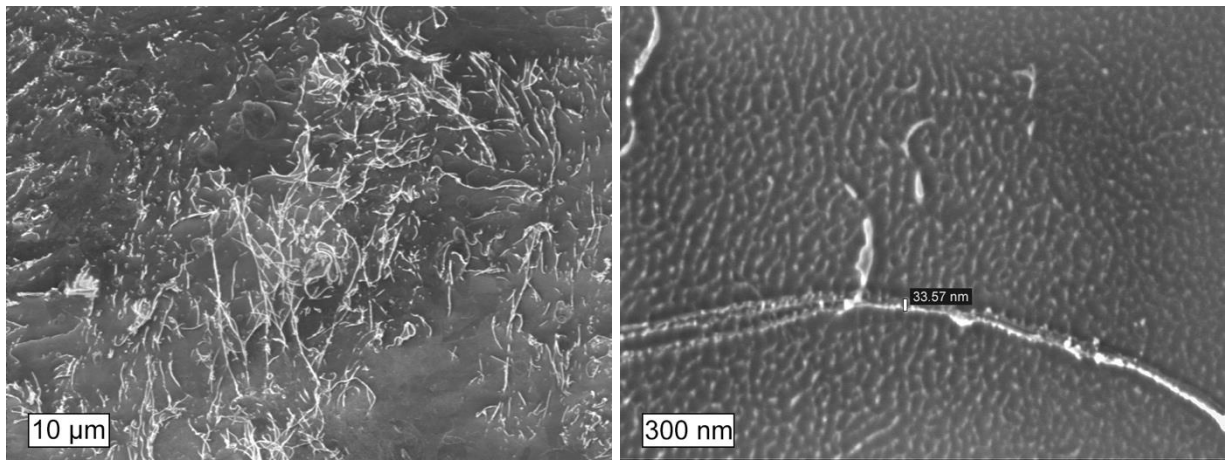


Figure 3. SEM-image of polycarbonate granule surface modified with SWCNTs.

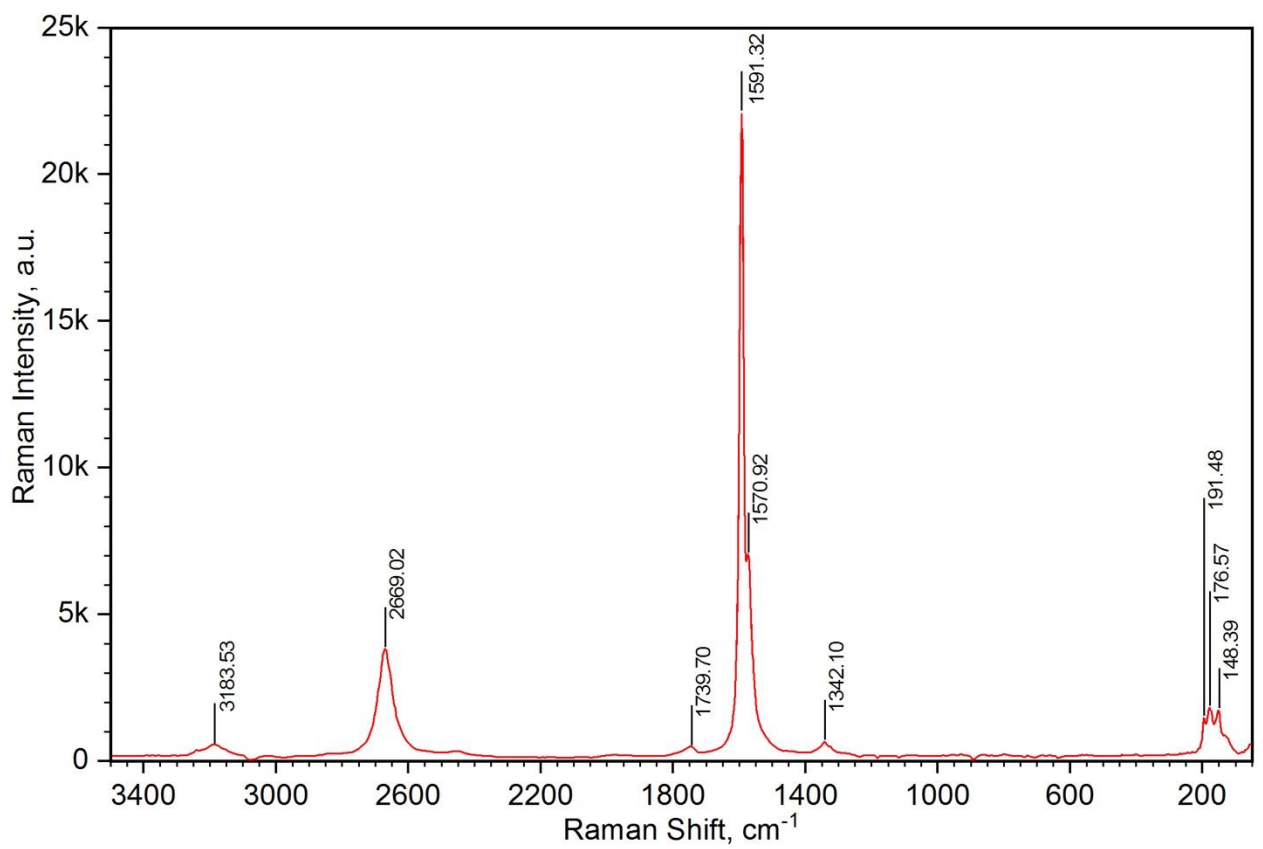


Figure 4. Raman spectrum of single-walled carbon nanotubes on surface of modified polycrystalline grain.

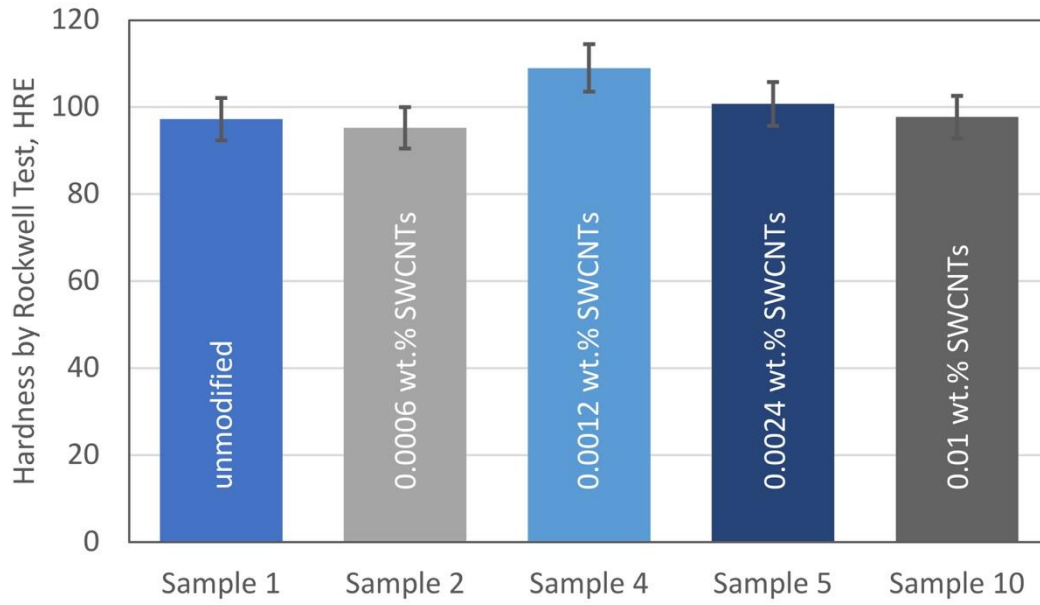


Figure 5. Hardness of polycarbonate discs by Rockwell test, RHE.

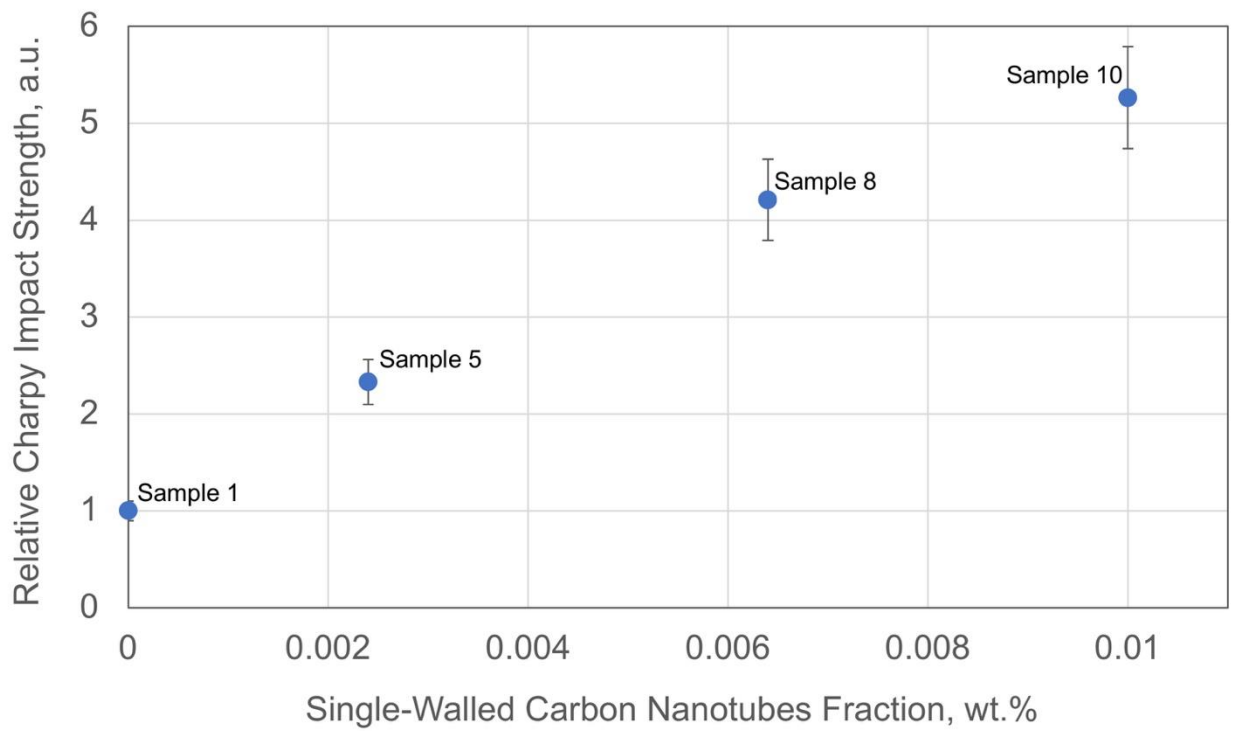


Figure 6. The Charpy impact test results for polycarbonate modified with SWCNTs (relative values normalised to the reference sample of pure polycarbonate without SWCNTs).

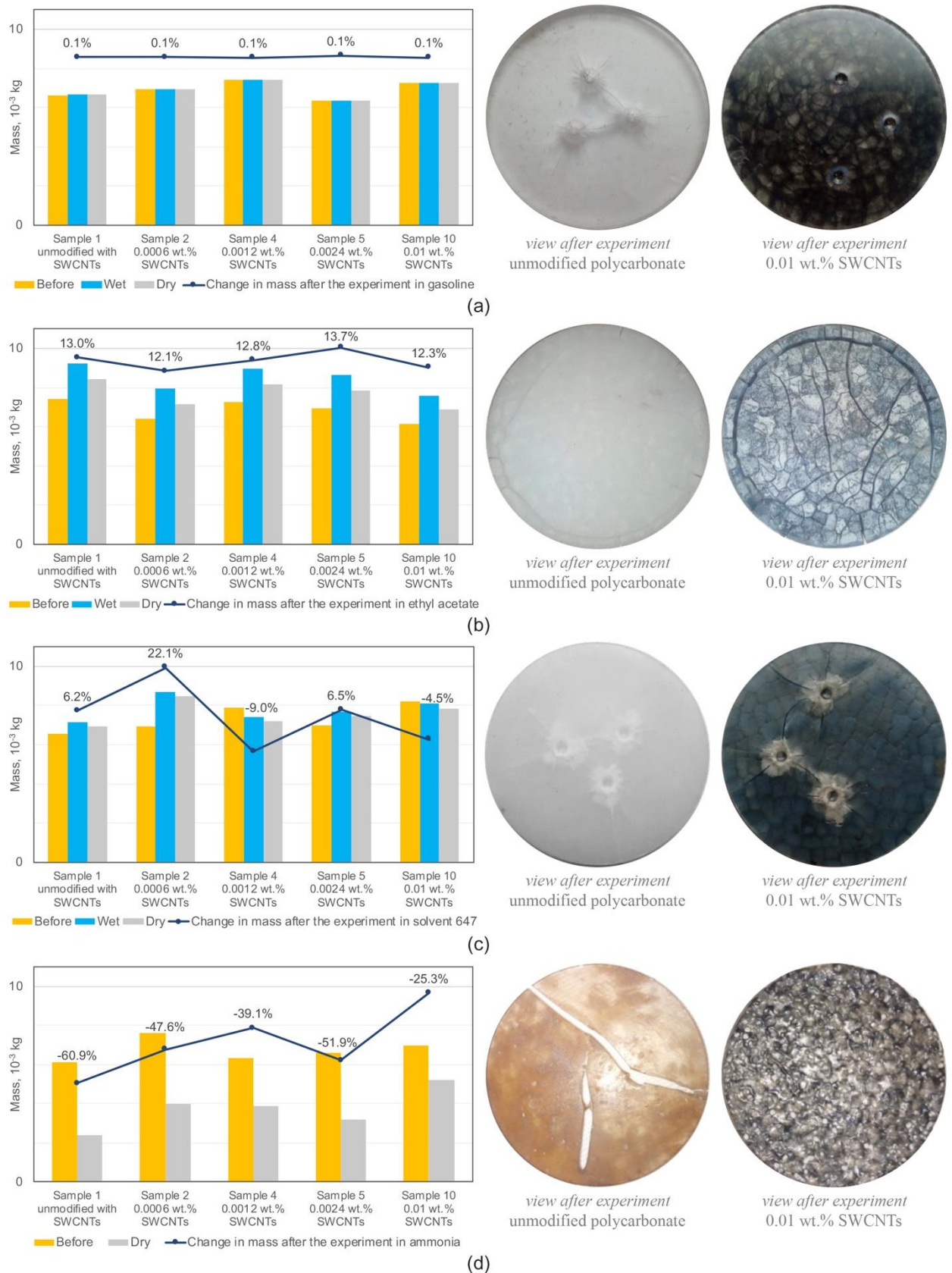


Figure 7. Results of exposure of polycarbonate samples modified with SWCNTs to gasoline (a), ethyl acetate (b), solvent 647 (c) and ammonia (d). First column demonstrates charts of mass change depending on the degree of modification with SWCNTs and aggressive environment before exposure, immediately after removing (wet) and after drying. Second and third columns show samples removed from the aggressive liquids after experiments.



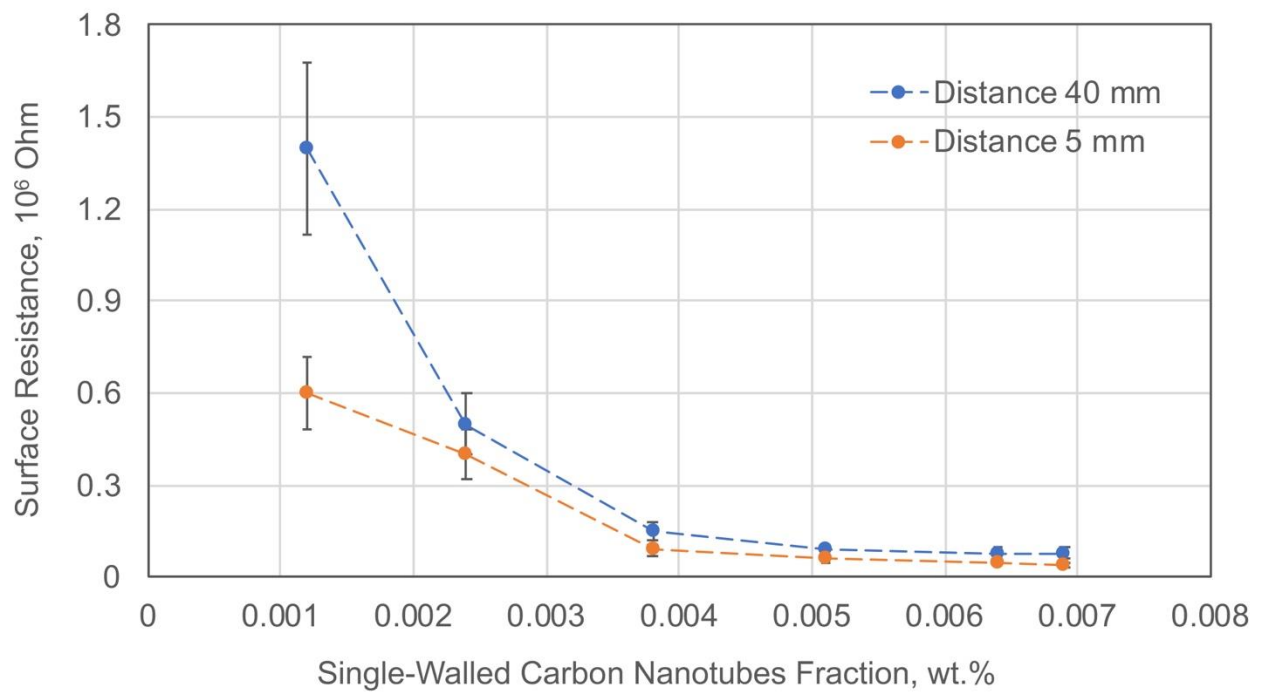


Figure 8. Surface resistance of polycarbonate modified with single-walled carbon nanotubes.