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ELECTRICALLY CONDUCTIVE COMPOSITE MATERIALS BASED ON POLYVINYLPYRROLIDONE COPOLYMERS WITH COMBINED FILLERS

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Composite materials based on copolymers of polyvinylpyrrolidone and 2-hydroxyethylmethacrylate with combined fillers consisting of metal powders and graphite were obtained by the method of polymerization filling. The developed materials are characterized by sufficiently high physical and mechanical properties, increased electrical conductivity and water content. It was established that the addition of graphite to metal-filled copolymers increases the sensitivity of the electrical resistance of composites to moisture changes.

Key words: composite hydrogels; copolymers; polyvinylpyrrolidone; 2-hydroxyethylmethacrylate; electrical conductivity.

Introduction

Polymer conductive composite materials are widely used nowadays in various areas of engineering, construction and electronics – for the manufacture of electric heating elements, supercapacitors, shielding and antistatic coatings, conductive adhesives, paints, pastes, air filter elements, in medicine – for stimulating the growth of bone tissues, also as antithrombogenic implants, etc [1–3].

Most polymer materials are dielectrics. One of the methods of obtaining electrical conductivity in polymers is their filling with conductive fillers – nano- and microparticles of metals and alloys, different types of carbon black, graphite, graphite fibers, etc. Modification with metal fillers is one of the common methods of providing electrical conductivity to polymeric materials. To create polymer metal-filled composites, polymer matrices based on thermosets [4], thermoplastics [5], elastomers [6], hydrogels [7] and fillers (metals, including precious ones [4, 8], ferromagnets [9], metal alloys [10], bimetals [11], hybrid [12]) are used.

Hydrogels, which contain metal particles in their structure, represent a new promising class of composite polymer materials [7, 13, 14]. The great interest in metal-filled hydrogels arose due to their combination of the properties from polymer matrix and metal filler [15]. Filled polymer hydrogels, depending on the nature of the metal filler, acquire new unique properties – sensitivity to various types of radiation, temperature, electric and magnetic fields; mechanical toughness; adhesion to various surfaces, especially to the skin or soft tissues; the ability to create microstructure constructions – three-dimensional platforms for cell cultivation (scaffold technology); antimicrobial and antifungal activity [15–18]. Such materials are already used in biotechnology, medicine, microelectronics, optics and optoelectronics, chemical catalysis, sensor devices and other fields [1, 17–20]. Metal-filled hydrogels differ from other metal-filled polymer materials in the ability to change their conductive characteristics depending on the temperature, pH medium, content of moisture or low molecular weight substances, which opens the possibility of their use in the manufacture of various types of sensors [21, 22].

It was established that copolymers based on 2-hydroxyethylmethacrylate (HEMA) and polyvinylpyrrolidone (PVP) [23–25] are promising materials for creating metal-filled polymer composites. The result of preliminary studies are composite hydrogels obtained by polymerization filling with finely dis-

persed metal powders of various nature – Fe, Co, Ni, Pb, Cu, Zn and FeCo, $SmCo_5$ alloys with a particle size of 5-60μm [26–28]. The developed materials are distinguished by synergism of properties, as they combine the characteristics of both hydrogels and metals. The sorption capacity of these materials in combination with electrical conductivity provides a perspective and expansion of the directions of their use. Due to the ability to change its electrical resistance depending on the moisture, the developed hydrogel composites can be used as detecting elements of moisture meters to determine the moisture content of various materials [29]. However, as studies have shown, the use of metal-filled hydrogels as moisture detectors requires solving a number of problems. Foremost, increase the sensitivity of the material to moisture changes. The material should change its resistance within a wide range in the shortest possible time. This problem can be solved by increasing the electrical conductivity of the composite. At the same time, the change in the resistance of the composite material during moisture absorption should be noticeable in the required interval of moisture changes.

In this view, an actual problem in the direction of creating metal-filled hydrogel moisture sensors based on pHEMA-gr-PVP copolymers is the development of new ways to increase their electrical conductivity, in particular, the sensitivity of electrical conductivity to moisture changes. One of the ways to solve this problem in the case of polymerization filling of hydrogels with metal powders is to introduce a relatively large amount of metal filler into the initial composition. However, this leads to an increase of the composite mass and decrease of its strength with elastic characteristics. In addition, due to the sedimentation phenomenon, there are difficulties in obtaining materials with a uniform distribution of filler particles in the sample volume. The method of using combined fillers of different nature is considered promising - combining metal powders with electrically conductive fillers with less weight and a more effective surface, for example, graphite.

The purpose of this work was to found the possibility of obtaining electrically conductive composite copolymers of polyvinylpyrrolidone with 2-hydroxyethylmethacrylate with combined fillers based on microparticles of metals and graphite.

Materials and research methods

The following substances were used: 2-hydroxyethylmethacrylate (Sigma Chemical Co), which was purified and distilled in vacuum (residual pressure = 130 N/m^2, T_B = 351 K); polyvinylpyrrolidone (AppliChem GmbH) of high purity with MM 28000 was dried at 338 K in vacuum for 2–3 hours before use; iron (II) sulfate was used of p. a. grades.

Obtaining of PVP with HEMA and composite hydrogel materials based on them were carried out according to the method described in [28]. For the preparation of the reaction composition, considering its processability, HEMA in the amount of 70 ÷ 80 mass parts and PVP in the amount of 20 ÷ 30 mass parts were used. The lower limit of the PVP content is driven by the fact that its lower content significantly increases the hardening time of compositions, especially metal-filled compositions, which were hardened without additional polymerization initiators. The upper limit is due to technological complications – higher content of PVP increases the duration of its dissolution in HEMA, increases the viscosity of the composition, which becomes difficult to dose. Fine powders of metals (Zn, Cu) with a particle size of 10÷50 μm and graphite AUSS 5420-74 were used as fillers for research.

The conductivity characteristics of FMF/ pHEMA-gr-PVP composites were estimated by the specific volume resistivity (ρ_v, Ohm×m) [28]. Water content (W, wt. %) was investigated by weight method of difference between the mass of dry and swollen samples, the swelling coefficient (k) – by changing the size of dry and swollen samples [28]. Strain-resilience characteristics: the hardness number (H, MPa) and the elasticity index (E, %) were determined on the hardness meter TShR-320 by measuring the difference between the depth of immersion of the indenter in the swollen sample under the action and after removal of the load [30].

Results and discussion

Previous studies [28, 29] established that composites filled with copper powder have the highest electrical conductivity among the used metal fillers (Fe, Co, Ni, Pb, Cu, Zn, FeCo, $SmCo_5$). Hydrogels that contain 50 wt. % copper are already conductors ($\rho_V < 10^3$ Ohm×m) and are sensitive to changes in moisture up to 10 %. For measuring moisture above 10 %, the sensitivity of the material decreases – with a

change in moisture, the resistance changes slightly. An increase in electrical conductivity due to the increase in the metal content inside the composite is undesirable, since, as shown by the obtained results [28, 29; 31], when the copper content is increased by more than 50 %, the electrical conductivity of the samples practically does not change.

To improve the conductive properties, an additional filler was added to the composition – graphite brand GLS-3 GOST 5420-74, which is widely used in the field of plastics processing to create conductive composite polymer materials. Studies have shown that the addition of even small amounts of graphite significantly improves the electrical conductivity of copolymers (Fig. 1). In this case, the conductivity depends on the shape of the filler particles. Unlike metal powders, graphite has an effective active specific surface, which is 2–3 m^2/g [32].

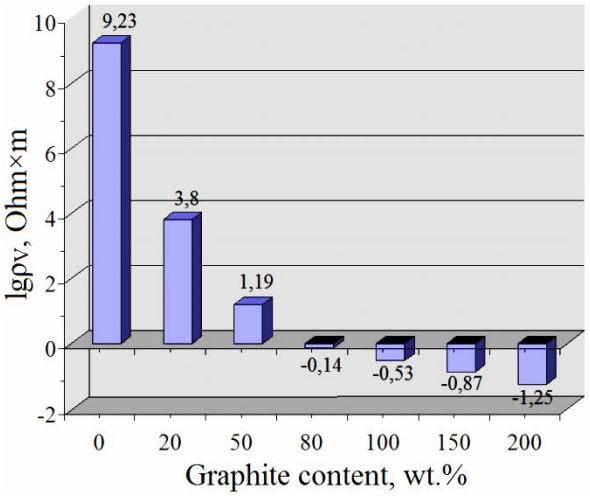

Fig. 1. Dependence of the specific resistance ρ_V of graphite-filled hydrogels on the filler content (HEMA:PVP = 70:30 mass parts, [Zn] = 10 wt. %)

The particles of metal fillers have a smooth rounded shape, therefore, during the polymerization process, the composition flows, fills the free volume around the filler particles, separating them from each other with a film of binder, which reduces the number of contacts. At the same time, due to the loose and irregular shape of the graphite particles, the number of contacts in the binder medium is sufficient to ensure electrical conductivity. The nature of the influence of the graphite presence on the physical and mechanical properties of pHEMA-gr-PVP composites depends on the physical state in which the material stays (Table 1).

By analyzing the obtained results, it can be noted that with an increase of the graphite content in the zinc-filled ([Zn] = 10 wt. %) composite, the surface hardness of the material in the solid state decreases. At the same time, the water content and the swelling coefficient increase. When the material is in a swollen state, with an increase of the graphite amount in the hydrogel, its strength (H, MPa) and elasticity (E, %) characteristics increase as well. With the use of the polymerization filling method in the presence of a solvent followed by drying of composite hydrogels, it is possible to increase the graphite content in the composition to 500 wt. % (Table 2). The electrical conductivity of such materials increases by several orders of magnitude and significantly exceeds the electrical conductivity of composites filled only with a metal filler.

For copolymers that contain graphite, the opposite effect of temperature increase on the nature of the change in electrical specific volume resistance of copolymers relative to metal-filled ones is observed [31]. In this case, the electrical conductivity drops sharply at the initial stage, up to the softening temperature, which equals to T_V = 98–110 ºC for such compositions (an increase in ρ_V takes place) (Fig. 2).

Table 1

Dependence of physical and mechanical properties of block copolymers on graphite content(T = 298K, HEMA:PVP = 80:20 mass parts; [Zn] = 10 wt. %)

No.	Graphite content, wt. %	In a solid state			In a swollen state		
		F, MPa	*k*	*W*, %	*H*, MPa	*P*, %	*E*, %
1	–	275	1.26	46	2.0	18	82
2	5	285	1.28	47	2.2	17	83
3	10	214	1.29	48	2.3	16	84
4	20	180	1.33	51	2.9	12	88

Table 2

Dependence of electrical conductivity of hydrogels obtained in solution on the content of graphite and solvent (HEMA:PVP = 70:30 mass parts; [Zn] = 10 wt. %)

No.	Graphite content, %	Amount of solvent (H_2O), mass parts	ρ_V, Ohm·m		γ_V, Ohm^{-1}· m^{-1}	
			in a swollen state	in a solid state	in a swollen state	in a solid state
1	50	–	–	29.49	–	0.04
2	50	50	3.72	1.23	0.3	0.8
3	50	100	1.00	0.48	1.0	2.1
4	300	150	0.11	0.03	9.4	33.3
5	500	200	0.08	0.05	12.6	20.0

In the temperature range > T_V ρ_V decreases slightly. The specified differences in the change in electrical conductivity of copolymers filled with graphite and metals can be associated with different mechanisms of charge transfer.

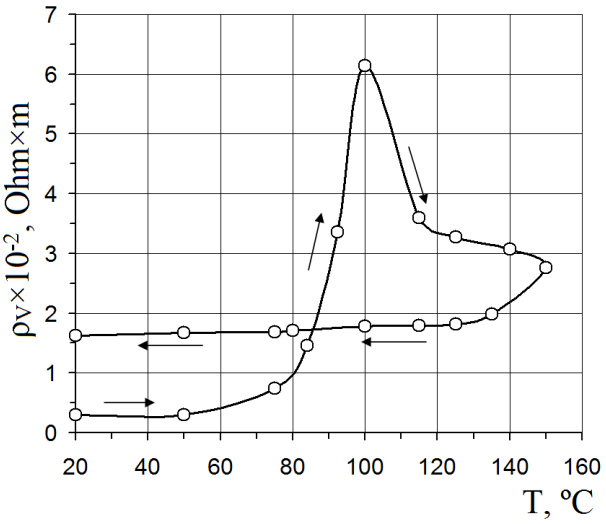

Fig. 2. Effect of temperature on the specific resistivity of the graphite-filled copolymer (HEMA:PVP = 70:30 wt. parts; [Zn] = 10 wt. %; [Graphite] = 50 wt. %)

The dependence $\rho_V = f(t)$ The dependence $\rho_V = f(t)$ for a graphite-filled material is typical for materials in which the transport of charge carriers is carried out by a network of electrically conductive filler particles in contact with each other spread in a dielectric matrix. Since the coefficient of thermal expansion of the polymer medium is several orders of magnitude higher than that of the filler, when the temperature rises, the thermal expansion of the matrix leads to a decrease in the contact area between graphite particles or even to the breaking of areas of a whole cluster into isolated clusters, which is manifested in a significant decrease in the electrical conductivity of the composite. The contact spots on the graphite particles during the heating process are covered with a polymer film, which explains the discrepancy between the heating and cooling curves.

It was established that the presence of predominant amounts of graphite in the composition of metal-filled pHEMA-gr-PVP copolymers affects the change of the electrical conductivity of the composites during swelling in wet media. In contrast to copolymers filled only with metal, for which at the first stages of moisture absorption in small amounts (up to 10 wt. %) an intense decrease in electrical resistance is observed (Fig. 3, curve 1), for a material with a graphite content of 50 wt. %, a sharp drop in electrical conductivity in the range of 1–5 % water content (Fig. 3, curve 2) takes place. Reducing the graphite content in the composite by two times contributes to a significant shortening of the interval between the initial electrical resistance of the dry sample with the resistance extremum that occurs at the initial stage of swelling. During the introduction to the composite of an additional filler – copper in the amount of 50 wt. %, the peak of the increase of electrical resistance on the curve $\rho_V = f(W)$ disappears (Fig. 3, curve 4). At the same time, the bending angle of the curve increases in the interval of water content measurement compared to the copper-filled sample (Fig. 3, curve 1). This indicates a significant increase in the sensitivity of the composite hydrogel with a combined filler to changes in moisture.

By analyzing the obtained results, it can be noted that the use of a combined metal/graphite filler involves the creation of electrically conductive composite hydrogel materials with increased electrical conductivity and sensitivity to changes in moisture, which makes them relevant for use as detectors in moisture measuring devices. One of the

advantages of using the developed materials as conductometric detector is the possibility of studying the kinetics of drying, as well as the ability to register the critical moisture content of materials based on the change in resistance.

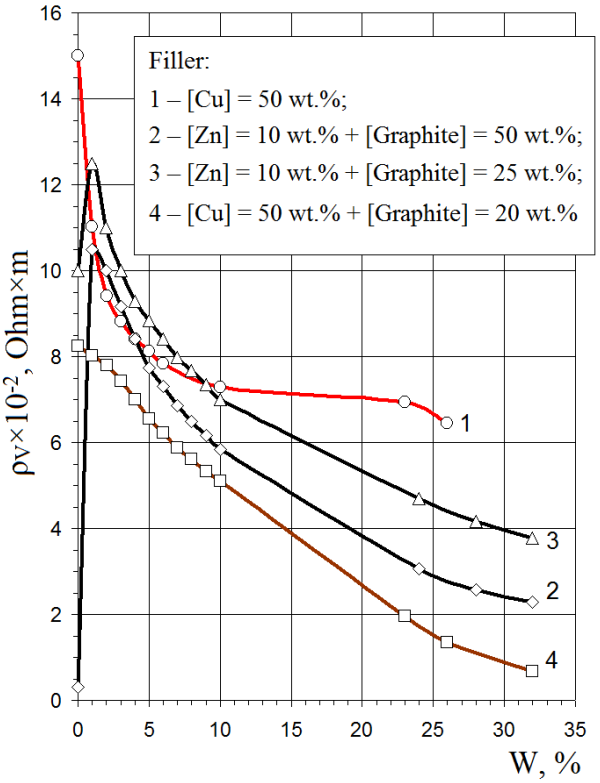

Fig. 3. Dependence of the specific resistance ρ_V of composite copolymers on the water content (HEMA:PVP = 70:30 mass parts; 1, 4 – [$FeSO_4$] = 0,05 wt. %; [PB] = 0,3 wt. %)

The developed material was tested in laboratory conditions for the possibility of its use as a detector for remote control of soil moisture. Structurally, the soil moisture meter is made in such a way that its main part – the moisture detector, is made of composite pHEMA-gr-PVP copolymer, in which there are two isolated electrical contacts connected to the resistance meter. The work of the detector consists in absorbing moisture from the soil with a simultaneous change in electrical conductivity, which is identified by a change in the resistance of the porous material of the detector. The gravimetric method "GOST 5180–84. SOILS. Methods of laboratory determination of physical characteristics." was used as a standard for measurements, according to which the absolute moisture of soil samples was determined. As a result of the experiments, it was established that the developed composite material is suitable for measuring soil moisture by the conductometric method, the average absolute measurement error relative to the data of the gravimetric method is 2.2 %.

Conclusions

As a result of the conducted research, the possibility of obtaining composite materials with increased electrical conductivity based on copolymers of polyvinylpyrrolidone with 2-hydroxyethylmethacrylate, modified with a combined metal/graphite filler was established. It was proven that the nature of the effect of the graphite presence on the physical and mechanical properties of pHEMA-gr-PVP composites depends on the physical state in which the material stays. As the graphite content increases, the surface hardness of the composite in the dry state decreases, while the strength and elastic characteristics of the swollen materials increase. It was established that composites containing small graphite impurities are characterized by significantly higher electrical conductivity and water content than metal-filled hydrogels in commensurate concentrations. The increased electrical conductivity, sorption capacity and sensitivity of the resistance changes of the obtained materials to changes in moisture make them promising for use as elements for devices, which measure the moisture of various materials by the conductometric method.

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ЕЛЕКТРОПРОВІДНІ КОМПОЗИЦІЙНІ МАТЕРІАЛИ НА ОСНОВІ КОПОЛІМЕРІВ ПОЛІВІНІЛПІРОЛІДОНУ З КОМБІНОВАНИМИ НАПОВНЮВАЧАМИ

Методом полімеризаційного наповнення одержані композиційні матеріали на основі кополімерів полівінілпіролідону з 2-гідроксіетилметакрилатом із комбінованими наповнювачами, які складаються з порошків металів та графіту. Розроблені матеріали характеризуються достатньо високими фізико-механічними властивостями, підвищеною електропровідністю та водовмістом. Встановлено, що додавання графіту до металонаповнених кополімерів підвищує чутливість електричного опору композитів до зміни вологи.

Ключові слова: композиційні гідрогелі; кополімери; полівінілпіролідон; 2-гідроксіетилметакрилат; електропровідність.