Original Article

Improvement of the Properties of Composite Materials Due to the Inclusion of Metal Phosphates in Polyethylene, Polypropylene and Polyamide-66

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Abstract - Despite the small share of nanoparticles in the chemical industry, the demand for modifiers is growing every day. This is due to their high physical, mechanical and operational properties, resistance to aggressive media, and ability to be used over a wide temperature range. According to the results of this study, introducing metal phosphates into the polymer improves the physical and mechanical properties of polyolefins. The physico-mechanical properties of the obtained polymer composite materials were improved up to 1-2 times compared to the initial polyethylene, polypropylene and polyamide-66 samples without filling. As a result of the addition of fillers, the elasticity, impact and fire resistance of polymers increased up to 2 times. Therefore, it is more important to produce polymer materials based on metal-containing compounds and apply them in practice. Including metal phosphates among the fillers increases the fire resistance of the composites. The rate of polymer liquefaction was determined by SOK(PTR) method. Also, IR, thermal analysis, TG and DTA, DSK, SEM and EDS analysis, X-ray diffraction, burning time and oxygen index were determined. As a result of the introduction of fillers, polymer molecules are bound with metal binders, and the elasticity, impact and fire resistance, and heat resistance of polymers have increased up to 2 times. The combustion time was reduced from 239 C to 125 C, and an increase in the oxygen index from 19% to 26% was achieved.

Keywords - Polyethene, Polypropylene, Polyamide-66, Ammonium polyphosphate, Metal phosphates, Zinc oxide, Loaddeformation temperature.

1. Introduction

Currently, there is a growing interest in the use of industrial polymers (PE, PP, PA) and polymer composite materials based on various nanoparticles by the general public. Among the fillers, minerals such as metal oxides have a special place [1, 2]. The low cost of these minerals in our country and the breadth of their physicochemical, mechanical and other properties further increase the possibility of their use in the polymer industry. Foreign and domestic scientists J.W. Gilman, Ch. A. Wilkie, A.B. Morgan, S.Bourbigot, M.L.Bras, Y.Mingshu and F.Wang, academicians A.A.Berlin, S.Sh.Rashidova worked on the creation of polymer composite materials. Special mention should be made of the research work carried out in recent years by SS Negmatov, NR Ashurov, AT Djalilov and young scientists P.J. Tojiev, N.Kh. Bozorova, D. Kenjaev and others [3, 4]. They have developed a technology for obtaining anti-corrosion coatings and similar materials by developing suitable additives for modifying polymers and studying the resulting product's wide range of properties. In our research, we envisage the development of a technology for obtaining flammable heat-resistant materials by modifying ammonium polyphosphate and metal phosphates into additives. As a result, using environmentally friendly composites using local raw materials in all areas of construction and production will further increase efficiency. It should be noted that ammonium polyphosphatemontmorillonite nanoparticles used in refractory intumescent polypropylene were obtained. The shape and morphology of crystalline nanoparticles were studied by Xray diffraction, Fourier transform infrared spectroscopy, scanning and transmission microscopy, and the properties of PP by oxygen index determination, Thermogravimetric analysis (TGA). The composition of the ammonium polyphosphate-montmorillonite nanoparticle was found to increase the fire resistance of PP relative to ammonium polyphosphate and its mixture with montmorillonite microparticles [5, 6].

2. Experimental Part

Scientific substantiation of the following solutions for the production of automotive and household plastic parts based on new composite materials: selection of various reactive modifiers for micro and nano-sized mineral modifiers added to polymers; modification of polymers using dispersed particles allows for significant change in the properties of the polymer by adding various additives, to regulate the technological and operational properties. In particular, for the purposeful improvement of the physicochemical properties of PE and PP, modification methods consisting of creating new composite materials are now widely used.

The choice of modifier, the composition of which depends on the nature of the polymer, is one of the most convenient and inexpensive ways to obtain a polymeric material with different properties [7, 8]-infrared spectroscopy (IR). The IR spectra of the synthesized compounds were studied on IRTracer-100 spectrometers. Samples were obtained in the form of viscous-liquid oligomer solutions in known solvents. The set of infrared spectrometers "IRTracer-100" (SHIMADZU CORP., Japan, 2017) consists of a total internal mirror (NPVO) MIRacle-10 and a diamond / ZnSe prism (spectral range by wavelength - $4000 \div 450$ cm-1 Fure spectrometer "IRTracer" The dimensions of 100» are as follows: signal and noise sensitivity - 60,000: 1, scanning speed - 20 per second/ Thermal properties (thermal properties) were studied by the method of differential scanning colonometer (DSK) [9, 10]. Scanned Electron Microscope (SEM): The morphology of samples filled and treated with metal phosphates was studied using electron Hitachi scanning microscopy. Thermoanalytical studies of the samples were performed on a DSK Netzsch Simultaneous Analyzer STA 409 PG (Germany) with a K-type thermocouple (Low RG Silver) ia al.miniy crucible. All measurements were performed in an inert nitrogen atmosphere with a nitrogen flow rate of 501/min. The temperature range of the measurements was 25-370 ° C, and the heating rate was 5 K / min. The sample size for one measurement is 5-10 mg. The measuring system is calibrated with a standard set of substances KNO₃, In, Bi, Sn, and Zn [11, 12].

3. Results and Discussion

In the laboratory, polymers have been treated with organometallic compounds as fillers. Using organometallic compounds as fillers, add to the bottom of a flat test tube 5% 0,25 g, ammonium polyphosphate 3,3 g, in proportion., with the addition of Al_2O_3 and 1.4 grams of a mixture based on PE granules heated in an electric furnace to a temperature of 130-250°C. The mixture was vigorously stirred while heating to a temperature of 160-250°C.

As a result, after 3-4 hours, a viscous mass in the form of cookies was formed. The resulting powdery viscous mass was placed in a porcelain mortar and dried in an oven at 80-90 0 C for a day. The dried sample was ground in a mortar, and low molecular weight compounds were washed several times, first with a 5% NaOH solution and then with distilled water. The prepared mass was dried in an oven at 80°C for 24 hours to remove excess water. The selected polymer granules and the necessary additives and modifiers were weighed on an electronic balance with an accuracy of 0.1 g. All reagents were placed in a special mixer and mixed at room temperature for 10 min at 620 rpm. After that, granules of polymer composites were obtained by mixing the polymer in a granulator within the liquidation temperature. The composites were then placed into an extruder [13].

The inclusion of metal phosphates in the composition of the polymer leads to an increase in the concentration of modifiers and an increase in yield stress (SOC).

In polymer composite materials (PKM), with an increase in the content of phosphate salts, the fluidity of the composition accelerates (Fig 1), and it was observed that this process continues until the concentration of the modifier reaches 3%.



Fig. 1 A graph of the dependence of the mass fraction of nickel oxide on the flow rate of polyethylene

A similar phenomenon is observed when ammonium polyphosphate salts are added to metals such as aluminum, iron, and zinc. In these polymer composite materials (PKM), the fluidity of the composition increases as the amount of ammonium salts increases. As can be seen from the graph, the flowability of polymer composite materials increased with increasing nickel metal concentration [14,15]. 1, 2, 3, 4 and 5% of different metal phosphates, it can be seen that SOK(PTR) of polymers modified by 3-5% higher than unmodified polymers (Fig. 1) and this pattern is the same increase for all metal phosphate salts observed. The mechanism mentioned above is the same for other metal salts.

The obtained results show that as the concentration of metal residues among polymer macromolecules increases, the fluidity of the obtained composites is determined to be higher. Adding metal phosphates to the polymer increased the thermal stability of the composite and shifted the decomposition onset temperature to a higher temperature range. The reinforcement of polyethylene metal phosphates leads to a slight increase in the temperature of the onset of crystallization and a slight increase in the temperature of the onset of liquefaction of the composites. Also, the modification of metal phosphates leads to a decrease in the degree of crystallinity of polyethylene.

As a result, the phase structure of polyethylene can be explained by strong adhesion between matrix particles and metal compounds, which leads to the formation of new adsorption layers at the phase boundary and the junctions of amorphous components. Suppose the interaction energy at the interface between the matrix and the modifier (dispersion and van der Waals forces) increases the interaction energy of polyethylene carbon macromolecules and leads to the formation of a crystalline phase. In that case, crystalline claws may disappear, and the degree of crystallization may decrease [16, 17].



Fig. 2 Dependence of SOK (PTR) on modifier concentration

Table 1. Kneological properties of FE mounted with metal phosphates					
	ZnO	NiO	Со	Al ₂ O ₃	
Composite composition	Flow Rate Indicator (PTR) gr/10 min				
PE+1%	8,1	8,5	8,6	9	
PE+1,5%	8,9	8,8	8,99	9,2	
PE+2%	9,2	9,1	9,3	9,7	
PE+2,5%	10,2	9,7	9,6	10,2	
PE+3%	11,1	10,2	10,1	10,8	
PE+3,5%	11,3	10,5	10,4	11,2	
PE+4%	12	11	11,1	11,5	
PE+4,5%	12,3	11,2	11,5	12,2	
PE+5%	12,9	12,5	12,8	13,5	

Table 1 Decological properties of DF modified with motall phosphates



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Table 2. Differential scanning calorimetry data for Polyethylene-Based composites modified with 3% metal phosphate							
Composite	T liquid, Operating	T Pippl,	ΔH , enthalpy,	T crist. start.,	T pikkr,	Cristal Level	
composition	systems	oC	J/g	oC	oC	%	
PE	125	116	152	134	139	63	
PE+3% Al ₂ O ₃	126	109	157	130	130	68	
PE+3% NiO	130	122	152	128	129	65	
PE+3% Co	127	123	153	132	135	71	
PE+3% Fe ₂ O ₃	129	103	152	120	132	66	
PE+3% ZnO	130	113	156	132	141	69	

Figure 3 shows DSK thermograms of melting (sections from 109 to 147°C) and crystallization (sections from 120 to 430°C) of the initial PE, Al₂O₃ polymers and their mixtures of 20 wt—metal % polar polymer.

For all composites, the Δ H liquid value is greater than the initial value of pure PE (152.4 J/g) and changes with increasing content of the modifier. This fact shows that the degree of crystallization of polyethylene usually does not depend on the degree of the modifier (Table 1). Based on the DSK diagram (Fig. 3), the process of heating polyethylene and nanocomposites is shown. All samples have an endothermic melting point. Table 2 shows the pour points for nanocomposites PE/modifier with a content of 1, 3, and 5%. For composites, the pour point slightly increases with the increasing content of the modifier. The onset temperature of the liquefaction and the width of the liquefaction peak of the nanocomposites indicate the dispersion of the crystals [18, 19].

The analysis of the polymer composite material obtained as a result of the reaction mixture of polypropylene filled with metal phosphates was carried out at the scientific research institute of AKM (Scientific Research Institute of Chemistry and Physics of Polymers), and the bending radius of the needle is 10 nm. Li silicon cantilever, the size of the scanning area was 1-50 microns. It shows the surface's topography and the interphase movement's fluctuation (phase detection), indicating the adhesion of local surfaces to each other and the change of the phase structure. Figure 4 shows the surface of polypropylene modified with aluminum oxide and ammonium polyphosphate (3D view).





(b) Fig. 4 PP/5% Al₂O₃ (a) – Three-dimensional image (b) – Two-dimensional image

As can be seen from the above results, the roughness level of the surface of pure polypropylene is 100 nm, and the roughness level of the polypropylene surface filled with metal phosphates is 210 nm. From the two-dimensional image, it can be observed that there is a spherical 3.75 nm particle of metal phosphates and small spherical particles around it.





(b)



Fig. 5 Image of polypropylene surface filled with metal phosphates in AKM: (a) - a three-dimensional image (3D-view) (b) - phase (c) - two-dimensional view (topography) (d) - size between particles

According to the above, modification of the polymer surface with filler particles leads to an increase in the degree of roughness on its surface. The change of phase movement, as shown in the picture, indicates that there are two phases on the surface of the polymers and that the work is being carried out on metal phosphates of the polymer composite material, which has formed a mechanical mixture with filler particles of polymer macromolecules [20, 21]. The surface effect of modified metal phosphates on polymer composites was investigated using SEM (Fig. 6). However, it can be seen that the formation of a coherent morphology between the metal particle modifier and polymer phases, the absence of large agglomerates that metal particles can form, can be considered as homogeneous mixing with their polymer matrices. (Fig. 6), from which it can be seen that the polymer composite material obtained based on PP/ZnO₂%, in the enlarged state, is distributed unevenly over the surface, and metal particles are evenly distributed throughout the polymer.



Fig. 6 PP/ZnO/(NH₄PO₃)n Micrographs of 5% SEM were enlarged by (a)-200-µm, (b)-250 µm times

Scanning Electron Microscopy (SEM) allows us to see that the morphology of the polymer is similar to other polymers described in the literature. SEM photographs of the polymer at 200,250 times magnification are shown in Fig. 6. As you can see from the images in this photo, there are mostly uneven grains and sharp edges but no tears or holes. This means that polypropylene particles are bonded with metals. This shows that the length of the polymer chains increases with increasing temperature and is proven to determine the packing and structure of the polymer molecule. We can see that the metal-bearing particles have similar morphology to polypropylene[22,23]. Our research was carried out using energy-dispersive spectroscopy (EDX) methods obtained on the "Map_001_wholespectrum" equipment at the Uzbekistan-Japan Youth Innovation Center under Islam Karimov Tashkent State Technical University (TDTU).

According to the conducted analysis, as shown in the table above, as a result of the filling of polypropylene with metals, it was found that the content of S-carbon is 88%, O-Oxygen is 8%, P-phosphorus is 0.8%, Zn-zinc is 2.17% indicates 3 tables.



Fig. 7 Energy-dispersive spectrum of polypropylene filled with metals

N₂	The composition of polypropylene filled with metals	Composition of elements Indicator in %
1	С	88,88%
2	0	8%
3	Р	0,8%
4	Zn	2.17%

Table 3. EDS analysis of metal-filled polypropylene



Fig. 8 TG and DTA PE modified with metal phosphates: 1 – PE + 3% MeF; 2 - original PE - J2210

In TGA and DTA analysis of polyethylene modified based on ammonium polyphosphate compounds of zinc oxide, two endothermic and two exothermic processes occur. The first phase of decomposition of the substances obtained for testing was 28.480 °C, at 10.768% from 310.180 °C to 31.768%. This is due to the formation of nitric oxide during decomposition. The second phase of the process was a loss of 3402 mg from 318°C or a loss of

100365%, the main phase from the decomposition of the metal to 390°C. Nitrogen oxides and compounds are visible. In the DTA analysis, heat absorption was observed at a temperature of 122.22 °C in the first stage of the initial decomposition process of heat and metal oxides. In the second stage, the decomposition of carbonates and nitrates was observed at a temperature of 470.60 °C.

Parameters	Standards	HDPE- J2210	PE+ 3% ZnO	PE+ 3% Al ₂ O ₃	PE + 3% Co	PE + 3% NiO	PE + 3% FeO
Density, g/sm ³	ASTM D 1505	0,96	0,98	0,96	0,99	0,99	0,99
Flexibility module, MPa	ASTM D 1238	1100	1300	1200	1260	1310	1350
Elongation,%	ASTMD 790	300	300	300	300	300	300
Consistency limit, MPa	ASTM D638	22	22	22	25	22,5	22
Izod effect viscosity, according to s / n,+23°C, kJ/m ²	ASTM D638	4	5	4	5,1	4,5	4,7
Cutting after 24 hours	ASTM D648	1,5	1,35	1,35	1,35	1,45	1,50
Combustion rate UL-94, mm	Sample thickness 3,2 mm	45	≤40	≤40	≤40	≤40	≤40

Table 4. Polyethylene-based composition physical and mechanical properties

The process of chemical modification of polymers involves improving their physical, mechanical and chemical properties by introducing new functional groups into the polymer macromolecule through copolymerization. In this study, nanocomposites were obtained by processing polyethene and polypropylene using metal oxides. As can be seen from Table 1, the properties of ammophos metal nanoparticles and polymer-based nanocomposites have changed relative to the original polymers.

The analysis results show that the inclusion of metal particles in the polymer composition improves the physical and mechanical properties of the polymers. Moreover, large-scale measures are being taken to develop a scientific basis for producing multi-component coatings and supplying the domestic market with import-substituting products. The changes in the physicomechanical properties of the polymer are given in Table 4 below [24, 25].

In the analysis of the above results, as a result of filling the polymers with metal phosphates, the metal binders of the polymer molecules increased the physico-mechanical properties of the polymers, in particular, the modulus of elasticity from 1100 MPa to 1350 MPa, and the bending temperature under load increased by 45-50 °C.

In order to study the fire resistance properties of composite materials filled with metal phosphates, the oxygen indicator parameters of the polymer were studied using test experiments.

In order to determine the oxygen index, a 50:25 mm sample was prepared in accordance with GOST 12.1.044-2018 and an experiment was carried out on the available

(Limited Oxygen Index Tester Model: GD –ISO 4589-1) device in the laboratory of the Tashkent Scientific Research Institute of Chemical Technology. 3 out of 50:25 mm sample was taken and placed in the device. The oxygen index was studied to 18% before burning the sample. At each stage of the practical experiment, the oxygen index was made to 1% in the process until the sample burned [26, 27].

In the process, when the oxygen index reached 19.2%, the combustion state of the sample was observed. A 50:25 mm coating sample was found to burn for 3 minutes and 86 seconds at an oxygen index of 19.2%. During the practical experiment, it was proved that the average value of the oxygen index of polymer composite materials filled with metal phosphates is equal to 40.

In particular, with the inclusion of fillers in the composition of PP polymers, the burning time decreased from 239 °C to 125 °C, and the oxygen index increased from 19 to 23%, respectively, compared to the original polypropylene. Also, with the inclusion of fillers in the composition of PE polymers, the burning time decreased from 253 °C to 127 °C, and the oxygen index increased from 17 to 24%, respectively, compared to the original polyethylene. (table 5).

As a result of filling polymers with metals, their burning time slows down by two times compared to the original polymer, and weight loss during combustion in air is relatively reduced. A positive change in the flammability indices of the inclusion of metals in the composition of all polymers confirms the difficulty of igniting the resulting composite materials.

Compositing structure	Burning time s	Mass loss during combustion in air %	Oxygen index, %
PP-J350	239	50	19
PP+1% MeF	125	30	20
PP+3% MeF	124	27	21
PP + 5% MeF	123	25	23
PE- 2210	253	61	17
PE+1% MeF	127	34	21
PE+3% MeF	126	32	23
PE + 5% MeF	125	31	24
PA-66	240	58	25
PA-66+1% MeF	113	28	26
PA-66+ 3% MeF	115	29	27
PA-66 + 5% MeF	116	30	28

 Table 5. Influence of composite materials filled with mineral oxides on heat resistance and flammability



Fig. 9 Flammability concentration (KI indicators) of polyethylene, polypropylene and polyamide-66 (1-4)

A positive change in the flammability indices upon introducing metal phosphates into the composition of all polymers confirms the difficulty of burning the resulting composite materials. As a result of filling polymers with metal phosphates, their combustion time slows down by a factor of two compared to the initial polymer, and the mass loss during combustion in air is relatively reduced.

The compositions of the synthesized additives with polymers were analyzed by X-ray diffraction. The

diffraction patterns were recorded on an XRD-6100 X-ray diffractometer (Shimadzu, Japan) with computer control. CuKa radiation (b-filter, Ni 1.54178; current mode and tube voltage 30 mA and 40 kV, respectively) and constant detector rotation speed (step 0.02 deg.), 4 deg/min (ω /2nd circuit)), and the scanning angle is from 4 to 80oS. Scan Range: 5.0000 - 70.0000 (deg) Scan Mode: Continuous Scan Scan Speed: 2.0000 (deg/min) Sampling Interval: 0.0500 (degree) Set Time: 1.50 (sec).



Fig. 10 X-ray diffraction patterns of the studied composites were obtained by XPA. a) PP/NiO 3%. Appearance in X-ray phase spectra

We can see the result of the diffractogram of PP in the table under the crystalline or amorphous structure of the polymer material obtained by the X-ray spatial analysis device. This allows you to measure the material's size, shape and internal structure.

T/r	2theta- scanning angle	d-Interplanar distance	I-Peak Intensity	FWHM- integral width of reflexes
1	9.38	10.3900	73.94	0.3500
2	14.20	6.2375	707.54	0.3500
3	16.18	5.4583	1000.00	0.3500
4	17.00	5.1950	847.42	0.4500
5	18.65	4.7549	502.74	0.7500
6	19.85	4.1568	165.26	7.0000
7	21.27	4.0602	406.33	0.8500
8	21.95	3.4880	267.43	0.7000
9	25.58	3.1188	80.57	0.6500
10	28.58	2.7981	48.90	1.1500
11	35.51	2.5541	42.53	0.4200
12	37.17	2.4140	297.58	0.2500
13	40.22	2.2153	65.54	1.4000
14	43.41	2.0970	40.52	0.2500
15	46.72	1.9340	46.28	0.3000
16	52.69	1.7311	52.57	0.1500
17	58.42	1.5780	65.54	1.4000
18	64.15	1.4474	46.28	0.3000
19	68.76	1.3680	40.52	0.2500

Table 6. Diffractogram data of PP obtained based on metals

In the experimental study of the size and shape of particles, accurate results can be achieved using. X-ray spatial analysis methods (Debay-Scherrer method) [28,29]. The sizes of coherent scattering regions (CTS) (size of nanocrystals) are determined by the Debye-Scherrer formula:

 $Dp = K \lambda / (B \cos \theta)$

Dp – Average size of crystals (nm) K – Scherrer's constant K varies from 0.75 to 2.09. K = 0.94 for spherical crystals with cubic symmetry

 λ – X-ray wavelength.Cu K α = 1.54178 Å B - FWHM (Full Width at Half Maximum) integral length of reflections in the

diffractometer $\cos\theta$ –cosine angle of X-ray diffraction 38.35% of polypropylene polymer molecules are crystalline, and 61.65% are amorphous.

According to the results of X-ray spatial analysis, it was found that the particle size in all composite materials is in the nanoscale [30, 31].

According to IR spectroscopy analysis, absorption bands characteristic of PE were observed. There is also an absorption band in the region of 1643, 1554 cm⁻¹ of the average intensity, which belongs to the vibrations of the C=O group [32, 33].

Table	Table 7. Results of calculating the size of nanoparticles of polypropylene composite filled with metals according to the Debye-Scherrer formula							
T \ n	2theta- Scanning	FWHM- integral width of	Dp (nm)- average crystallite	Dp (nm)				
1 /1	angle	reflexesB	Size $Dp = K \lambda / (B \cos \theta)$	Average				
1	4.69	0.3500	2.37					
2	7.1	0.3500	3.59					
3	8.09	0.3500	4.09					
4	8.5	0.4500	5.53					
5	9.325	0.7500	10.1					
6	9.925	7.0000	100.5					
7	10.635	0.8500	13.08					
8	10.975	0.7000	11.12					
9	12.79	0.6500	12.03					
10	14.29	1.1500	23.7	18.5				
11	17.755	0.4200	10.7					
12	18.585	0.2500	6.72					
13	20.11	1.4000	40.7					
14	21.705	0.2500	7.8					
15	23.36	0.3000	10.1					
16	20.345	0.1500	4.4					
17	29.21	1.4000	59.1					
18	32.075	0.3000	13.9					
19	34.38	0.2500	12.4					



Fig. 11 PE/ZnO 3%. visibility in the IR spectrum

A strong absorption observed in the 1080-145 cm⁻¹ region is associated with flat deformation vibrations of methyl groups. Intense absorption lines of metals appear at 667 and 597 cm⁻¹.

The IR spectrum of the composite material obtained in the presence of zinc oxide shows that the presence of S and N (hydrogen) bonds is observed in the region of 2915 and 2848 cm⁻¹. CH₂ and CH₃ groups are observed in the region of absorption frequencies of 1400, 1471 cm⁻¹. Absorption at the frequency of 600-719 cm⁻¹ confirms the characteristics of metals.

4. Conclusion

In conclusion, it should be noted that introducing metal phosphates into the polymer improves the physical and mechanical properties of polyolefins. As a result of joining Rolimer molecules with metal binders, the heat resistance of

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polymers increased by 2 times. Burn time increased from 239 °C to 125 °C, and the oxygen index increased from 19% to 26%.

At the same time, as a result of filling polymers with metal phosphates, the modulus of elasticity increased from 1100 MPa to 1350 MPa, and the bending temperature under load increased by 45-50 °C. The rheological properties of the polymers, i.e., the flow rate indicator (PTR), increased from 1% to 5% in g/10 min. to 12.9 min. These results show that with increasing concentrations of metal residues between polymer macromolecules, the fluidity of the obtained composites was proved to be higher.

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