

PACS numbers: 61.41.+e, 62.23.Pq, 81.05.Lg, 82.35.Np, 83.60.-a, 83.80.Wx, 83.85.Jn

Regulators of Formation and Properties of Nanofilled Polypropylene Threads

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The influence of the content of silica nanoparticles and the combined substances, namely, aluminium oxide/silica and zinc oxide/silica, on the rheological properties of the polypropylene (PP) melt and the characteristics of modified monothreads is studied. As established, the effective viscosity of PP/nanofiller compositions increases in the entire concentration range studied (0.1–3.0 wt.%) and depends on the chemical nature of the additive. In the presence of silica, the viscosity of melts increases by 20%; when mixed ZnO/SiO₂ and Al₂O₃/SiO₂ oxides are added, it increases by 30% and 50%, respectively. The elasticity of all the investigated systems decreases with increasing content of additives, judging by the indicators of jet swelling. The ability to process nanofilled compositions is improved, as evidenced by an increase in the maximum deformation of the melt jet in the longitudinal tensile field. The presence of silica nanoparticles and mixed oxides in the structure of PP monothreads improves their operational characteristics: the breaking strength and modulus of elasticity increase by 1.3–1.5 times, the percentage of strength retention in the loop and knot increases, and shrinkage decreases. Modified threads, like a polypropylene, are monolithic, have a smooth surface and are uniform in diameter along their length. Nanofillers containing a metal-oxide phase on the surface are more effective compared to the original silica.

Досліджено вплив вмісту наночастинок кремнезему та комбінованих речовин оксид Алюмінію/кремнезем і оксид Цинку/кремнезем на реологічні властивості розтопу поліпропілену (ПП) та характеристики модифікованих монониток. Встановлено, що ефективна в'язкість композицій ПП/нанонаповнювач зростає в усьому дослідженому діапазоні концентрацій 0,1–3,0 мас.% і залежить від хемічної природи добавки. В присутності кремнезему в'язкість розтопів підвищується на 20%, а з додаванням змішаних оксидів ZnO/SiO₂ й Al₂O₃/SiO₂ — на 30% і 50% відповідно. Еластичність всіх досліджених систем зменшується з ростом вмісту добавок, судячи з показників розбухання струменів. Здат-

ність до переробки нанонаповнених композицій поліпшується, про що свідчить збільшення величин максимальної деформації струменя розтопу в поздовжньому розтягувальному полі. Наявність наночастинок кремнезему та змішаних оксидів у структурі ПП-монониток забезпечує поліпшення їхніх експлуатаційних характеристик: зростають у 1,3–1,5 рази міцність за розриву та модуль еластичності, підвищується відсоток збереження міцності у петлі та вузлі, зменшується усадка. Модифіковані нитки, як і поліпропіленові, є монолітними, мають гладеньку поверхню та рівномірні за діаметрами по довжині. Нанонаповнювачі, що містять металоксидну фазу на поверхні, є більш ефективними в порівнянні з вихідним кремнеземом.

Key words: polypropylene, nanoadditives, silica, viscosity, degree of deformation, monothreads, operational properties.

Ключові слова: поліпропілен, нанодобавки, кремнезем, в'язкість, ступінь деформації, мононитки, експлуатаційні властивості.

(Received 27 September, 2023; in revised form, 28 September, 2023)

1. INTRODUCTION

Fibrous materials and products from them have always played an important role in people's lives. Until the beginning of the 20th century, the raw material for them was natural fibres. The deficiency of natural fibres and the growing requirements for the quality and functional characteristics of fibrous materials led to the creation of an alternative source of raw materials—artificial and synthetic fibres. Today, the possibilities of giving synthetic fibrous materials the desired characteristics are practically unlimited thanks to a wide range of methods of their modification. One of the most effective of them is the formation of composite fibres and threads by introducing various types of additives into their structure, especially substances in the nanostate. Natural or specially synthesized substances are used as nanofillers. They are chosen taking into account the possibility of providing products with predetermined characteristics, cost, the possibility of secondary processing, impact on biodegradability, *etc.* Layered aluminosilicates, silicas, carbon derivatives, nanoparticles (NP) of metals, their oxides, *etc.* satisfy a significant number of requirements for nanoadditives. The presence of nanoadditives in the structure of fibrous materials gives them new, often unique properties. Natural or modified clays provide composites with a sharp improvement in strength and modulus of elasticity, heat and fire resistance, and the ability to be dyed with various dyes [1, 2]. Carbon nanotubes (CNT) provide the effect of reinforcement and shielding from electromagnetic

and microwave radiation, provide dielectric, anti-corrosion, biosensor properties, *etc.* [3–5]. Materials with high antimicrobial, thermal, electromagnetic, sorption, antiallergic, photocatalytic and antistatic properties combined with improved operational characteristics and self-cleaning ability are created from synthetic threads filled with NP of metals or metal oxides [6–12]. Magnetically sensitive nanofilled biopolymer fibres are multifunctional, which allows them to be used in a wide variety of fields [7]. A wide range of shielding materials from electromagnetic radiation is obtained by introducing magnetic nanoparticles (iron oxide, cobalt, platinum) [10, 11]. The properties of nanofilled polymer composites, including threads, are also significantly influenced by other factors: additive content, the degree of interaction between the polymer and NP in the transition layer, surface modification of nanoparticles, production conditions, *etc.* Polyvinyl alcohol fibres with a high degree of CNT orientation were obtained using the method of layer-by-layer planting, which made it possible to increase their strength by 25 times and provide electrical conductivity equal to that of copper [5]. Synthesis of Ag nanoparticles by thermal decomposition of silver salt in the process of forming polylactide threads contributes to the formation of NP uniform in size and their uniform distribution, which provides antimicrobial action already at content of 0.01 wt.% [12].

Combined substances are more effective and allow expanding the spectrum of functional characteristics of modified products. Polyurethane nanofibrous material filled with zinc oxide NP exhibits significant antibacterial activity against a number of microorganisms, in particular *E. coli*, and the introduction of bimetallic ZnO/Ag nanoparticles ensures their complete inhibition [13]. Polyvinyl alcohol nanofibers containing Ag/TiO₂ nanoparticles exhibit antimicrobial, photocatalytic, and dirt-repellent effects [14]. Of great interest are bifunctional substances, in which NP of metals or metal oxides are deposited on the surface of mineral sorbents (montmorillonite, diatomite, zeolite, silica) [15–23]. Due to the combination of properties of various components, and often their synergistic action, materials containing such fillers have a number of advantages. They simultaneously exhibit a complex of various characteristics: heat and fire resistance, high sorption and antitoxic properties, photocatalytic activity, which allows purifying air and wastewater from many pharmaceuticals [16–19]. They are characterized by a prolonged bactericidal effect, which is regulated by the rate of release of metal nanoparticles/metal oxides from the sorbent-base. This makes it possible to achieve high antimicrobial activity of materials with a minimum amount of the drug and, due to this, to reduce their cost and ecological burden on the environment. The effectiveness of the

modification of fibrous products with such nanofillers has also been established in a number of our works [20–23]. Polypropylene microfibers containing silver/silica or silver/alumina nanoadditives in their structure have a bactericidal effect on a number of strains of microorganisms and fungi. At the same time, their mechanical indicators, dimensional stability, specific surface area and hygroscopicity are increased [21, 22]. The injection of the Ag/SiO₂ combined additive into PP monothreads increases their strength and elasticity and provides antimicrobial activity. Modified threads have better manipulation characteristics and biological compatibility with the tissues of a living organism, which allows them to be used as surgical suture material and raw material for the manufacture of mesh implants [23].

The purpose of the work is to study the possibility of obtaining polypropylene threads with improved properties by forming them from a melt filled with nanoparticles of original silica or modified with metal oxides.

2. OBJECTS AND METHODS OF RESEARCH

For research was used polypropylene of brand Tatren HG 1007 with melt flow index 10 g/10 min (230°C/2.16 kg, ISO 1133-1) and a melting point of 169°C. Pyrogenic silica and combined additives, in which nanoparticles of zinc and aluminium oxides are applied to the surface of silica NP, were chosen as nanofillers. A300 brand silica with a specific surface area (S_{BET}) of 320 m²/g produced by the Kalush Experimental Plant. Mixed oxides ZnO/SiO₂ and Al₂O₃/SiO₂ were synthesized by the chemical gas-phase deposition method at the O. O. Chuiko Institute of Surface Chemistry of the National Academy of Sciences of Ukraine. The content of zinc and aluminium oxides in the combined additives was of 161.9 and 164.0 µg/m², respectively, and the specific surface area was of 307 and 293 m²/g. The concentration of nanoadditives (C) in the PP melt was of 0.1–3.0 wt.%. The ingredients were mixed in the melt on a worm-disc extruder. The effective viscosity (η) of melts was studied using a capillary microviscometer in the range of shear stresses (τ) (0.1–5.7)·10⁴ Pa at a temperature of 190°C. Processing of the results of viscometry was carried out using generally accepted methods for constructing true flow curves. The flow regime (n) was determined by the tangent of the angle of inclination of the tangent at a given point of the flow curve to the abscissa axis. Calculations were performed using a specially developed program [24]. The elasticity of the melts was estimated by the values of the coefficient of expansion (swelling) of the jet (B) at the exit from the forming hole according to the method [25]. The coefficient of jet expansion was

calculated as the ratio of the diameter of the annealed extrudate, obtained under the conditions of free flow of the melt from the forming hole, to the diameter of the die. The ability of the melt to form fibres was determined by the value of the maximum degree of extraction (F_{\max}), which was estimated as the ratio of the speed of receiving the melt jet to the linear speed of the melt in the die. Composite monothreads were formed on a laboratory bench using a die with a diameter of 780 μm with a degree of extraction of 10. Thermal orientation stretching of monothreads was performed at a temperature of 150°C with the maximum multiplicity for each sample. Multiplicity of extraction was determined as the ratio of the speed of reception to the speed of feeding threads. The study of the mechanical properties of monofilaments, their shrinkage, capillarity and hygroscopicity was carried out according to standard methods. Tensile strength, loop and knot strength, modulus of elasticity and tensile elongation were determined using a PM-3 tensile machine.

3. RESULTS AND DISCUSSION

3.1. Rheological Properties and Ability to Process of Nanofilled Polypropylene Melts

The obtained experimental results regarding the influence of the content and chemical nature of nanofillers on the rheological properties of the polypropylene melt showed that all investigated additives significantly affect its viscoelastic characteristics. The addition of silica nanoparticles and mixed oxides to the PP melt helps to increase the viscosity in the entire studied range of concentrations and shear stresses (Figs. 1, 2). At this, the growth of η is more intensive with the content of nanoadditives of 0.1–1.0 wt.%. With a further increase in their concentration, the increase in viscosity slows down.

The increase of viscosity at a constant shear rate for filled polymer melts is because the solid particles do not deform during the flow, and as their content increases, the flow resistance of the melt increases. As can be seen in Fig. 1, the effect of solid filling for mixed oxides is higher compared to the original silica. This may be the result of the fact that the thickening of the composition is also affected by the degree of interaction of the particles between themselves and the dispersion medium. The formation of a structural mesh in the melt helps to increase its viscosity. The maximum increase in η of the PP melt upon addition of $\text{Al}_2\text{O}_3/\text{SiO}_2$ nanoparticles is the result of their higher structuring ability.

Figure 2 shows the dependence $\lg\eta = f(\lg\tau)$ for PP melt with different content of aluminium oxide/silica nanoadditive.

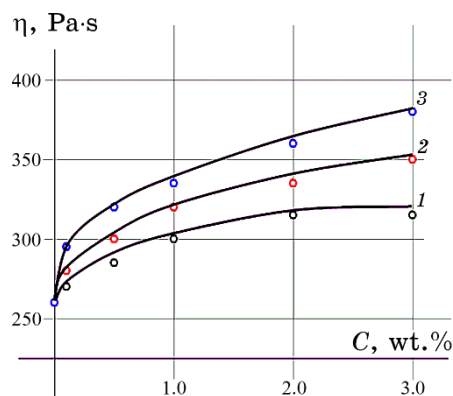


Fig. 1. Dependence of the effective viscosity of the polypropylene melt on the content and chemical nature of the nanofiller: 1— SiO_2 ; 2— ZnO/SiO_2 ; 3— $\text{Al}_2\text{O}_3/\text{SiO}_2$.

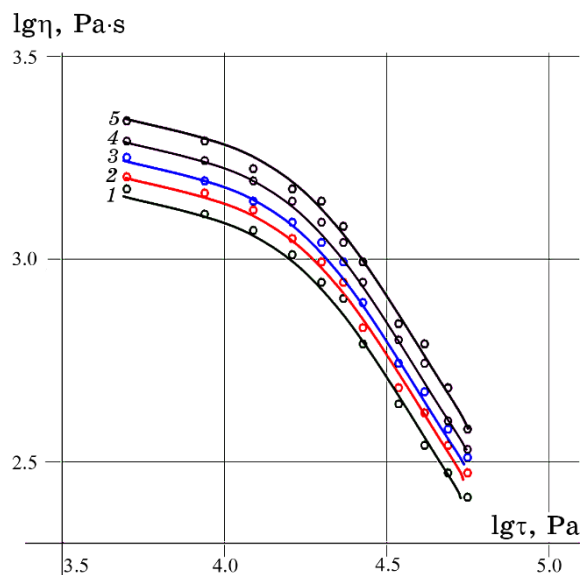


Fig. 2. Dependence of the viscosity of the polypropylene melt under different shear stresses on the content of the $\text{Al}_2\text{O}_3/\text{SiO}_2$ additive, wt.%: 1—0.0; 2—0.1; 3—0.5; 4—1.0; 5—3.0.

It can be seen that at a constant shear stress, the viscosity of the system increases with an increase in the concentration of the filler. At this, in the studied range of τ , the slope of the curves remains practically constant for all nanofilled melts. This testifies to the independence of the flow pattern of the PP melt filled with mixed

oxide NP from its concentration. For systems containing SiO_2 or ZnO/SiO_2 nanoparticles, the ‘viscosity–shear stress’ dependence curves have a similar form.

The flow regime of all investigated nanofilled melts, as well as the original PP, is non-Newtonian and is described by a power law. The index n , which characterizes the degree of deviation from the Newtonian flow, is 2.0–2.1 for all systems regardless of the chemical nature of the additives and their content.

The elasticity of the modified polypropylene melt decreases with the introduction of nanoparticles of the original or silicon metal oxides modified already in the amount of 0.1 wt.%, which is evidenced by a decrease in the equilibrium swelling of the extrudate. At a concentration of additives of 3.0 wt.%, the values of B are reduced by $\cong 20\%$ for all investigated nanofillers. During the processing polymer dispersions, it is generally accepted that fillers improve the technological properties of polymers by reducing the transverse dimensions of the jet that comes out of the forming hole. A decrease in the high elasticity of melts for such systems is associated with an increase in the stiffness of the polymer, which in turn leads to a limitation of the mobility of macromolecular chains under shear stress conditions.

An important indicator that allows us to judge the influence of nanoadditives on the formability (flowability) of the polypropylene melt is the ability of the jet flowing out of the die opening to undergo longitudinal deformation. At this, the parameter characterizing the possibility of uniaxial stretching of the melt is the value of the maximum degree of longitudinal deformation (F_{\max}). Figure 3 shows the dependence of F_{\max} on the concentration of additives for a

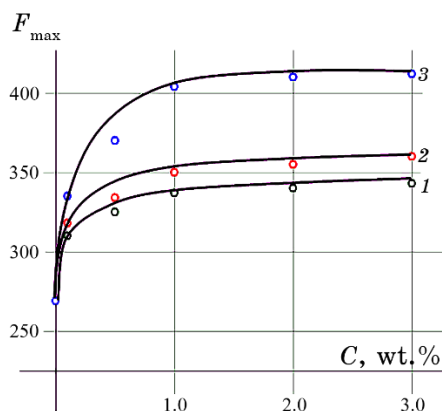


Fig. 3. Dependence of the maximum degree of drawing of the polypropylene melt on the content and chemical nature of the nanofiller: 1— SiO_2 ; 2— ZnO/SiO_2 ; 3— $\text{Al}_2\text{O}_3/\text{SiO}_2$.

PP melt filled with silica nanoparticles and mixed oxides under constant shear stress. Higher F_{\max} values for modified compositions indicate an improvement in their fibre-forming properties, since it is known that formability correlates with the values of the maximum degree of longitudinal deformation. The ability to uniaxial stretching of melts of PP/nanofiller mixtures increases with increasing content of additives: at a concentration of 1.0 wt.% value of F_{\max} increases by 1.2 times for compositions with silica and by 1.3–1.5 times for systems with mixed oxides. A further increase in the concentration of NP to 3.0 wt.% has little effect on the degree of jet stretching. The melt flowability of PP/nanofiller mixtures is also significantly affected by the chemical nature of the additives. The highest modifying effect is exhibited by the mixed oxide $\text{Al}_2\text{O}_3/\text{SiO}_2$, in the presence of which the maximum degree of deformation of the jets in the field of the longitudinal velocity gradient is achieved. The improvement of the fibre-forming properties of nanofilled PP melts is due to the increase in the strength of the jets thanks to the increase in the viscosity of the compositions. Indirect evidence of this is the correlation between the values of viscosity growth and longitudinal deformation of the jet (Figs. 1, 3).

3.2. Properties of Nanofilled Polypropylene Monothreads

For all nanofilled polypropylene melts, the process of forming and drawing monothreads from them was stable, which was to be expected, based on the results of the performed rheological studies. The resulting modified threads, like polypropylene, were monolithic, uniform in diameter along the entire length, and had a smooth surface after forming and thermal orientation drawing.

Important characteristics of materials are indicators that determine their behaviour under various imposed loads. The mechanical properties of fibres and threads are judged by the values of the modulus of elasticity, tensile strength limits, elongation at break, retention of strength in loops and knots, *etc.* The results of the study of the influence of the content of silica additives and mixed oxides on the tensile strength (TS) and the modulus of elasticity (E) of polypropylene monothreads are shown in Figs. 4, 5. As can be seen in Fig. 4, the breaking strength of nanofilled PP threads is increased with the addition of only 0.1 wt.% silica or mixed oxide. Increasing their content to 1.0 wt.% leads to an increase in TS values by 1.3–1.5 times. In the future, the strength of the threads practically does not change. The reinforcing effect of silica modified with metal oxides is much higher than that of the original silica. At the same time, the chemical nature and content of additives have little effect on the elongation of the threads at break. They are

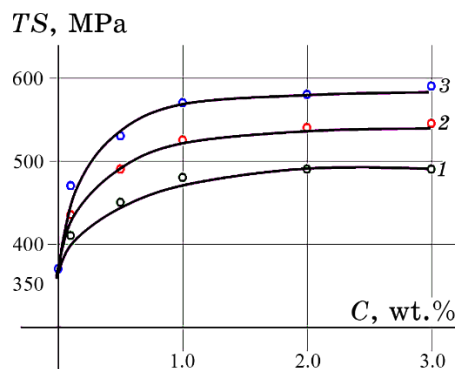


Fig. 4. Dependence of the tensile strength of polypropylene monothreads on the content and chemical nature of the nanofiller, wt.%: 1—SiO₂; 2—ZnO/SiO₂; 3—Al₂O₃/SiO₂.

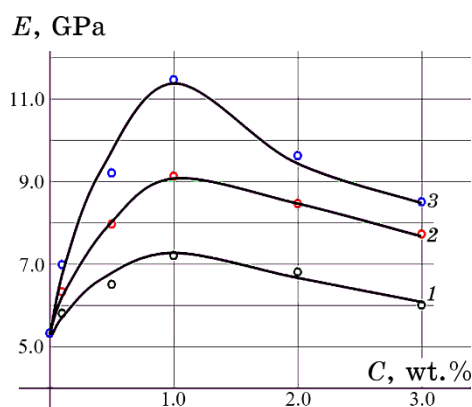


Fig. 5. Dependence of the modulus of elasticity of polypropylene monothreads on the content and chemical nature of the nanofiller, wt.%: 1—SiO₂; 2—ZnO/SiO₂; 3—Al₂O₃/SiO₂.

in the range typical for polypropylene threads and make up 10–14%.

The dependence of the modulus of elasticity of modified monothreads on the concentration of additives is more complex and extreme (Fig. 5). The maximum values of E were achieved for all investigated nanofillers at their content of 1.0 wt.%. With a further increase in the concentration of additives, the dimensional stability of monothreads deteriorates, judging by the values of E : the modulus of elasticity decreases, but remains greater than that of polypropylene threads. The values of the modulus of elasticity are consistent with the strength indicators and for the threads modified

with mixed oxides, they are higher than for the original silica. The maximum effect is achieved when using aluminium oxide/silica nanoparticles.

For nanofilled PP threads, the indicators of strength preservation in the loop and knot are also higher than in unmodified ones (Table).

This indicates that such threads are more elastic than polypropylene, and have better manipulation characteristics and can reliably fix the surgical knot.

The improvement of the mechanical properties of modified PP monothreads is the result of filling with highly dispersed additives and their positive effect on the morphology of the system and the ability to process. The injection of fillers with a high specific surface area is one of the simplest methods of strengthening materials. If silicas are used as modifiers, a noticeable effect occurs when S_{BET} exceeds 50 m²/g. The possibility of obtaining ultra-strong materials by filling with substances in the nanostate is associated with the peculiarity of their structure and the high ability to form various interactions of nanoparticles between themselves and chains of polymer macromolecules (ionic, hydrogen, covalent, van der Waals, electrostatic, *etc.*) [11]. The mixed oxides obtained by chemical gas phase deposition retain all the positive properties of the original silica, while additional active centres appear on the surface [17,

TABLE. Operating characteristics of modified polypropylene monothreads.

Nanofiller		Tex	Strength in the loop, MPa	Preservation strength at the node, %	Capillarity, mm	Hydrophilicity, %	Shrinkage, %
Chemical formula	Contents, mass %						
Without additives		5.3	290	78.4	16	0.10	7.4
Al ₂ O ₃ /SiO ₂	0.1	4.0	380	79.6	16	0.15	6.1
	0.5	3.9	420	83.3	18	0.20	5.3
	1.0	3.8	510	90.5	20	0.22	4.8
	2.0	4.0	470	84.9	20	0.24	4.9
	3.0	4.1	480	78.2	21	0.25	4.9
ZnO/SiO ₂	1.0	4.0	450	89.3	19	0.20	4.7
SiO ₂	1.0	4.2	420	86.0	21	0.25	4.9

18]. The increased chemical activity of silicas containing a metal oxide phase on the surface determines their higher reinforcing effect among nanofilled threads. The second factor that improves the properties of modified threads can also be the improvement of their supramolecular structure. It is known that, in the presence of nanofiller, the crystallization temperature of the polymer increases, which is associated with the heterogeneous effect of nanoparticle nucleation [23, 26]. At the same time, the polymer crystallization process takes place at a higher temperature, that is, under conditions closer to equilibrium. This contributes to the formation of larger and more perfect crystallites. By adjusting the crystalline structure of polypropylene due to the introduction of modified montmorillonite into its melt, the authors of Ref. [27] obtained composites with balanced values of stiffness and impact viscosity.

The positive effect of original and modified silica nanoparticles on the structure of PP threads also helps to increase their dimensional stability at high temperatures—shrinkage during boiling decreases by almost 1.5 times (Table). At the same time, the threads maintain a smooth surface and homogeneity in transverse dimensions. The presence of nanofillers helps to improve the hygienic properties of polypropylene threads—the indicators of hygroscopicity and capillarity increase. With an increase in the additive content up to 3.0 wt.% hydrophilicity increases by 2.5 times, but it remains at the level characteristic for synthetic fibres.

Thus, the performed studies showed the possibility of obtaining modified polypropylene monothreads by injection into their structure nanoparticles of original silica and combined substances aluminium oxide/silica or zinc oxide/silica. At the same time, they retain the advantages of threads from the original PP—monolithic, smooth surface and small elongation at break.

4. CONCLUSIONS

The possibility of modifying polypropylene monothreads by forming from a melt filled with silica nanoparticles and mixed ZnO/SiO₂ or Al₂O₃/SiO₂ oxides is shown. It was established that the nanofilled threads have improved mechanical properties—the breaking strength increases by 30–50% in the entire range of additive concentrations. The modulus of elasticity reaches its maximum value at a nanoparticle content of 1.0 wt.%. In the future, it slightly decreases, while remaining higher than that of the threads from the original PP. The improvement of the mechanical properties of the studied PP monothreads is due to the ability of highly dispersed solids to form a large number of various interactions of nanoparticles between themselves and chains of polymer macromolecules. The

increased chemical activity of silicas containing a metal oxide phase on the surface determines their more significant reinforcing effect.

It was established that the investigated nanoadditives improve the technological properties of the polypropylene melt by reducing the swelling of the jet at the exit from the die and increasing its deformation in the longitudinal tensile field. Ability for processing increases with increasing concentration of all studied nanofillers. At this, the value of F_{\max} increases more intensively up to the additive content of 1.0 wt. %: for compositions with silica, it increases by 1.2 times, and by 1.3–1.5 times for systems with mixed oxides. The swelling of the jet at the exit from the die is reduced by $\cong 30\%$ at the maximum concentration of fillers, regardless of their chemical nature.

It is shown that the flow of the studied nanofilled melts, as well as of the initial polypropylene, is described by a power law—the degree of deviation from the Newtonian regime is 2.0–2.1 for all systems. The viscosity of the mixtures increases with an increase in the content of nanoadditives in the entire studied range of concentrations and shear stresses. For filled polymer melts, the increase in viscosity at a constant shear rate is because solid particles do not deform during flow, and as their content increases, the flow resistance of the melt increases. For mixed oxides, the effect of filling is more pronounced, compared to the original silica, which may be due to additional structuring of the melt due to the formation of specific connections in the transition layer. The maximum increase in the viscosity of the PP melt (by 46%) occurs for compositions containing NP of aluminium oxide/silica.

Polypropylene threads filled with nanoparticles of original silica and modified metal oxides are stronger and more elastic, have low shrinkage, a smooth surface, and can have an antimicrobial effect. Based on the results of the work performed, we consider it expedient to carry out further research on the possibility of their use, in particular, in medicine as a surgical suture material and raw material for the manufacture of mesh implants.

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