


Thermally and organomodified montmorillonite as effective regulators of the structure formation process in polypropylene/polystyrene blends

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Abstract

The influence of the concentration of thermally and organomodified montmorillonite on the processes of structure formation in a blend of polypropylene/polystyrene (PP/PS) was studied. It is established that in nanofilled compositions, as well as in the original, is formed a microfibrillar structure, the dimensional characteristics of which depend on the content of the additive and the method of its modification. The investigated clays by content (0.2–2.0) wt. % by weight of polypropylene have a compatibilizing effect in the blend of PP/PS, which reduces the average diameter of microfibers by 1.6 times and increases the homogeneity of their distribution by diameter: statistical indicators decrease (standard deviation, variance, coefficient of variation). The modifying effect of thermally modified clay is higher—the improvement of the dimensional characteristics of PP microfibers is achieved at its minimum content in the blend (0.2 wt. %). The formation of anisotropic PP structures in the PS matrix and the relaxation of the accumulated stresses at the exit of the molding hole is the main factor that causes high values of the coefficient of swelling of the extrudates (4.0–7.7). The change in the microstructure of the extrudates of nanofilled systems during spinning depends on the composition of the blend and the method of modification of montmorillonite. The average diameters of microfibers decrease from 2.2 μm (in the original blend) to (1.3–2.0) μm (in three-component) depending on the content of additives. The ability to regulate the microstructure of incompatible polymer blends by introducing additives of thermally and organomodified montmorillonite will contribute to the creation of nanocomposites with controlled morphology, as well as new fine-fiber materials with improved filtering characteristics.

Keywords

Polymer blend, montmorillonite, swelling, microstructure, microjet

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Introduction

The unique structure and properties of natural aluminosilicates provide wide opportunities for the creation of a number of multifunctional polymeric materials. Polymer/clay nanocomposites show a sharp improvement in strength and modulus of elasticity,¹ heat and fire resistance² and gas permeability,³ even with a lower clay content compared to other nanofilled polymer systems. Composites for water purification,⁴ new ecologically safe adsorbents for environmental restoration,⁵ biomedical materials,⁶ etc. were created on the basis of biopolymers. The basis of clays are silicon and alumina ions, which form respectively tetrahedral and octahedral two-dimensional grids, interconnected into layers (plates), which self-organize into packages with an interlayer distance in the nanometer range. The outer and inner surfaces of the plates are hydrophilic and polar, which promotes wetting and penetration into the space between the layers of both low- and high-molecular compounds that have polar groups in their structure. Due to this, layered silicates are the most effective modifiers for polar polymers.⁷

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A more difficult task is to obtain nanocomposites based on non-polar or weakly polar polymers. The incompatibility of hydrophilic clays and hydrophobic polymers is the main problem. To solve this problem clays are pre-modified using some of the following techniques: clay cations are replaced by organic cations by way of ion exchange; water-soluble polymers, alkyl ketones, methyl acrylate, surfactants, are adsorbed on the surface of the particles; organosilanes are grafted to the clay surface with the formation of the Si-O-Si bonds; organic molecules capable of van der Waals or ion-dipole interaction with the clay surface are introduced, etc.^{1,7,8} The nature of the packaging of the modifier molecules in the interlayer space determines the organophilicity of clays and the value of increase in the distance between the silicate plates, and, as a result, the structure of nanocomposites when mixed them with polymers.

Layered clay nanofillers also proved to be effective modifiers of polymer blends due to their influence on interphase phenomena and processes of structure formation of the dispersed phase component in the matrix.^{9–15} The improvement in the performance of composite products filled with aluminosilicates is due to changes in the morphology of polymer dispersions. Thus, the introduction of clays with different degrees of hydrophobicity in the blend of polystyrene/polypropylene allowed to increase the strength of the products by reducing the size of the droplets of PS and increase the homogeneity of their distribution.⁹ Thinning of droplet-matrix structures also took place in the PS/polymethylmethacrylate blend under the influence of clay additives.¹⁰ Natural nanosized aluminosilicates are used to create so-called “green” polymer nanocomposites. Biodegradable composites with high mechanical properties are obtained from blends of polylactide (PL) with thermoplastic starch and polybutyleneadipate/teterephthalate copolymer filled with organomodified and coated with Na⁺ montmorillonite ions, respectively.^{11,12} The addition of alumina nanoparticles in a blend based on secondary polyethylene terephthalate allows to use it repeatedly due to the improvement of the mechanical properties of the compositions.¹³ With the predominant location of flat particles of organomodified clay at the interphase area in polyamide (PA)/PL blends, a qualitative change in the type of morphology of polyamide in the PL matrix occurs—a transition from the drop-matrix to the interlaced type, as a result of which the heat resistance and plasticity of the composite material increase without negative impact on its rigidity and strength.¹⁴ Polymer composites with appropriate morphology are a semi-finished product for the production of materials with adjustable porous structure by selective removal of one of the components. Thus, the porosity and average pore diameter in microporous polymer membranes obtained by the template method were regulated by changing the number of particles of organomodified montmorillonite in the film.¹⁵

Thus, using of layered aluminosilicates with different degrees of hydrophobicity, the choice of methods for their modification, the directional formation of organophilic layers and taking into account the properties of the components of the polymer blend allows to obtain materials with adjustable morphology and pre-predicted characteristics.

The aim of the work is to study the influence of the method of modification of montmorillonite and its concentration on the processes of structure formation in a blend of polypropylene/polystyrene to obtain fine-fiber materials with improved filtering characteristics.

Experiment

Materials

The studies were performed on a blend of polypropylene/polystyrene (PP/PS) composition of 30/70 wt. %, in which the matrix component was polystyrene (PS) brand PSM-115 with a melt flow rate (MFR) of 8 g/10 min and a glass transition temperature of 112°C, and fiber-forming—isotactic polypropylene brand Sabic 575P with MFR 11 g/10 min and a melting point of 169°C. As a modifier was used natural clay of montmorillonite type produced by the company “Dashbent” of two brands C4T₂K (M1) and P1T₁KA (M2), which was introduced into the blend in the amount of (0.2–2.0) wt. % by weight of polypropylene. To increase the compatibility of the nanoadditive with the PP melt, the clay samples were pre-modified.

Clay M1 was subjected to heat treatment in a muffle furnace, gradually heated (for 3 h) to a temperature of 750°C and kept at this temperature for 3 h.

A sample of M2 clay was treated with a surfactant brand *Santreat 3050* manufactured by “Melzer.” To do this, a portion of clay (50 g) was pre-dispersed in a small amount of distilled water (50–100 mL) and thoroughly ground to a homogeneous state. The resulting suspension was quantitatively transferred to a flask and the volume was gradually adjusted to 1000 mL with stirring. The filled flask was heated to 80 ± 5°C. After reaching the desired temperature and with constant stirring, gradually dropwise (over 3 h), were added 30 mL of surfactant. The flask was then cooled and the modified clay and working solution were separated by decantation. The precipitate was washed with water, filtered, quantitatively transferred to a heat-resistant beaker and dried in an oven at a temperature of 80 ± 5°C.

After cooling to room temperature, samples of the modified clays were thoroughly ground in a porcelain mortar to a powdery state and sieved through a 0.2 mm metal sieve to remove impurities and large aggregates of particles.

Preparation of blends

PP/PS blends filled with different amounts of modified clays were obtained by extrusion on an extrusion-spinning machine UFTP-2U in three stages. In the first stage were received granules PP/clay with a maximum concentration of the additive—5.0 wt. %. The original PP granules were pre-moistened with a 10% solution of surfactant in water. Then, gradually, with constant stirring, clay was applied to their surface, dried at a temperature of 80 ± 5°C for 5 h, and then mixed in the melt. In the second

stage, PP/clay samples containing 0.2; 0.5; 1.0; and 2.0 wt. % of the additive by weight of the PP were prepared by dilution. To do this, the granules PP/clay of composition of 95/5 wt. % were mixed in the melt with the calculated amount of the original PP. In the third stage, a blend in the ratio of 30/70 wt. % was prepared from PP/clay granules with different amounts and types of clay and PS and then extruded. In all cases, the extrusion temperature was (255–260)°C. A spinneret with a single hole with a diameter of 1.1 mm was used, with a melt flow rate of 11.8 cm³/min. As a result, from the compositions PP/PS/clay, which contain (0.2–2.0) wt. % by weight of PP thermally or organomodified clay, composite strands were obtained under conditions of free flow of melt from the forming hole and in spinneret extraction due to forced removal of the extrudate at a speed of 13 m/min.

The coefficient of die swell (B) was calculated using equation (1)

$$B = D_{\max}/D_0 \quad (1)$$

where D_{\max} —the maximum diameter of the jet (mm) at a distance of 250 mm from the die in terms of free flow of melt from the forming hole; D_0 —diameter of the hole of the die, mm.

The deformation multiplicity of PP microfibers in spinneret drawing (DR) was calculated using equation (2)

$$DR = d_0^2/d_x^2 \quad (2)$$

where d_0 , d_x —average diameter of microfibers (μm) before and after spinneret drawing, respectively.

Selective dissolution of the matrix component (PS) from the composite extrudates was carried out with a solvent inert to PP—ethylacetate, at room temperature. The types of structures formed by PP in the PS matrix were determined by photomicrographs of polypropylene residues after extraction. It was used an optical microscope brand “Biolam,” equipped with a digital camera at optical magnification 15 × 20. Data on the dimensional characteristics and morphometric composition of the studied samples were obtained using the method of image analysis in the program “ImageJ.” Statistical processing of experimental data was performed using software packages “Statistica” and “Excel.” At this, the following statistical characteristics were calculated: minimum, maximum, average diameters of microfibers, standard error of the average, standard deviation, variance, coefficient of variation, asymmetry of distribution.

Results and discussion

Influence of the concentration of thermally and organomodified clay on the elastic properties of the melt of the PP/PS blend. Rheological properties play an important role in the processing of polymer melts and their blends. During the flow of melts at the exit of the molding hole, there is an expansion (swelling) of the extrudate (Barus effect or the phenomenon of elastic recovery), which affects the size and shape of the jet. The expansion of the extrudate is due to the manifestation of the elastic properties of polymeric liquids, and the relaxation of reversible deformations after the exit of the melt jet from the forming hole. For blends of polymers, the values of expansion are much higher compared to the original polymers because there is a relaxation of stresses accumulated by macromolecules in each phase, as well as the structures of the dispersed phase due to their deformation and orientation in the flow direction.^{16,17}

The results of research (Figure 1) showed that in the accepted conditions of extrusion for the original PP/PS composition are realized additive values of the coefficient of die swell (B), which significantly exceed the value of (B) of based blend.

High values of (B) indicate the formation of anisotropic structures of polypropylene, which are new relaxing elements, characteristic only of two-phase systems. The maximum degree of anisotropy occurs when the dispersed phase forms liquid microjets, which make a major contribution to highly elastic deformations at flowing of polymer dispersions. In our previous studies using various blends and modifiers, it was shown that the swelling of the extrudates can be an indirect characteristic of the fiber formation of the polymer of the dispersed phase in the matrix.¹⁷

Experimental results on the effect of the amount of montmorillonite on the degree of elastic recovery of the jets of melts of PP/PS blends showed that the nature of the dependence of (B) on the clay content is significantly influenced by the method of its modification.

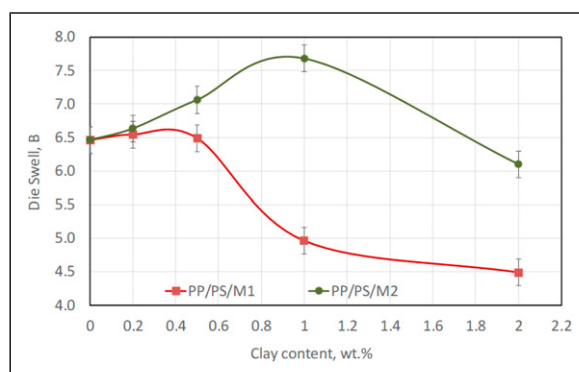


Figure 1. Dependence of the amount of extrudate swelling (B) on the clay content in the compositions PP/PS/M1 and PP/PS/M2.

Thus, subject to introduction into the blend (0.2–0.5) wt. % of heat-treated clay M1 the amount of swelling does not change, and further increase of concentration causes its significant drop. For compositions containing organomodified clay M2, the dependence “die swell – composition” has the form of a curve with a maximum that is achieved at an additive concentration of 1.0 wt. %.

The different nature of the influence of clays on the degree of elastic recovery of PP/PS melt jets can be explained by fundamentally different structural transformations in montmorillonite in the process of its thermal and organomodification. In the first case, there is a deep restructuring associated with the processes of dehydration, dehydroxylation and partial destruction of individual layers.¹⁸ In the second, the modification relates mainly to the interlayer space, without significantly affecting the general structural order of montmorillonite.¹⁹

It should be noted that the reduction of (B) when using clay M1 as a modifier of PP/PS compositions leads to the stabilization of the jet during molding and significantly increases its ability to spin.

Thus, the obtained results indirectly indicate the formation of a microfibrillar structure in a blend of PP/PS and the influence of clay additives on the process of its formation.

The influence of the concentration of thermally and organomodified clay on the processes of structure formation in the PP/PS blend. Natural nanosized aluminosilicates have a compatibilizing effect in blends of polymers, regardless of saturation of them in the interphase, which allows to adjust the morphology of composites in a wide range.^{9,10} Thus, the method of SEM microscopy showed that in the compositions of thermoplastic starch/poly lactide modified with organoclay additives, their particles are dispersed in both phases, while significantly changing the morphology of the blend.¹¹ The ability of clay particles to exhibit a compatibilizing effect was confirmed by dynamic mechanical analysis, which showed the rapprochement of the glass transition temperatures of the components of the blend.¹² The high efficiency of clays is associated with the flat shape of their particles, which allows them to better adapt to the two-dimensional structure of the interfacial layer.

Visual analysis of polypropylene residues after extraction of polystyrene from extrudates of bi- and three-component blends confirmed our prediction on the formation in situ PP microfibers in the PS matrix during flowing of the initial and nanofilled blends. Quantitative studies of the microstructure by image analysis method showed that the addition of thermally and organoclay in a blend of PP/PS have a modifying effect in the entire studied range of concentrations (Figure 2, Table 1).

Micrographs of PP microfibers and histograms of their distribution by diameters (Figure 2) show that the binary blend is characterized by a more inhomogeneous structure. The diameters of microfibers are in the range (0.6–7.4) μm , their average diameter (\bar{d}) is 2.7 μm , and the largest share of fibers (~20%) refers to the size range of (2.0–2.5) μm . Provided the introduction of only 0.2 wt. % of thermally or organomodified montmorillonite, the average diameter of PP microfibers decreases to 1.7 and 2.2 μm , respectively. The homogeneity of the distribution has improved, as evidenced by the decrease in indicators that characterize it: standard deviation ~ at 3 times, variance at 8 times, coefficient of variation at ~2 times (Table 1).

A further increase in the amount of additive in the system affects the microstructure of the extrudates in different ways. In blends containing thermoclay, coarsening of morphology occurs, and in the presence of an organomodified additive, the dependence $\bar{d} = f(\text{Clay content})$ is extreme—the minimum values correspond to the concentration (0.5–1.0) wt. %. Such a dependence of the degree of dispersing of the component of dispersed phase on the content of modifier occurs when using classical compatibilizers²⁰ or nanofillers,²¹ due to the release of additives in a separate phase subject to achievement of a critical concentration and reduction their surface activity. For heat-treated clay the best result is achieved with a minimum content, which indicates its higher efficiency compared to organomodified. As shown above, this may be due to the different nature of the structure changes and the degree of hydrophobicity in the modification of montmorillonite samples. During the heat treatment of clay by its gradual heating, at the beginning (100–150)°C dehydration processes take place, when clay minerals lose adsorbed and hydrated water. Upon further heating to a temperature of (700–750)°C dehydroxylation processes take place—the clay loses chemically bound water in the interlayer space.^{18,22} As a result, its structure, color, as well as the ability to absorb and retain water (hydrophobization) change significantly. Under the conditions of organomodification by a surfactant, oriented adsorption layers are formed in the interlayer space of clay particles, which lyophilize it in relation to the polymer. As a result, the distance between the individual silicate plates increases several times, and the surface of the silicate particles is hydrophobized to ensure its compatibility with non-polar polymer molecules.

A significant effect of the hydrophobicity of clays on the morphology of the compositions was established, in particular, by the authors⁹ when studying a complementary PS/PP blend: clay particles with a wetting coefficient $k \leq 1$ are located at the interphase area and contribute to a decrease of PS droplets in the PP matrix, and clay particles with $k > 1$ accumulate in polystyrene droplets, while the morphology of the blend practically does not change.

Influence of longitudinal deformation on the processes of structure formation in PP/PS blends filled with thermally and organomodified montmorillonite. The formation of fibers and films from melts of polymer blends is almost always associated with their longitudinal deformation after leaving the molding hole. The multiplicity of the spinneret drawing is one of the important indicators that affects the process of structure formation of the component of dispersed phase in the matrix before curing. The change in the microstructure of extrudates of polymer blends in the field of the longitudinal velocity gradient depends on its size, chemical nature and the ratio of the components of blend, their rheological characteristics at the molding temperature. At this, there are several competing processes associated with the subsequent deformation and orientation of the particles of dispersed phase, coalescence, migration, destabilization and decay of already formed microjets by the capillary mechanism, etc.¹⁷

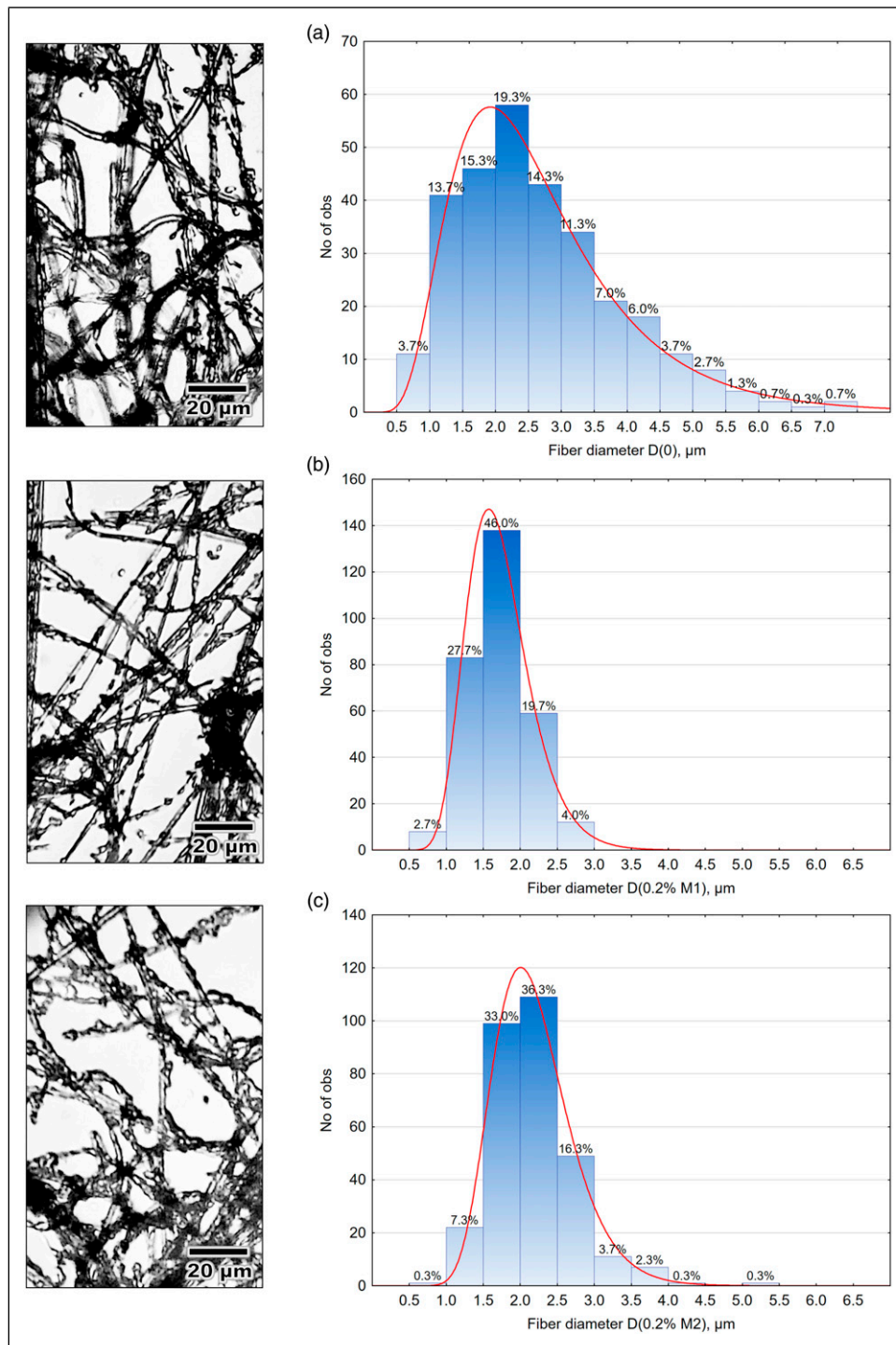


Figure 2. Microphotographs and histograms of the distribution of PP microfibers by diameters obtained from the original blend of PP/PS (a); blends filled with 0.2 wt. % of clay M1 (b), and M2 (c).

Table I. Influence of thermally and organomodified clay content on statistical indicators of PP microfibers fiber distribution by diameter.

Indicators	The initial PP/PS blend	Content of clay, wt. %							
		M1				M2			
		0.2	0.5	1.0	2.0	0.2	0.5	1.0	2.0
Mean diameter, μm	2.7	1.7	1.8	2.2	2.7	2.2	1.7	1.7	2.4
Minimum diameter, μm	0.6	0.7	0.7	0.8	0.5	0.9	0.6	0.6	1.3
Maximum diameter, μm	7.4	2.9	3.5	4.0	6.6	5.1	3.6	3.1	4.1
Median, μm	2.4	1.7	1.8	2.1	2.6	2.1	1.6	1.7	2.4
Variance	1.6	0.2	0.2	0.3	0.5	0.3	0.2	0.2	0.3
Standard deviation	1.3	0.4	0.5	0.5	0.7	0.5	0.5	0.4	0.6
Coefficient of variation, %	46.7	24.1	27.6	24.9	26.6	25.1	27.4	25.6	22.9
Skewness	0.9	0.2	0.6	0.7	0.9	1.2	1.1	0.4	0.3

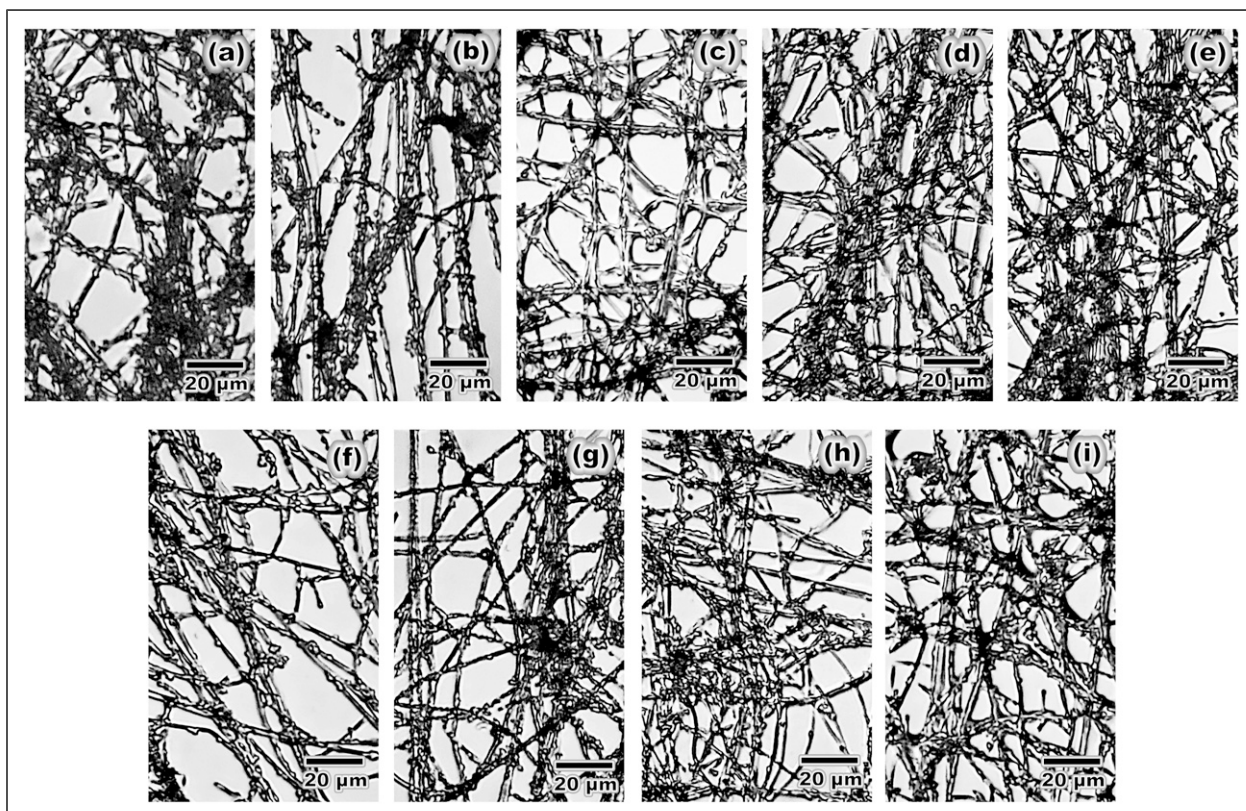


Figure 3. Microphotographs of PP microfibrils obtained by spinneret drawing: the initial PP/PS blend (a); blends modified with clay M1 (b-e) and M2 (f-i). The clay content in the compositions (wt.%): 0.2 (b, f); 0.5 (c, g); 1 (d, h); 2 (e, i).

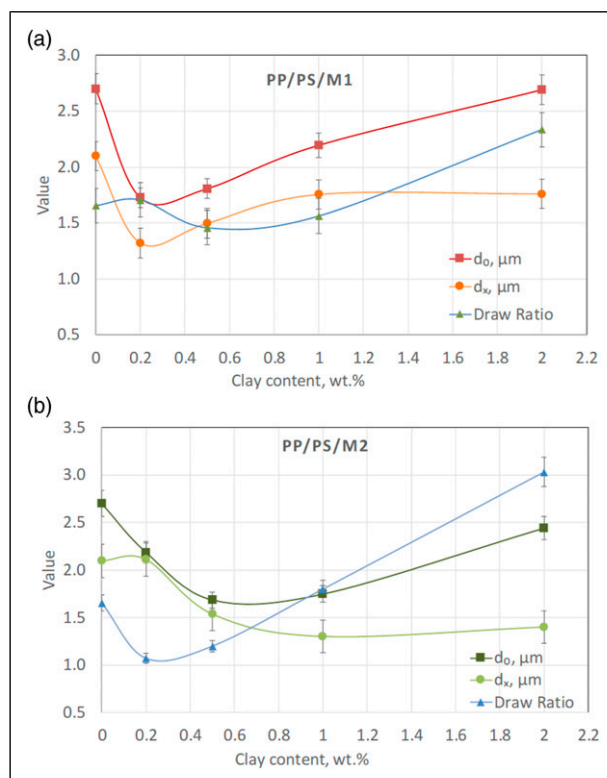


Figure 4. The influence of the concentration of thermally (a) and organomodified (b) montmorillonite in PP/PS blend on the average diameter of PP microfibrils before (d_0) and after (d_x) spinneret drawing, as well as the multiplicity of their deformation.

Spinneret drawing causes partial suppression of the die swell effect. It happens a decrease in the diameter of the extrudate, which contributes to its faster cooling while maintaining the formed microfibrillar structure. The degree of longitudinal deformation of the polymer of dispersed phase in the blend depends on the strength of interaction of the components of dispersion at the interphase area. In case of insufficient interaction, microfibrils can be extracted to a lesser extent than the composite extrudate as a whole, or can be destroyed if the extraction multiplicity exceeds the allowable value for this polymer.

The results of study of the microstructure of extrudates with spinneret drawing (Figure 3) showed that the matrix-fibrillar morphology created during formation is preserved both for the initial and for nanofilled blends, regardless of type and amount of clay.

Data of morphometric analysis of PP microfibers, obtained under conditions of forced removal, show that their average diameters decrease for all studied compositions: in bi-component—in 1.3 times, and in three-component—in (1.2–1.6) times, depending from the content of the additive (Figure 4). A decrease in such statistical characteristics as the values of the coefficient of variation and standard deviation indicates an increase in the homogeneity of the distribution of microfibrils by diameter.

At this, as can be seen from Figure 4, the nature of curves of the dependence of deformation multiplicity of PP microfibrils on the content of nanoadditives for thermally and organomodified clay are different. In compositions containing thermally modified clay in an amount of (0.2–1.0) wt. %, the value of deformation is almost unchanged, and is at the level of unmodified blend.

At the same time, the use of organomodified clay in the amount of (0.2–0.5) wt. % leads to a significant decrease in the ability of microfibrils to stretch under the conditions of spinneret drawing. This may be due to both the specific effect of clay of this type on the elastic properties of the dispersion (Figure 1) and insufficient homogeneity of distribution of M2 particles at their low concentrations in the blend.

Regardless of the method of modification, increasing the concentration of montmorillonite from 1.0 to 2.0 wt. % increases the degree of extraction of microfibrils, compared with the unmodified blend, by about 40 and 80% for thermally and organomodified clay, respectively. This may be due to improved adhesion between the macromolecules of the blend components in the transition layer due to its saturation with nanosized aluminosilicate particles.

Conclusions

It was found that introduction into the melt of a blend of polypropylene/polystyrene of thermally or organomodified montmorillonite in an amount of (0.2–2.0) wt. % allows you to control the process of *in situ* formation of PP microjets (microfibril) in the PS matrix. The clays show a compatibilizing effect in the compositions at all investigated concentrations, which causes a decrease in the diameters of microfibrils in the modified systems to 1.7 μm vs 2.7 μm in the initial blend. At the same time, the homogeneity of the distribution of microfibrils by diameters increases, as evidenced by the decrease in such indicators as standard deviation (\sim in 3 times), variance (in 8 times), coefficient of variation (\sim in 2 times). Thermomodified clay is a more effective structural modifier: the minimum values of the average diameter of PP microfibers are achieved at a lower its content (0.2 wt.%) in the composition.

It is shown that the nature of dependence of the expansion coefficient of jet of the polymer blends (B) on the clay content is determined by the method of its modification. For blends with thermally modified clay, the indicator does not change with the content of the additive (0.2–0.5) wt. %, and then significantly (\sim in 1.42 times) decreases with increasing concentration to 2.0 wt. %. In compositions with organomodified clay, the dependence (B)–composition has extreme nature with a maximum value of (B) at a concentration of 1.0 wt. %.

It was found that the addition to the original PP/PS blend $>$ 0.5 wt. % of both types of investigated clays contributes to the effectiveness of further reducing the diameters of PP microfibrils (1.2–1.7 times, depending on the content and type of additive) due to spinneret drawing. At this reduces the variability in the distribution of PP microfibrils in diameter, which shows an increase in their homogeneity in this indicator.

Regulation of the microstructure of incompatible polymer blends by introducing thermally and organomodified montmorillonite additives into their melts will promote the creation of nanocomposites with predetermined morphology and properties, as well as new fine-fiber materials with improved filtering characteristics.

Declaration of conflicting interests

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