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INFLUENCE OF BINARY ADDITIVES OF COMPATIBILIZERS ON THE MICRO- AND MACRORHEOLOGICAL PROPERTIES OF MELTS OF POLYPROPYLENE–COPOLYAMIDE MIXTURES

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We have investigated the influence of binary additives of compatibilizers copolymer of ethylene with vinyl acetate/siloxane liquid on the rheological properties of melts and the processes of structure formation in polypropylene–copolyamide mixtures. It has been shown that there exists an optimal ratio of compatibilizers at which the maximum influence of additives on the process of specific fiber formation shows up. The mechanism of their action is connected with an increase in the length of the interphase layer, a decrease in the surface tension, and an increase in the lifetime of the liquid jet. The use of compatibilizers influences the viscosity of melts only slightly, and the capability of four-component compositions to be processed markedly increases.

The rapidly developing production of particles from polymer materials used in different fields of technology calls for imparting new given properties to them. The world's experience has shown that a promising way of solving this problem lies not in the development of new raw materials but in modification of the polymers and oligomers brought to a commercial level. Mixing polymers is a simple, available, and often the most effective method of their modification. Materials based on mixtures of polymers are characterized by nonlinearity and synergism of properties that are due to their unique two-phase structure. The last few years have seen an active search for physicochemical factors determining the compatibility or segregation of components, the formation of the characteristic microheterogeneous structure, as well as the features of the conjugation of micro- and macrophases.

One of the most interesting processes observed in processing mixtures of two polymers is the phase separation in the form of a specific structure formation. Polymers form most commonly an incompatible mixture, but their separation is prevented by the very high viscosity of the phases of melts. For example, on going from a wide reservoir to a narrow one the dispersed component is released in the form of very fine jets stretched by the flow of the second component (matrix). We named this phenomenon the specific fiber formation [1].

Mixtures of polymers are specific colloidal systems. They are characterized by the presence of an interphase layer whose properties differ from the analogous indices of the volume phases of the components. It is compatible with the materials of both phases and provides the transfer of stresses from the dispersive medium to the dispersed phase. The surface-tension value on the interface σ can be controlled by introducing special substances (compatibilizers) bearing a similarity to one or both components of the mixture [2]. Thermodynamically, the surface tension is the work needed for the formation of a unit area of a new surface by stretching the old one. Thus, by decreasing σ it is possible to decrease the quantity of energy expended in forming new surfaces, i.e., promote the dispersion and deformation of drops of the dispersed-phase polymer. Our investigations have shown that additives of compatibilizers permit enhancing the fiber-formation process [2–4]. However, despite the wide application of compatibilizers for modifying polymer mixtures, their use for creating new, fine-fibered polymer materials has up to now been little studied.

The aim of the present work is to investigate the influence of additives of compatibilizers on the processes of flow and structure formation in melts of propylene–copolyamide (PP–CPA) mixtures.

Objects and Methods of Investigation. The objects of investigation were PP–CPA mixtures in a proportion of 50/50 mass %, in which PP acts as a dispersed phase. We used samples of commercial polymers: PP of brand 21060 and CPA containing 50% of caprolactam and 50% of PA-6/66 hexamethylenadipinate. For compatibilizers, we

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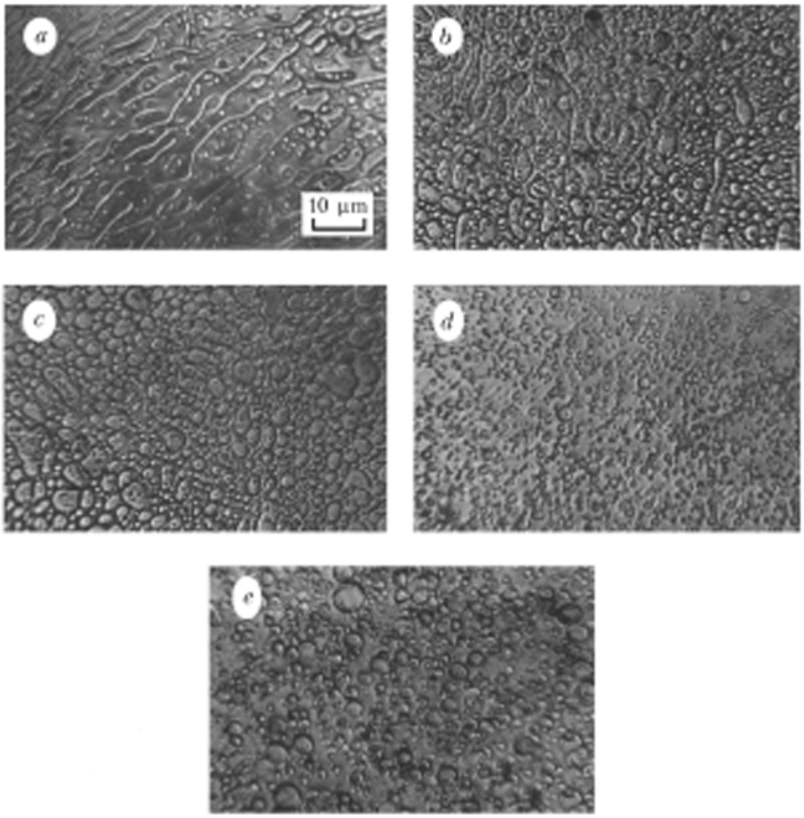

Fig. 1. Microphotographs of the cross sections of extrudates of PP–CPA/CEVA/PES-5 mixtures in proportions (mass %) of: 50/50/0/0 (a); 50/50/0.5/0.3 (b); 50/50/1.0/0.3 (c); 50/50/1.7/0.3 (d); 50/50/5.0/0.5 (e).

chose mixtures of PES-5 silicon organic liquid and copolymer of ethylene with vinyl acetate (CEVA) with the following CEVA/PES-5 component ratio: 0.5/0.3; 1.0/0.3; 1.7/0.3; 5.0/0.5 mass %. The quantity of additives was calculated from the mass of polypropylene in the mixture. The properties of the starting PP, CPA, CEVA, and PES-5 are given in [2, 3]. Mixing of the polymers with the additives was carried out on a combined LGP-25 screw-disk extruder. The viscosity η of the melts of the mixtures was measured by means of an MV-2 viscosimeter in the range of shear stresses $\tau = (0.1–5.7)\cdot 10^4$ Pa at a temperature of 190°C.

The results were processed by conventional methods. The elastic properties of the melts of the mixtures were estimated by the swelling B of extrudates by the method of [5]. The guaranteed experimental error in determining η and B was ±2–3%. The capability of the melt for longitudinal deformation was determined by the value of the maximum spinneret stretching Φ_m calculated as a ratio of the highest velocity of reception of a melt jet to the velocity of its efflux from the spinneret. The guaranteed error is ±7%. The structure formation was estimated qualitatively by microphotographs of the longitudinal and cross sections of mixture extrudates and quantitatively by determining with a microscope the number and sizes of all types of structures in the residual after extracting the matrix polymer from the extrudate. Analysis was carried out by means of an MBI-15 microscope. The data obtained were processed by the methods of mathematical statistics, as a result of which the mean diameter d of the microfibers and the dispersion of their distribution δ^2 were determined and diameter distribution curves of the microfibers were plotted.

The mechanisms of disintegration of liquid jets (microfibers) were investigated by the method of [6] based on the measurement of the disturbance-wave growth rate on the liquid-jet surface in accordance with Tomotika's theory [7]. To this end, we placed longitudinal sections of mixture extrudates on the heating microscopic stage, increased the temperature, and photographed different stages of the process of disintegration of the jet into a chain of drops. From the photographs obtained we determined the radii of the starting microfiber R and drops r and the maximum length of the disturbance wave λ_m. Then we calculated the instability coefficient q and the lifetime (disintegration time) t_{life} of

TABLE 1. Characteristics of the Microstructure of Extrudates of the Compatibilized PP–CPA Mixtures

Content of CEVA/PES-5 compatibilizers, mass %	Continuous-length fibers			Short fibers	Particles	Films
	$\bar{d}$, μm	φ, %	δ^2, μm^2	φ, %		
5.0/0	4.0	70.0	3.3	13.1	10.1	6.8
0/0.5	4.1	72.0	5.0	12.6	9.3	6.1
0.5/0.3	3.8	75.5	3.0	11.0	9.5	4.0
1.0/0.3	3.4	88.8	2.8	5.1	4.1	2.0
1.7/0.3	1.8	91.8	1.9	3.6	3.0	1.4
5.0/0.5	2.8	78.0	2.2	11.3	6.0	4.7

the jet, as well as the value of the surface tension σ on the interface. The temperatures and heats of the phase transitions of the investigated samples were determined with the use of a DuPont 2000 DSC V4.0B differential scanning calorimeter in the temperature range 223–493 K at a heating rate of 1 K/min, as well as a DSK-D calorimeter; the heating rate of a sample was 2 K/min in the temperature range 123–540 K; the reference substance was Al_2O_3 [8]. The error in determining the transition temperatures was ±1 K. The phase-transition heats were determined with an error of ±3.5%. For calibration in calculating the thermal effects, we recorded the melting thermogram of parabenzoic acid with a known thermal effect of 151.9 J/g.

Results and Discussion. *Influence of binary additives of compatibilizers on the morphology of mixtures.* To realize the phenomenon of specific fiber formation, it is essential, on the one hand, that both components are compatible, yet not to such an extent that they wet each other, and, on the other hand, that the compatibility does not lead to a mutual dissolution with the formation of a homogeneous system. Nonpolar polypropylene is not compatible with copolyamide whose macromolecules are polar. The interphase layer is not extended and there is a slight adhesion on the interface; therefore, the transfer of stresses from the dispersion medium polymer to the dispersed-phase drops is difficult. The fiber formation of PP in the CPA matrix is little pronounced even for mixtures containing 20 and 30 mass % of polyprolylene [1]. At the same time, an increase in the content of the fiber-forming component in the mixture is economically justified. Thus, the problem of improving the compatibility for this pair of polymers is highly topical.

The investigations performed have shown that the introduction of compatibilizers is one of the most effective factors that permit regulating the morphology of PP–CPA mixtures, namely the degree of dispersion, the uniformity of distribution, and the relation between different types of structures (Fig. 1). The addition of even small amounts of binary mixtures of compatibilizers radically changes the microstructure formed. The presented microphotographs of the cross sections of extrudates of PP–CPA mixtures in a proportion of 50/50 mass % show that the starting mixture has a layered morphology (Fig. 1a). The introduction of CEVA-PES-5 in a proportion of 0.5/0.3 mass % leads to a stepwise change in the character of the microstructure of extrudates: polypropylene becomes a dispersed phase; the dispersion is fine and homogeneous, though some particles have an irregular form (Fig. 1b). As the content of CEVA in the mixture is increased at 1.7 mass %, the PP particles decrease in size and acquire a regular form (Fig. 1c–e). The latter points to a decrease in the interphase tension in the mixture, i.e., to a compatibilization. A further increase in the content of both components leads to a predominance of coalescence processes, which shows up as an increase in the diameter of PP drops and their homogeneity in sizes (Fig. 1e).

The detailed quantitative characteristics of the microstructure of extrudates of three- and four-component mixtures have shown that the main type of structure in the compatibilized PP–CPA mixtures is PP microfibers of continuous length (Table 1), and their diameter and homogeneity therewith depend on the quantity of compatibilizers. It has been established that there exists their optimal ratio in the binary mixture at which the maximum influence on the structure formation processes is manifest. From the point of view of realizing the phenomenon of specific fiber formation, one should recommend the CEVA/PES-5 in a proportion of 1.7/0.3 mass %. In this case, the diameter of microfibers is minimal (1.8 μm) and their number is maximal (91.8%), the diameter distribution curve is the narrowest, and coarse fibers are absent (Fig. 2).

Mechanisms of disintegration of microfibers in the compatibilized PP–CPA mixtures. One of the microrheological processes forming the structure of polymer dispersion, along with the deformation and streamwise coalescence of dispersed-phase polymer drops, is the breaking of liquid jets.

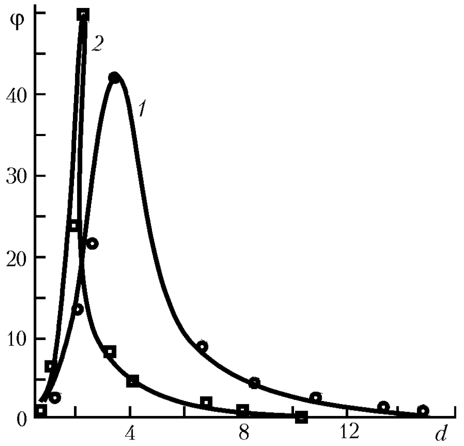

Fig. 2. Experimental curves of the diameter distribution of PP microfibers in extrudates of compatibilized PP–CPA/CEVA/PES-5 mixtures in proportions (mass %) of: 1) 50/50/0/0.5; 2) 50/50/5.0/0.5; (φ, %; *d*, μm.

TABLE 2. Parameters of the Disintegration Kinetics of PP Microfibers in the CPA Matrix

Content of CEVA/PES-5 compatibilizers, mass %	*r*/*R*	λ_m, μm	*q*	t_{life}, sec	t_{life}/R, sec/mμ	σ, mN/m
Without additives	1.9	13.8	0.0618	56	37	2.60
5.0/0	1.9	31.7	0.0362	70	54	1.85
0/0.5	1.8	27.6	0.0327	70	41	1.90
0.5/0.3	1.8	19.4	0.0315	85	61	1.55
1.0/0.3	1.8	16.9	0.0254	120	69	1.39
1.7/0.3	1.8	12.0	0.0133	150	90	0.53
5.0/0.5	1.9	14.9	0.0182	85	62	1.30

It is known that a liquid cylinder (jet) is thermodynamically unstable and in the course of time breaks up into a chain of drops. The reason for this is the appearance on its surface of wave disturbances whose amplitude increases exponentially with time [7]. Breaking occurs when the amplitude of the disturbance wave is equal in value to the jet radius. The lifetime of the liquid jet t_{life} is directly proportional to the diameter of the initial cylinder and inversely proportional to the tension [9]. Consequently, all other things being equal, a decrease in the value of σ promotes the stabilization of jets of a smaller diameter, i.e., finer fibers are produced. On the other hand, a decrease in the interphase tension is followed by the formation of a transition layer of sufficient extent which provides the transfer of deformation stresses from the matrix to the dispersed-phase polymer drops. The degree of interaction between the components on the interface also influences the mechanisms of disintegration of liquid jets [1]. Since, in the flow of the binary PP–CPA mixture in a proportion of 50/50, fibers are not formed, we used for comparison the results of the investigation of the mixture in a proportion of 20–80 mass % (Table 2). Analysis of the data on the disintegration of PP microfibers in the CPA matrix confirms the considerable increase in the reduced lifetime t_{life}/R of polypropylene microfibers in compatibilized mixtures. The stability of microfibers depends on the quantity of additives. For instance, while upon the introduction of a binary mixture consisting of 0.5% of CEVA+0.3% of PES-5 t_{life}/R increases to 61 sec/μm, as the concentration of CEVA is increased to 1.7% the reduced lifetime is 90 sec/μm. We associate the established mechanism mainly with the decrease in σ at the PP–CPA boundary due to the compatibilization. And this factor determines the realization of the special fiber formation in PP–CPA mixtures for compositions corresponding to the region of phase change and provides a decrease in the diameter of microfibers even compared to the mixture containing 30 mass % of PP (the mean diameter of microfibers is 5.7 μm) [3]. Particular emphasis should be placed on the increase in the content of the fiber-forming component in the mixture to 50 mass %, which provides an increase in the yield of microfibers and simplifies the process of matrix polymer extraction, as well as CPA and solvent regeneration.

TABLE 3. Melting Temperatures and Heats of the Starting Polymers and Their Mixtures

Sample	T_{mel}, K	ΔH, J/kg
PP	450/450	81.0/84.2
PP+0.5 mass % PES-5	450/448	62.3/66.3
CPA	450/450	26.7/24.5
PP+0.5 mass % PES-5	443/444	34.7/32.2
PP–CPA 50/50 mass %	450/450	70.7/68.2
PP–CPA+0.5 mass % PES-5	445/447	44.3/45.3
PP–CPA+CEVA/PES-5 5.0/0.5 mass %	448/445	41.1/42.7
PP microfibers*	448/445	39.7/50.2

*From the PP–CPA+CEVA/PES-5 mixture in a proportion of 5.0/0.5 mass %.

From Table 2 it is seen that in using compatibilizers of different chemical nature and their binary compositions the r/R ratio is practically a constant quantity and equals 1.8–1.9. This points to a common mechanism of breaking of liquid PP jets in the CPA matrix in the starting and modified mixtures.

It is known that the morphology of polymer dispersions can be controlled by increasing or decreasing the interphase tension between the components [10]. The values of σ calculated on the basis of data on the disintegration kinetics of microfibers have confirmed the decrease in the surface tension for compatibilized mixtures (Table 2). Analysis of the results indicates that binary compositions are more effective than the starting components: the introduction of the additive CEVA/PES-5 in a proportion of 1.7/0.3 mass % into the propylene–copolyamide mixture leads to a decrease in the interphase tension by about a factor of 3.5 compared to the starting CEVA or PES-5. The result obtained can be explained by the simultaneous action of both compatibilizers. Siloxane liquids are incompatible with most organic substances; even when introduced in a quantity of $\approx 10^{-4}\%$ they are displaced onto the interface and because of the low surface-tension spread over the entire surface, thus decreasing the σ value. CEVA and CPA macromolecules are able to enter into specific interactions with the formation of hydrogen bonds, which has been confirmed by the method of IR spectroscopy [2]. At the same time, CEVA has in its structure hydrocarbon segments providing PP affinity. Thus, on the interface a relation between PP and CPA macromolecules through the compatibilizer is provided. The above factors promote an enhancement of the PP fiber formation in the CPA matrix.

It is known that the compatibilization effect is a function of the additive concentration and becomes maximal under the condition of its optimal content [11]. The results of our investigations agree with this conclusion: a change in the CEVA/PES-5 ratio leads to a decrease in the interphase tension from 2.60 to 0.53 mN/m, respectively, for the starting and modified mixtures (the CEVA/PES-5 ratio is 1.7/0.3 mass %). The latter can be explained by the fact that only at a certain total content of the compatibilizers are they located on the interface and act most effectively. Upon reaching a critical quantity the additives pass into separate phases and their surface activity decreases.

Phase transitions in PP–CPA mixtures. To establish the influence of compatibilizers and their compositions on the relaxation and phase transitions in PP–CPA melts, we have carried out investigations by the method of differential scanning calorimetry. Samples of the mixtures and source polymers were heated, cooled after the melting process, and then reheated under the same conditions. The results are presented in Table 3 (on the left the data for the first heating and on the right the data for the second heating are given). It has been established that the PES-5 additives have no effect on the melting temperature T_{melt} of polypropylene but lower T_{melt} of CPA, which is explained by the formation of specific interactions on the level of hydrogen bonds between the additives and the CPA macromolecules. The same mechanism also takes place for the compatibilized PP–CPA mixture. Siloxanes produce a plasticizing effect on PP, leading to a considerable decrease in the melting heat ΔH. CPA is a low-crystalline polymer and, therefore, its melting heat is lower than in PP. The increase in ΔH upon the introduction of 0.5 mass % of PES-5 is due to the structurization of its melt in the presence of the additive. The total melting heat of PP–CPA–PES-5 mixtures in a proportion of 50/50/0.5 mass % sharply decreases compared to the binary composition, which is due to the change in the PP structure in the mixture. It is known that the crystallization rate of the dispersed-phase polymer depends on the size of its particles. It decreases with increasing dispersity due to the increase in the portion of the polymer in the transition interphase layer, which just leads to a decrease in the crystallinity. Thus, it has been established [12] that at

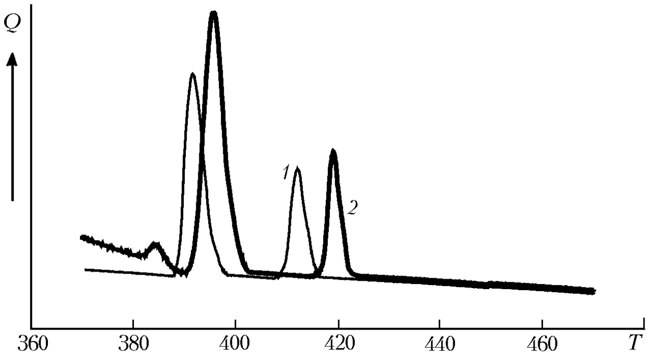

Fig. 3. Melting thermograms of extrudates of PP–CPA/CEVA/PES-5 mixtures in proportions (mass %) of: 1) 50/50/0/0; 2) 50/50/5.0/0.5. *Q*, AU; *T*, K.

TABLE 4. Influence of Compatibilizers on the Rheological Properties of the PP–CPA Mixture Melts*

Content of CEVA/PES-5 compatibilizers, mass %	η, Pa·sec	*B*	Φ_m, %
Without additives	230	3.6	3700
5.0/0	300	5.0	8700
0/0.5	190	5.0	4100
0.5/0.3	180	5.7	9300
1.0/0.3	190	6.3	11, 500
1.7/0.3	200	7.1	16, 500
5.0/0.5	390	5.1	21, 600

*At $\tau = 5.7 \cdot 10^4$ Pa.

a particle size of polyolefin (PO) in polypropylene of 0.1–0.2 μm the dispersed-phase, high-crystalline polymer (PO) remains in the amorphous state. As has been shown, the starting PP–CPA mixture has a layer morphology (see Fig. 1a). Upon the introduction of 0.5 mass % of PES-5 the polypropylene is present in extrudates in the form of fine microfibers, i.e., in the fine-grained state which is responsible for the decrease in the melting heat of the mixture by nearly one-half. The use of binary additives leads to a further decrease in the diameter of the microfibers, which just predetermines the decrease in ΔH of the four-component compositions. The crystalline structure of PP in the form of microfibers is less perfect, despite the prolonged annealing in the process of extraction of the matrix polymer. This is evidenced by the decrease in T_{melt} of PP of the microfibers and the sharp decrease in ΔH (from 81.0 to 39.7 J/kg).

The experiments performed with the use of a Du Pont 2000 DSC V4.0B calorimeter confirm the effective compatibilizing effect of PES-5 and its binary mixture with CEVA. On the crystallization thermograms of the mixtures, an additional low-temperature peak appears (Fig. 3). In our opinion, this points to an increase in the extent of the transition layer on the interface in which polypropylene is in a very fine-grained state. Its crystallization proceeds on homogeneous nuclei and the third peak pertains to that portion of PP which is located in the transition layer. The proposed hypothesis is in good agreement with the previous electron-microscopic investigations of the structure of the microfiber surface, namely with the presence of superfine fibrils throughout its surface [1]. Thus, the mechanism of action of the compatibilizers and their binary compositions is a decrease in the surface tension on the interface and the formation of a transition surface layer of sufficient extent.

Influence of binary mixtures of compatibilizers on the rheological properties of PP–CPA melts. It is known that in processing melts of polymers and their mixtures into fibers and films two basic rheological characteristics — viscosity and elasticity — are of crucial importance. These indices strongly depend on the phase morphology of polymer dispersions. The results of the investigations point to a different influence of some compatibilizers and their binary mixtures on the rheological properties of PP–CPA melts (Table 4). Upon the introduction of CEVA and PES-5, the viscosity of the mixture melt is a result of both the specific interaction between the CEVA and CPA macromolecules, which structurizes the melt and increases its viscosity, and the plasticizing action of silicons and the formation of liquid jets of the dispersed-phase polymer (microfibers) in the CPA matrix, which decreases η. The binary additives also produce a different effect on the viscosity of the mixture melt. The introduction of the composition 0.5% of CEVA + 0.3% of PES-5 provides a further decrease in η. An increase in the content of CEVA leads to an increase in the viscosity. The established mechanism is associated with the competing influence of the plasticization, the structurization of the melt, and the realization of the specific fiber formation. The results obtained point to the possibility of processing modified PP–CPA mixtures under the same conditions as for the starting ones.

The melt elasticity estimated by the swelling value B correlates with the specific fiber formation and serves as its indirect characteristic [1]. From this point of view, some compatibilizers and their mixtures enhance the formation of PP microfibers in the CPA matrix. The greatest swelling takes place when the diameter of the microfibers is minimal and their number is maximal (Tables 1, 4).

The ability of melts of the investigated compositions to be processed was determined by the value of the maximal spinneret drawing Φ_m. The results obtained indicate that the use of compatibilizers promotes the deformation of the melt in the stretching field. The PP–CPA mixture melt has the least Φ_m, which is due to the weak interaction of the components on the interface and the layer morphology. Noteworthy is the considerable improvement of the capability of the melt to be processed upon introduction of the binary composition of the compatibilizers: 1.7% of CEVA + 0.3% of PES increases Φ_m by more than a factor of 4.5 (Table 4). As we see it, this is due to both the plasticizing action of PES-5 and the enhancement of the PP fiber formation in the CPA matrix. It is known that the formation of an anisotropic structure increases the capability of the mixture melt for deformation [13].

The experiments performed have confirmed the correctness and efficiency of the chosen technique for modifying PP–CPA mixtures. The mechanism of action of binary CEVA–PES-5 compositions is an increase in the kinetic stability of dispersion and a decrease in the interphase tension, which promotes the deformation of PP drops into liquid jets. The specific fiber formation is best realized at an optimal ratio of the compatibilizers. On the basis of the investigated mixtures we have created fine-fiber polymer materials with new properties — complex filaments from PP microfibers with a higher abrasion resistance and filtering materials (FM) with a high efficiency of air cleaning from submicron welding aerosol impurities. The FMs absorb oils as effectively as do mineral sorbents and exhibit a sterilizing effect with respect to Pasteurellas and the pathogenic mechanism of malignant anthrax.

NOTATION

B, swelling value of the extrudate; d and $\bar{d}$, diameter and mean diameter of microfibers, μm; ΔH, melting heat, J/kg; q, instability coefficient; Q, heat flow, AU; R, liquid-jet radius, μm; r, radius of the dispersed-phase polymer drop, μm; T, temperature, K; T_{melt}, melting temperature, K; t_{life}, lifetime of the jet, sec; δ^2, dispersion of the diameter distribution of microfibers, μm^2; η, viscosity of the polymer mixture melt, Pa·sec; λ_m, breaking disturbance wavelength, μm; σ, surface tension on the interface, mN/m; τ, shear stress, Pa; φ, numerical portion, %; Φ_m, values of the maximum spinneret stretching, %. Subscripts: m, maximum; life, life; melt, melting.

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