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REGULARITIES OF MODIFYING, MORPHOLOGY AND PROPERTIES OF THERMOPLASTICS CONTAINING POLYVINYLPYRROLIDONE

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Abstract. The peculiarities of modifying polyamide 66/6 and polystyrene thermoplastics with polyvinylpyrrolidone in the viscous state have been studied. By applying instrumental methods, the changes in morphology and phase transitions of modified materials have been revealed. The influence of the modifier on the strength and thermophysical properties of the researched thermoplastics was determined.

Keywords: modifying, compatibility, polystyrene, polyamide 66/6, polyvinylpyrrolidone.

1. Introduction

The development of advanced technologies and modern industries requires the application of materials, which possess a complex of specific properties. Polymer materials and composites correspond to the most modern requirements, the range of which is expanding due to the modification of the polymer, which allows changing the properties purposefully.¹⁻³ The blending of two or more polymer materials is not only one of the simplest and the most technologically available methods but in some cases, it is the more efficient method of modifying polymers. By applying polymers of different chemical natures and changing the preparation conditions of the blend components, as well as the parameters of their processing, one can get products of the desired properties.⁴⁻⁶

The main factor, which prevents the widespread application of materials based on polymer blends, is the thermodynamic incompatibility of the components. This, in turn, affects the technological features of blends during their processing and the operational properties of the products based on them.^{7,8} Therefore, when creating polymer blends, first of all, it is necessary to determine the degree of components combination, which depends on the blending conditions, molecular weight, polymers ability

for reciprocal dissolution, their morphology, as well as the presence of plasticizers, fillers and other additives.^{9,10}

The considerable interest in polyvinylpyrrolidonecontaining polymer blends (PVP) as modifying additives is caused on the one hand by the great practical value of polymers with PVP chains in their structure and, on the other hand, by the specific features of their processing.¹¹ PVP is a representative of functionally active polymers, which has a number of unique properties, including selective sorption, surface activity, the ability to be combined with different compounds, and so in the literature.^{12,13}

To compare the influence of PVP on the properties of thermoplastics differed by their morphology, chemical nature, and content of functional groups in macromolecules, the polyamides (partially crystalline materials) and polystyrene (amorphous material) were chosen. The reason for such a choice is the ability of mentioned compounds to change a number of operational properties of thermoplastics in the desired direction: polyamides increase the water and vapor permeability and polystyrene reduces the ability to accumulate static electricity charges and surface functionalization; possibly it increases the impact resistance.^{14,15}

The aim of research is to determine the technological properties of the blends based on industrial thermoplastics and PVP, as well as the operational properties of the products based on them in regard with their supramolecular structure.

2. Experimental

The samples of the industrial thermoplastics used for research were as follows: PA-66/6 brand AK-60/40 TU 6-05-1032-73; PSS – production of JSC "Concern Styrene" (Gorlivka, Ukraine) and purified PVP of 28,000 molecular weight (AppliChem GmbH, Germany).

To obtain modified materials the bulk components were pre-mixed in the required ratio by applying the drum-type mixer. After obtaining a mechanical blend of bulk it was dried for 6-7 h in an air dryer of 2B-131 type at 353–363 K and relative humidity of 5–10 %. Then

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PVP-containing blends of thermoplastics were prepared by direct mixing in a screw pre-plasticizer of a KUASY 25-32/2 casting machine (casting volume up to 16 cm³) with further extrusion, grinding, and cooling of the material in the form of a rod. The temperature in the zones of the material cylinder was 463–483, 483–503, and 503– 518 K; the plasticization time was 30–35 s.

Research by DSC (differential scanning calorimetry) was conducted by applying a DSC Z20 PC (NETZSCH, Germany). The heating and cooling rates were 10 K/min. Experimental data were presented in the form of dependence of specific heat on temperature.

DMTA studies were carried out applying the DMA242C instrument (NETZSCH, Germany). Samples of polymers in the form of rectangular bars, which were subjected to sinusoidal loads of different frequencies, were used for research. The temperature diapason of the study ranged from 223 to 423 K. The heating rate was 1 K/min. The experimental data were presented as the elasticity modulus and the rate of mechanical losses depending on the temperature.

Tensile strength and elongation at break were determined according to ISO 527 using TIRA Test 2200 tensile machine.

Vicat softening point of the researched materials was defined respectively to ISO 306: 2013, and the load was 50 N.

The formation of the flow curves of melting of polymer blends was carried out on a MFI apparatus "IIRT-M" using capillaries in length 8 and 25 mm and a diameter of $2,095 \pm 0,005$ mm. The volume flow of material in the capillary was calculated by the formula:

$$Q = \frac{S \cdot \pi \cdot d^2}{4 \cdot t},\tag{1}$$

where *S* is the displacement of the piston, m; *d* is the diameter of the piston, m; *t* is the time of movement.

The effective rate of displacement in the capillary was calculated according to Eq. (2):

$$\gamma = \frac{4 \cdot Q}{\pi \cdot r^3},\tag{2}$$

where r is the radius of capillary, m.

Tensile shear in the capillary was calculated according to Eq. (3):

$$\tau = \frac{\Delta P \cdot r}{2 \cdot L} = \frac{9,81 \cdot 4 \cdot r \cdot P}{\pi \cdot d^2 \cdot 2 \cdot L},$$
(3)

where P is the weight of cargo, kg; ΔP is a pressure drop between the ends of the capillary; L is the capillary length, m.

Taking into account the correction of Rabinovich, the shear rate was determined by constructing in the logarithmic coordinates the auxiliary curve of the strain stress vs. shear rate, using the Weissenberg-Rabinovich method. By differentiating the obtained dependence, we found the index of viscosity anomaly due to the tangent of the angle of the curve inclination. Hence, the shear rate:

$$\gamma = \frac{(3 \cdot n + 1)}{4 \cdot n} \cdot \gamma_{ef}, \qquad (4)$$

where *n* is an index of viscosity anomaly (degree of non-Newtonian behavior); γ is the corrected value of the shear rate, s⁻¹.

The effective maturation viscosity was calculated as the ratio of the displacement stress to the shear rate:

$$\eta = \frac{\tau}{\gamma},\tag{5}$$

Using the rheological Eq. (6), its constants were determined:

$$\tau = m_0 \cdot exp\left(\frac{E}{RT}\right) \cdot \gamma^n, \qquad (6)$$

where τ is the displacement stress for this polymer mixture, Pa; η is a maturation viscosity, Pa·s; γ is the shear rate, s⁻¹; *n* is an anomaly of viscosity index; *E* is the imaginary activation energy of the viscous flow, J/mol; *m*₀ is a constant that depends on the nature of the material, Pa·s.

The activation energy of the viscous flow of polymers and their blends melting was determined according to Eq. (7):

$$E_a = 2,303 \cdot R \cdot lg \frac{(\eta_{T2} - \eta_{T1})}{(1/T_2 - 1/T_1)},$$
(7)

where $\hat{\eta}_{T1}$ and $\hat{\eta}_{T2}$ are the viscosities of the material at temperatures T_1 and T_2 , respectively.

3. Results and Discussion

The ability of plastics to be processed, the choice of its optimal processing method, and the calculation of process parameters are determined primarily by their technological properties. The choice of processing method and optimization of technological parameters of the process is carried out by the next characteristics of raw materials: viscosity and fluidity of the melt, particle size distribution, content of moisture and volatile matters, flowability, bulk density, and shrinkage. Accurate information about the material technological parameters also allows to adjust the modes of its processing, calculate the size of the molds, ensure high productivity and reduce the amount of raw material waste.

In the manufacture of products made of polymers blends and composites based on them, the technological properties will also depend on the component composition of materials, the components nature, and so on. Therefore, for the processing of polymer blends, it is necessary to determine the full range of quantitative values of their technological properties.¹⁶

The dependence of the shear stress of materials based on PA-66/6 on the PVP content is shown in Fig. 1

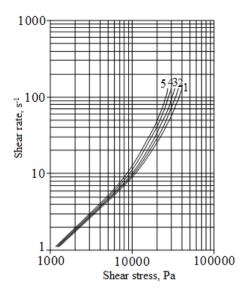


Fig. 1. Flow curves of PA-66/6 – PVP blends at a temperature of 463 K. PVP content, % wt.: 1 - 0; 2 - 1; 3 - 2.5; 4 - 5; 5 - 10

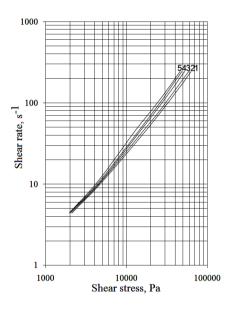


Fig. 2. Flow curves of PA-66/6 – PVP blends at a temperature of 483 K. PVP content, % wt.: 1 – 0; 2 – 1; 3 – 2.5; 4 – 5; 5 – 10

It was found that the PA-66/6 flow curves of different PVP content are similar (Fig. 1). For pure PA-66/6 the viscosity anomalies appeared at low shear rate values ($\approx 10 \text{ s}^{-1}$). This is obviously due to the peculiarities of the chemical nature of mentioned polyamide, namely, the presence of different fragments in the PA-66/6 structure and a slightly different density of intermolecular hydrogen bonds in the presence of PVP. The effective viscosity of PA-66/6-PVP blends also decreases with increasing PVP content. The appearance of viscosity anomaly at the shear rates greater than 10 s⁻¹ at the temperatures of 463 K and 483 K (Fig. 2) and in the entire range of shear rates at 503 K (Fig. 3) in the case of PA-66/6 – PVP blends is apparently due to the different nature of intermolecular interactions between PA-66/6 blocks of different chemical nature and between the PVP and PA-66/6 macromolecules.

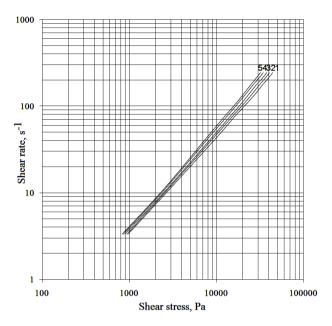


Fig. 3. Flow curves of PA-66/6 – PVP blends at a temperature of 503 K. PVP content, % wt.: 1 – 0; 2 – 1; 3 – 2.5; 4 – 5; 5 – 10

Due to different intermolecular interactions in the melts of PA-66/6 – PVP blends, areas with different densities of engagements are formed, which in turn will affect the redistribution of the specific density of the blend. With increasing temperature, the appearance of viscosity anomaly of the melts in PA-66/6 – PVP blends becomes less noticeable. The decrease in the viscosity anomaly was caused by a reduction in the number of structural processes during the melting of blends,¹⁷ with the deformation and orientation in the direction of PA-66/6 flow with PVP associates.

The study of PVP influence on the rheological characteristics of its flow with an amorphous polymer, in particular PS, was of considerable interest. As we can see (Fig. 4) from the flow curves of PS - PVP blends, there is also a decrease in their effective viscosity with increasing PVP content and their appearance is similar to the flow curves of PA-66/6 – PVP blends. However, the main feature of PS-PVP blends, in contrast to polyamide-PVP blends is that temperature changes have a slight effect on the appearance of the blends' effective viscosity depending on PVP content. We can draw a preliminary conclusion about the absence of intermolecular interactions between the components and the possibility of thermodynamic incompatibility between PS and PVP in the viscous state.

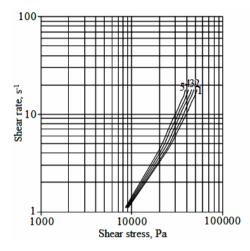


Fig. 4. Flow curves of PS – PVP blends at a temperature of 453 K. PVP content, % wt.: 1 – 0; 2 – 1; 3 – 2.5; 4 – 5; 5 – 10

Features of interfacial phenomena in polymer blends are determined primarily by the nature of the initial ingredients.^{18,19} However, most polymer pairs do not have thermodynamic compatibility. In such blends, the formation of a common crystal lattice is impossible, but we can assume the presence of compatibility between the components based on the formation of common supramolecular structures in the absence of a clear boundary of the transition layers. The nature and structure of transition layers in blends of thermodynamically incompatible polymers, mainly, have a significant impact on their technological and operational properties.

The DSC method is one of the physical and physicochemical methods of thermal analysis, which can be used to determine energy changes of different natures and morphology in polymer materials, in particular in polymer blends. It provides an opportunity to obtain sufficiently comprehensive information. This method also helps to study the correlation between kinetic parameters, characteristics of the main relaxation transitions, conformational mobility of the chain, and barriers to intermolecular interactions.

To determine the change in the supramolecular structure of polymers under PVP influence and change the parameters of phase transitions in the studied blends the DSC analysis of the obtained materials was conducted (Fig. 5).

It should be noted that the structure of PA-66/6 is described as two-phased containing blocks with different chemical structures making the different domains. When PVP is added to the mentioned system, it affects the formation of these domains in different ways. The presence of a wide range of phase transitions indicates the heterogeneity of supramolecular structures and the recrystallization processes in PA-66/6 – PVP blends. This is, first of all, caused by PVP selective interaction with flexible and rigid PA-66/6 blocks, which in turn leads to the formation of supramolecular structures of different types. In the PA-66/6 – PVP blends, in addition to structural formations formed by flexible and rigid sections of macromolecules, there are intermediate sections, the size of which is determined by the interaction of adjacent fragments of chains and the influence of PVP as an agent, which promotes general heterogeneity in the system.²⁰

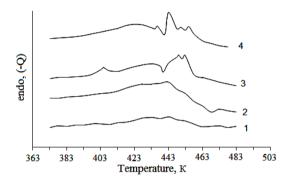


Fig. 5. DSC curves of PA-66/6 – PVP blends. PVP content, % wt.: 1 – 0; 2 – 2; 3 – 5; 4 – 10

The nature of derived DSC dependences during heating the samples obtained by injection molding from the previously obtained blends (Fig. 6) indicates that in PS - PVP blends there is no clear change in temperature of phase transitions of the initial polystyrene with the increasing PVP content.

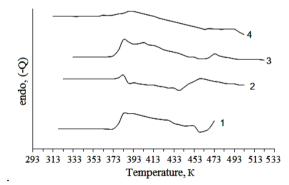


Fig. 6. DSC curves of PS – PVP blends. PVP content, % wt.: 1 - 0; 2 - 1; 3 - 2.5; 4 - 10

In the area of phase transitions of PVP-containing samples, in contrast to pure PS, anomalies of endothermic nature are manifested in the temperature range from glass transition temperature to melting temperature. With the increase of PVP content in PS-PVP blends, a decrease in the endothermic peak is observed. The above-mentioned observations indicate a decrease in the enthalpy of the studied materials and changes in the free volume because the possibility of intermolecular bonds between hydrophobic macromolecules and hydrophilic PVP is negligible and interactions can be determined by only the hydrophobic nature between the macrochains of PS and PVP. With the increase in the PVP amount in the blends containing PS there is an endothermic change in the slope of the curves within the temperature range of the T_{II} transition, which appears as a decrease in the line curvature. The T_{II} transition is proposed to be considered from the standpoint of non-equilibrium thermodynamics as a transition that combines both thermodynamic and relaxation characteristics. It is assumed that the T_{II} transition is close to the phase transition of the second kind. Therefore, a decrease in the angle of the curves from this point of view indicates a smaller number of intermolecular bonds between macromolecule segments and their destruction under the impact of thermal fluctuations.

At the same time, comparing the location of the phase transition areas during the heating and cooling of PS - PVP blend a significant difference between the values of T_{II} , which are obtained in the conditions of heating and cooling, is noticeable. This phenomenon is probably due to the effect of supercooling in the processes of melting-crystallization of the described polymer materials.

Changes in the mobility of the segments of PS – PVP blends are also confirmed by the DMTA method. The nature of the obtained dependences (Fig. 7) shows that in PVP-containing samples there is a shift towards lower temperatures of the maximum tg δ , which corresponds to the dipole-group relaxation process. We also observe the shift of the maximum of the dipole-segmental process towards higher temperatures. The value of accumulation modulus E' for PVP-containing samples increases.

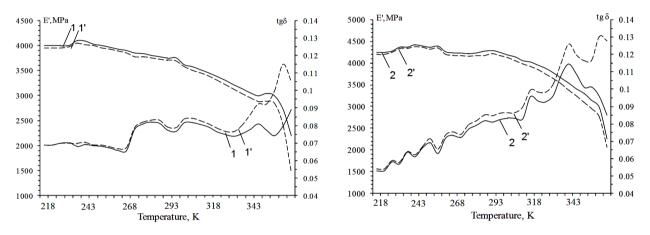


Fig. 7. Dependence of dynamic properties change of PS-PVP blends on temperature. 1, 1' - PS; 2, 2' - PS 90%/PVP 10%. 1, 2 - 10 Hz; 1', 2' - 1 Hz

The above-mentioned fact indicates the changes in the average relaxation times of the respective processes at low temperatures in the interfacial layers and a decrease in their thickness. The obtained data show that the separation limit in PS – PVP blends has different effects on relaxation mechanisms. There is mainly an increase in the mobility of polymer chain segments at the interface, which indicates a decrease in the packing density of polymers in interfacial layers.²¹ The described fact leads to an increase in the free volume of the system and an increase in microand macroheterogeneity. It should be noted that polyamide with polyvinylpyrrolidone at its high concentration, most likely, can form an interpenetrating network.²²

Changes in the morphology and phase transitions of the researched polymers will obviously determine the performance properties of the developed materials. During mechanical tests under static loads, the main tensile strength can be determined by applying the stresselongation curve (tensile curve). It is also possible to determine the destructive nature of the material and its physical state under test conditions. Tensile curves of materials with different PVP content were obtained.

Tensile curves of materials based on polyamide 66/6 - PVP blends are shown in Fig. 8.

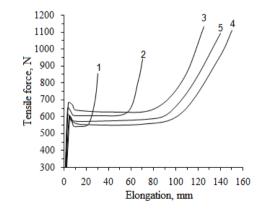


Fig. 8. Tensile curves of polyamide 66/6 – PVP blends. PVP content, % wt.: 1 - 0; 2 - 1; 3 - 2.5; 4 - 5; 5 - 10. Tensile velocity 25 mm/min

For polyamide the nature of the tensile curves is a possible reason for more defective structure of PA-66/6, low degree of crystallinity, and high values of the free volume. When the tensile stress in PA-66/6 reaches a certain value, the weakest crystalline formations begin to break down intensively under mechanical loads. This provides sufficient mobility of the macromolecule segments oriented in the direction of tension. In places of supramolecular formations transition from wide areas to thin ones, where the stress value constantly increases due to the reduction of the sample cross-section, the recrystallization process takes place. It means the destruction of some crystallites and the formation of others. Such abrupt change of crystallites from one phase to another has all the signs of a phase transition,²³ which also confirms the data of the DSC analysis.

It was found that during the "neck" formation in blends based on PA-66/6 the movement of entire sections can also occur. In this case, along with recrystallization, the initial crystallites may remain in the "neck".²⁴ Mentioned crystallites can be strongly deformed but their segments, in general, are oriented in the direction of action of the applied load. As a result of these processes, on the tensile curves of PA-66/6 – PVP blends there is a further increase in tensile stress and the values of yield strength are less than the tensile strength.

The analysis of the results within the research of polyamide – PVP blends allowed to determine such characteristics as yield strength σ_{ys} , elastic deformation ϵ_{ed} , tensile strength σ_{ts} , and tensile strength at break σ_{tsb} . The results are summarized in Table 1.

PVP content, wt. %	σ _{ys} , MPa	σ _{ts} , MPa	ε _{ed} , %	σ _{tsb} , MPa
0	14.3	20.4	60	19.5
1	15.4	22.3	140	21.6
2,5	16.1	26.9	250	26.1
5	14.5	26.4	300	25.4
10	14.2	26.1	280	24.6

Table 1. Strength characteristics of PA-66/6 – PVP polyamide blends

With increasing PVP content in the studied blends based on PA-66/6, there is an increase in strength with extremum at PVP content of 2.5 wt. %. The presence of extremum in PA-66/6-based blends is probably due to the fact that PVP interacts differently with the blocks of PA-66/6 macromolecules differing by nature.²⁵

It is known that polystyrene is an amorphous polymer, which does not crystallize. Therefore, it was interesting to research the effect of PVP on the strength of samples based on blends of amorphous PS and PVP which, as noted above, are incompatible. Tensile curves of the PS – PVP blends have the following form (see Fig. 9).

As one can see, the dependence of the tensile force on the elongation of the samples of PS – PVP blends, compared with blends based on polyamides, is somewhat different. With an increase in PVP content in polystyrene, a significant decrease in tensile strength (Table 2) and a slight increase in elongation are observed. The decrease in the value of σ_{ts} is obviously caused by

intermolecular interactions between macromolecules of PS under influence of PVP. 26

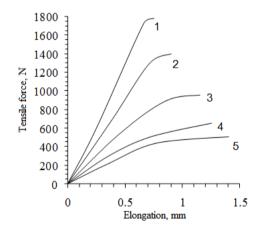


Fig. 9. Tensile curves of PS – PVP blends. PVP content, % wt.: 1-0; 2-1; 3-2.5; 4-5; 5-10. Tensile rate 25 mm/min

Indicator	PVP content,% wt.					
	0	1	2,5	5	10	
σ _{ts} , MPa	42.4	33.3	22.8	15.6	12.1	
ϵ_{ed} , %	1.5	1.8	2.3	2.5	2.8	

 Table 2. Strength characteristics of PS-PVP blends

PVP content,%	PA-66/6 – PVP			PS-PVP		
wt.	σ _{ys} , MPa	σ _{ts} , MPa	ε_{ed} , %	σ _{ts} , MPa	$\epsilon_{ed}, \%$	
0	20.9	28.4	40	45.9	1.0	
1	22.4	29.9	100	36.4	1.3	
2.5	23.1	31.4	200	24.8	2.6	
5	22.1	29.1	270	18.1	1.8	
10	20.9	29.0	240	15.2	2.0	

Table 3. Strength indicators of blends after heat treatment

Among the many methods of purposeful change of the structure and properties of finished products, especially products based on polymer blends, heat treatment (annealing) occupies an important place. The strength at break characteristics of the samples based on PA-66/6 – PVP and PS – PVP blends depending on their heat treatment are given in Table 3.

A comparison of the obtained strength characteristics of heat-treated samples, and the samples without heat treatment indicates an increase in tensile strength and yield strength during tensile; the relative elongation at break decreases. Thermal annealing has a different effect: for polystyrene-based samples an accelerated relaxation and reduction of internal stresses are observed; for polyamide recrystallization and an increase in the crystallinity degree are obvious.¹⁸

Along with the physicomechanical properties of thermoplastic – PVP blends, we studied the effect of PVP content on their thermophysical parameters, in particular the Vicat softening point (Fig. 10).

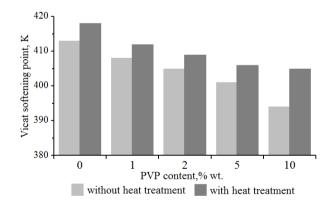


Fig. 10. Influence of PVP content on the Vicat softening point of polyamide 66/6 – PVP blends

In PA-66/6-based blends, PVP interacts with blocks of polyamide macromolecules, which differ by their chemical natures, and therefore the density and regularity of these interactions naturally will be lower. Therefore, heat resistance will be determined primarily by those blocks which interact with PVP to a lesser extent, as well as have more freedom degrees and hence – greater mobility. This feature of the interaction naturally leads to some reduction in the Vicat softening point of the developed materials.

It also should be noted that heat treatment of a PA-66/6 - PVP-based material supports the increase in their heat resistance. This is obviously due to the streamlining of the supramolecular structure of the material and increasing its crystallinity degree.

The increase in heat resistance of PS-PVP blends with increasing PVP content (Fig. 11), due to incompatibility between the components, regards probably only with the higher softening temperature of PVP, compared to PS.

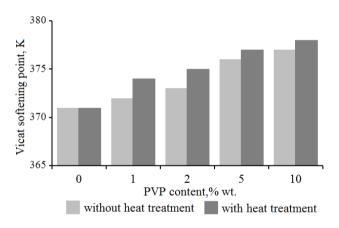


Fig. 11. Influence of PVP content on the Vicat softening point of PS-PVP blends

In addition, the preliminary orientation of the melt after mixing in the pre-plasticizer of the injection molding machine leads to a better ordering of supramolecular formations in PS - PVP blends and thus slightly increases their heat resistance.

The identified features of mixtures of industrial thermoplastics containing polyvinylpyrrolidone open the possibility of the targeted creation of composite materials of specified characteristics for a particular application.

4. Conclusions

The regularities of modification with polyvinylpyrrolidone of the industrial thermoplastics, polyamide 66/6 and polystyrene, which differ in chemical nature, fluidity and supramolecular structure, have been established.

The influence of PVP on the rheological and technological parameters of blends based on polyamide and polystyrene has been defined. It is shown that under PVP influence the effective viscosity of all blends decreases and the viscosity anomalies increase at shear rates greater than 10 s^{-1} . Using the methods of DSC and DMTA the higher compatibility between components in polyamide – PVP blends in contrast to PS – PVP blends has been determined. It was proved that PVP causes morphological changes in polyamide due to the redistribution of intermolecular interactions and helps to increase their degree of crystallinity by 5–6 %. Phase transition temperatures in PVP-containing samples based on polyamides are shifted toward areas of higher temperatures.

The influence of PVP on the strength properties of polyamide and polystyrene is defined. It is shown that with increasing PVP content in PA-66/6-based blends there is an increase in strength with extremum at PVP content of 2.5 wt. % and a significant increase in the elongation at break. With an increase in the PVP content in polystyrene, there is a significant decrease in tensile strength by 70 % and a slight increase in elongation at break from 1.5 to 2.8 %, as a result of thermodynamic incompatibility between components.

It is shown that the increase of PVP content in PA-6/66 – PVP blends leads to a decrease in the Vicat softening point due to differences in the values of crystallinity degree and the nature of supramolecular formations. The increase in heat resistance of PS-PVP blends with an increase in PVP content is associated only with a higher softening temperature of PVP compared to polystyrene.

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ЗАКОНОМІРНОСТІ МОДИФІКУВАННЯ, МОРФОЛОГІЯ ТА ВЛАСТИВОСТІ ТЕРМОПЛАСТІВ, ЩО МІСТЯТЬ ПОЛІВІНІЛПІРОЛІДОН

Анотація. Досліджено особливості модифікування термопластів поліаміду 66/6 і полістирену полівінілпіролідоном у в'язкотекучому стані. За допомогою інструментальних методів виявлено зміни морфології та фазових переходів модифікованих матеріалів. Визначено вплив модифікатора на міцність і теплофізичні властивості досліджуваних термопластів.

Ключові слова: модифікація, сумісність, полістирол, поліамід 66/6, полівінілпіролідон.