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Investigation of Polyvinyl Chloride and Thermoplastic Polyurethane Waste Blend Miscibility

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In this study the miscibility of polyvinyl chloride (PVC) and poly-ε-caprolactone based thermoplastic polyurethanes (TPU) waste blends were investigated by dilute solution viscometry. The miscibility criteria α, Δb, ΔB, and $\Delta[\eta]$ were used to assess the degree of miscibility of polymers in tetrahydrofuran solution. Also, to assess the miscibility and microstructure of PVC/TPU blends obtained by solution casting have been characterized by X-ray diffraction. The tensile strength and deformability properties varying on the blend composition were determined. It was found that PVC and TPU are partially miscible, their blend is amorphous and show two-phase structure. TPU changes the mechanical behaviour of PVC the blends. Increase of TPU content causes PVC elongation at break increase and tensile strength decreases.

Keywords: polyvinyl chloride, thermoplastic polyurethane, miscibility, dilute solution viscometry, mechanical properties.

1. INTRODUCTION

Polymer blending is a practical method for the development of new polymeric materials [1, 2]. Most products of polymer blends are designed to meet the synergistic improvements of parent components, lower cost, and improve processability [3, 4]. The main advantages of the blend systems are simple preparation and easy control of physical properties by compositional change. However, the properties of blend greatly depend upon the miscibility of homopolymers. The extent of compatibility or miscibility results in altogether different morphologies of the blends, ranging from single phase to multiphase systems. There are numerous techniques for studying the miscibility of the polymer blends, such as spectroscopy, microscopy, thermal analysis, dynamic mechanical analysis, dielectric measurements, diffraction, etc. [5 – 8]. The dilute solution viscometry method has been proven to be a simple and effective tool for investigation of the miscibility and interaction in dilute solutions of polymer blend components [2, 9 – 11].

Polyvinyl chloride (PVC) plays an important role in the plastic industry and is one of the most versatile thermoplastics, with widespread applications in different areas ranging from packing to health care devices, toys, building materials, electrical wire insulation, cloths and furnishing [12, 13]. Pure PVC is a relatively rigid and brittle, thermal and photo unstable polymer, therefore, it must be combined with a number of additives before reprocessing [14].

Polyester based thermoplastic polyurethane is engineering thermoplastic with elastomeric properties. Due to the excellent physical properties, chemical resistance, abrasion resistance, good adhesion and ease processing, its application can be found in almost all industrial branches [16, 15]. A high performance engineering polymer might be produced by blending of PVC with thermoplastic polyurethane. It was found that polyesters act as high molecular-weight polymeric plasticizers and are used in the production of nontoxic flexible PVC blends [1, 10].

Instead of phthalates additives based on poly-ε-caprolactone (PCL) are able to use in PVC formulations [3]. PVC/PCL blends present relatively good compatibility and low migration due to significantly higher molecular weight. The compatibility, morphology, thermal and mechanical properties of PVC/PCL blends have been reported in [3, 17].

Poly-ε-caprolactone-based thermoplastic polyurethanes (TPUs) are widely used for orthopaedic industry, i. e. for production of various types splints. However, splints production generates substantial quantities of waste and utilization problem of such waste arise. The possibility of recycling and secondary use of TPUs was investigated in [15].

The aim of this study was to investigate the PVC and TPU waste blends miscibility by dilute solution viscometry method. Thermal and mechanical properties as well as structure of PVC/TPUs blends obtained by solution blending were also analysed.

2. EXPERIMENTAL

2.1. Materials

The wastes from three types of low temperature poly-ε-caprolactone-based thermoplastic polyurethanes (TPU-1, TPU-2 and TPU-3 from T-Tape Company, Netherlands) were used for investigations [15]. These materials were obtained from orthopaedic device manufacturing process; they were generated at the cutting stage. All TPUs are linear segmented block copolymers, consisting of

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diisocyanate hard and poly-ε-caprolactone soft segments. The main properties of these commercially available TPUs are presented in Table 1. All TPUs used have low glass transition temperature, show similar yielding properties during drawing and close tensile strength values (about 24 MPa – 30 MPa). However, the strain at break of TPU-1 is about four-time higher than that of other thermoplastic polyurethanes.

Table 1. Main properties of used poly-ε-caprolactone-based thermoplastic polyurethanes

TPU type	T_g, ºC	Tensile properties		
		Yield point, MPa	Tensile stress, MPa	Elongation at break, %
TPU-1	60 – 65	17.6	24.1	2700
TPU-2	70 – 75	16.0	29.6	685
TPU-3	60 – 65	17.8	27.2	724

PVC resin Polanvil S-70 has been produced by Anwil Group, Poland. The main characteristics of PVC resin are presented in Table 2.

Table 2. The main characteristics of PVC resin

Property	Value
Reduced viscosity, ml/g	122 ±4
K-value	69.5 ±1
Apparent density, g/ml	0.42
Mechanical impurities, %	6
Volatile matter, %	0.2
Plasticizer absorption, phr	32
Residual chlorethylene content, mg/kg	0.1
Thermal stability, s	106

Tetrahydrofuran (THF) from PENTA (Czech Republic) was used as a common solvent for PVC and TPU samples.

2.2. Blends preparation for tensile tests

TPU content in blend composition was varied from 5 wt% up to 40 wt%. Initially TPU was masticated by 1200 s on the open mill PD 320 with friction of 1.25. After that it was dissolved together with PVC resin in fast-moving mixer up to homogenous solution for 4 h. Obtained PVC/TPU solutions in THF with solid content of 10 % were poured on a polytetrafluorethylene plate and dried until total solvent evaporation.

2.3. Testing methods

Viscometric investigations. To analyse polymer–polymer miscibility the dilute-solution viscometry (DSV) method was used. Viscometric measurements were carried out at 24 ºC temperature using an Ubbelohde-type capillary viscometer VPZ-2 (Russia) with capillary diameter of 0.56 mm. Efflux time of the THF solvent was about 65 s. The effluence time of investigated solution was determined.

Initial concentration of tested polymers (ratio 1 : 1) solutions in THF solvent 0.6 g/100 ml was selected. Five dilutions were made for each system by adding the appropriate aliquots of THF solvent, respectively, 2/3, 1/2, 1/3, 1/4, and 1/5 of initial concentration.

The specific viscosity η_{sp} was calculated from the efflux time measurements. The intrinsic viscosity $[\eta]$ was determined by the extrapolation to infinite dilution (zero solute concentration) of Huggins plot. Miscibility by the calculated Δb, $\Delta[\eta]$, α, ΔB criteria were determined.

Basically, the dilute-solution viscometry is based on the well-known classical Huggins' equation that expresses the specific viscosity η_{sp} of the polymer as a function of the concentration c, when one of the components is alone in the solution [5, 18]:

$$\eta_{sp} = [\eta]c + bc^2 , \tag{1}$$

where $[\eta]$ is intrinsic viscosity, b is the interaction term and the slope of η_{sp}/c versus c plot. Parameter b is related to Huggins viscosity constant k by expression:

$$b = k[\eta]^2 . \tag{2}$$

An analogue of Eq. (1) proposed by Krigbaum and Wall can be applied to polymer 1–polymer 2–solvent ternary system:

$$\eta_{spm} = [\eta]_m (c_1 + c_2) + b_m (c_1 + c_2)^2 , \tag{3}$$

where subscripts 1 and 2 correspond to the polymer 1 and polymer 2, respectively; b_m is related to the Huggins constant k_m by

$$b_m = k_m [\eta]_m^2 , \tag{4}$$

where $[\eta]_m$ is the intrinsic viscosity of the polymer blend and can be obtained from

$$[\eta]_m = [\eta]_1 w_1 + [\eta]_2 w_2 , \tag{5}$$

where w_1 and w_2 are the weight fractions of polymer 1 and polymer 2, respectively: $w_i = c_i / c_m$, i = 1,2 in the polymer blend. Combining Eq. (3) and Eq. (5) the following equation for polymer blend was obtained:

$$\frac{\eta_{spm}}{c} = [\eta]_1 w_1 + [\eta]_2 w_2 + b_{11} c_1^2 + 2 b_{12} c_1 c_2 + b_{22} c_2^2 . \tag{6}$$

Compare Eq. (5) with Eq. (1) it is evident that term b for the polymer 1 and polymer 2 blend is given by:

$$b_m = w_1^2 b_{11} + 2 w_1 w_2 b_{12} + w_2^2 b_{22} . \tag{7}$$

Here,

$$b_m = k_m [\eta]_m^2 , \tag{8}$$

$$b_{11} = k_1 [\eta]_1^2 , \tag{9}$$

$$b_{22} = k_2 [\eta]_2^2 , \tag{10}$$

$$b_{12} = k_{12} [\eta]_1 [\eta]_2 , \tag{11}$$

where b_{12} represents mutual interaction between the polymer 1 and polymer 2. In the ideal mixture b_{12} is related to b_{11} and b_{22}. In the binary system polymer-solvent it is obtained by the equation:

$$b_{12}^{id} = \sqrt{b_{11} b_{22}} . \tag{12}$$

Eq. (12) cannot be satisfied if specific molecular interaction between polymer 1 and polymer 2 existed. Then, compatibility of polymer blends can be characterized by a parameter Δb:

$$\Delta b = b_{12}^{ex} - \sqrt{b_{11} b_{22}}\ , \tag{13}$$

where b_{12}^{ex} is experimentally determined using Eq. (7). Negative values of Δb are found for solutions of systems with incompatible polymers, while positive values refer to attractive interaction and blend components compatibility.

Based on complete empirical equation developed by Catsiff and Hewett, Williamson and Wright suggest that the interaction coefficient between the two polymers b_{12} can be calculated as follows:

$$b_{12}^{id} = \frac{b_{11} + b_{22}}{2}. \tag{14}$$

Chee proposed that parameter ΔB, obtained as [5]

$$\Delta B = b_{12}^{ex} - \frac{b_{11} + b_{22}}{2}, \tag{15}$$

can be employed to predict polymer-polymer compatibility, also. In this case $\Delta B \geq 0$ indicates compatibility, whereas $\Delta B < 0$ shows incompatibility of blend components.

Sun, Wang and Feng proposed the criterion α of polymer-polymer miscibility evaluation:

$$\alpha = k_m - k_{m1}, \tag{16}$$

where k_m is Huggins' constant for the mixed polymer system and k_{m1} corresponds to the long range hydrodynamic interaction of pairs of molecules that has been defined as:

$$k_{m1} = \frac{k_1 w_1^2 [\eta]_1^2 + 2\sqrt{k_1 k_2}\, w_1 w_2 [\eta]_1 [\eta]_2 + k_2 w_2^2 [\eta]_2^2}{\left(w_1 [\eta]_1 + w_2 [\eta]_2\right)^2}. \tag{17}$$

The sign of parameter α can be used to predict the miscibility of polymer blends: when $\alpha \geq 0$ – polymers blend is miscible, while $\alpha < 0$ – immiscible. In addition, α can be used to indicate the nature and strength of the molecular interaction; in the case of $\alpha > 0$ dominates attraction, at $\alpha < 0$ – repulsion, while at $\alpha \approx 0$ no interaction exist.

The difference between the values of experimental intrinisc viscosity $[\eta]_{exp}$ and ideal one $[\eta]_{id}$ calculated by

$$\Delta[\eta] = [\eta]_{exp} - [\eta]_{id} \tag{18}$$

can be used as compatibility criterion, too. In this case polymers blend is miscible when $\Delta[\eta] \geq 0$ and immiscible when $\Delta[\eta] < 0$.

Kulshreshtha et. al. [19] proposed a "crossover" criterion, i. e. a crossover in the plot of η_{spm}/c versus concentration c signifies incompatibility, because phase separation was formed with revelsal of phases at intermediate compositions.

X-ray diffraction. Wide angle XRD (WAXRD) analysis of materials was performed using diffractometer DRON-6 equipped with a copper target (λ = 1.54 Å) and flat diffracted beam pyrolitic graphite monochromator. Diffraction patterns were recorded at 35 kV and 20 mA. Scanning was carried out in the 2 range 2° = 70° at the step size of 0.02° and counting time of 0.5 s/step.

Mechanical properties. Mechanical properties at tension were determined using an universal testing machine FP 10/1 (Germany) according to the requirements of LST ISO EN 8256 standard. The sample gauge length between clamps was 50 mm and the stretching speed was 50 mm/min.

3. RESULTS AND DISCUSSION

DSV method was applied to analyse PVC/TPU waste blend polymers miscibility in solutions. The plots of η_{sp}/c versus c for various PCL/TPU blends and individual polymers are presented in Fig. 1. The graphs are linear and the points are fitted to the straight line very well. It can be found that in the plots of PVC/TPU-1 and PVC/TPU-2 no crossovers of curves are observed. Therefore, according to Kulshreshtha's criterion it should be concluded that these two ternary systems are compatible. On the other hand, the crossover can be seen in the case of PVC/TPU-3 blend, showing that phase separation occurs in this blend.

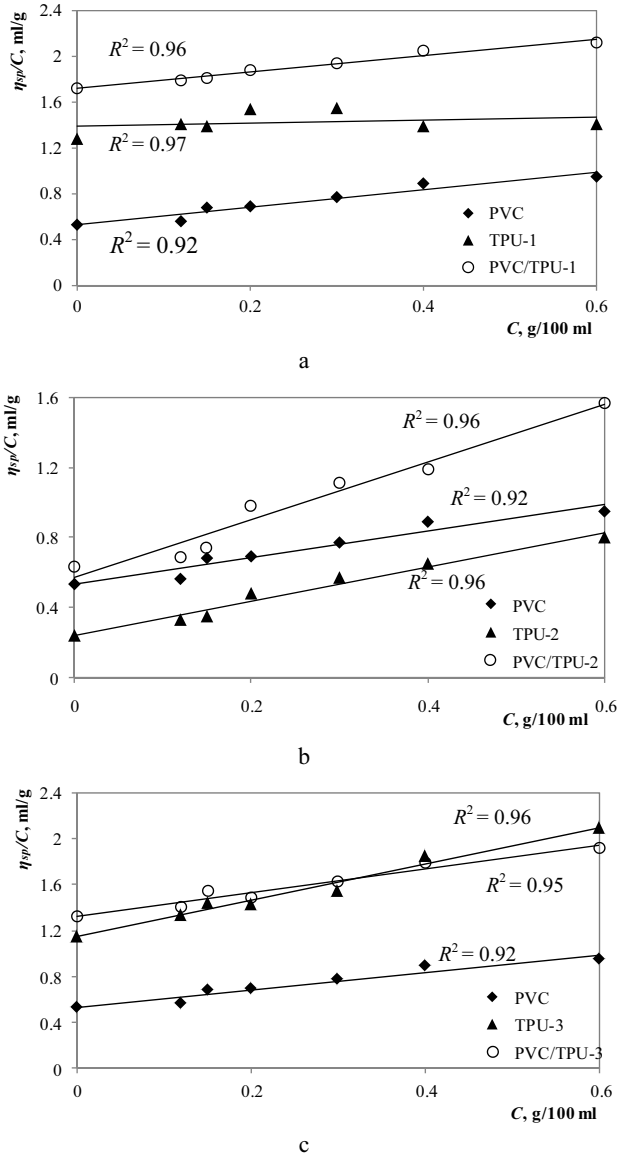

Fig. 1. Dependence of reduced viscosity upon concentration in THF of PVC, TPU, PVC/TPU systems: a – PVC/TPU-1, b – PVC/TPU-2, c – PVC/TPU-3

The intercept of the line with the axis of reduced viscosity η_{sp}/c vs. concentration c corresponds the values of intrinsic viscosities $[\eta]$ of the tested material. As

follows from Fig. 1, all TPUs increase the intrinsic viscosity of blends indicating attraction forces between PVC and TPU polymers [20]. As a result, the polymers chains expand in size and take the high volume in solution. It is evident that intrinsic viscosity of the investigated blends ($[\eta]_{PVC/TPU-1}$ = 1.72 ml/g, $[\eta]_{PVC/TPU-2}$ = 0.63 ml/g, $[\eta]_{PVC/TPU-3}$ = 1.32 ml/g) are slightly higher compared to that of pure PVC resin ($[\eta]_{PVC}$ = 0.53 ml/g) and TPU polymers ($[\eta]_{TPU-1}$ = 1.28 ml/g, $[\eta]_{TPU-2}$ = 0.24 ml/g, $[\eta]_{TPU-3}$ = 1.15 ml/g).

The increase of $[\eta]$ value shows strong interaction between blend constituents. In this case the specific interaction, such as van der Waals, may exist between polymers macromolecules [21 – 24]. On the other hand, the increase of intrinsic viscosity $[\eta]$ of the PVC/TPU blends indicate not so much strong interaction between PVC and TPU polymer as for one of polymers with the solvent [25, 26].

For the more detailed clarification of PVC and TPU polymers miscibility the criteria $\Delta[\eta]$, Δb, α, and ΔB were calculated. Values of these criteria are listed in Table 3. As can be seen, all PVC/TPU blends show positive values of $\Delta[\eta]$. From this point of view, the blend may be considered as miscible. The larger value of $\Delta[\eta]$, the stronger intermolecular interaction in solution will be. According to this, the strongest intermolecular interactions are observed for system PVC/TPU-1 blend, while weakest for PVC/TPU-2 one.

Table 3. Miscibility criteria for PVC/TPU blends

Composition	$\Delta[\eta]$, ml/g	Δb, $(ml/g)^2$	α	ΔB, $(ml/g)^2$
PVC/TPU-1	0.82	–12.57	–37.54	–12.92
PVC/TPU-2	0.15	42.23	–48.64	42.03
PVC/TPU-3	0.48	–1.11	–38.73	–2.5

However, these results disagree with other criteria. The parameters Δb, α, and ΔB for PVC/TPU-1 and PVC/TPU-3 have negative values, suggesting that these blend may exhibit phase separation and, hence, immiscibility. Moreover, negative values of criteria Δb, α, and ΔB indicate repulsive forces between blend components [24, 26]. Meanwhile, in the case of PVC/TPU-2 blend only α criteria has negative values, while positive Δb and ΔB values show that attractive forces between blend components dominate. Such unconformity of miscibility parameters can occur due to the very strong interaction between blend polymers [10].

Thus, obtained results show that miscibility of PVC and TPU blends depend on the criteria, which were chosen for the determination of compatibility. PVC/TPU-2 blend mostly coincided miscibility criteria, therefore, it was chosen for the further investigations. As it can be seen from Table 1, this waste shows highest glass transition temperature and strength properties.

WAXRD was used to study the microstructure of the PVC blend with TPU-2. Fig. 2 shows obtained diffractograms for PVC, TPU-2 and their blends of various compositions. As can be seen, no peak within 2θ range of 3°– 70° was observed in PVC that is characteristic for amorphous polymers. If there are any crystalline derivatives they are very small and sparsely.

On the contrary, TPU-2 diffractogram shows relative sharp peaks at positions close to the orthorhombic crystals of soft poly-ε-caprolactone segments reflections at 2θ = 21.3º and 23.6º, and amorphous halo under the crystalline reflections. The ratio of crystalline and amorphous reflections indicate highly crystalline polymer. However, TPU blends with PVC characterize a predominantly amorphous structure with small crystalline or quasi-crystalline fractions and no peaks pertaining to TPU-2 in tested PVC blends (Fig. 2). It is associated with formation of homogenous blends. The addition of the crystallisable TPU-2 component to the amorphous PVC changes the blends mobility and free energy of the nucleation, resulting of the specific interaction between the carbonyl groups of PCL and the hydrogen groups of PVC, indicated miscibility of blend components.

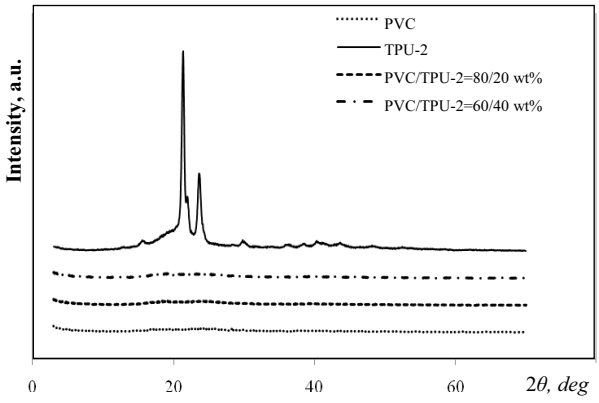

Fig. 2. WAXRD diffractograms of pure PVC, TPU-2 and their blends

The stress-strain diagrams of PVC upon TPU-2 content are presented in Fig. 3.

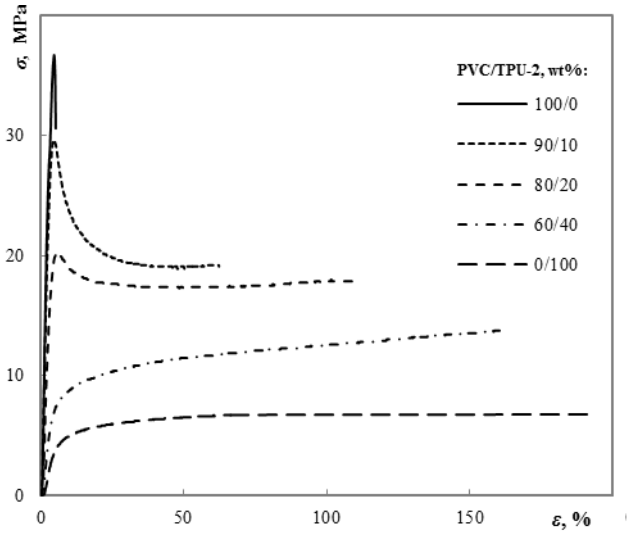

Fig. 3. Stress-strain curves of PVC/TPU-2 films upon blend composition

It can be observed that addition of TPU-2 into PVC composition significantly influences the mechanical behaviour of blend. Pure unplasticized PVC and its blends with TPU-2, which content is not higher that 20 wt%, behave like brittle plastics. However, further increase of the

TPU-2 content changes blend behaviour under tension. It is evident that PVC/TPU-2 = 60/40 wt% blend becomes tough and does not exhibit yield point. It shows that thermoplastic polyurethane TPU-2 is miscible with PVC and acts as the plasticizer in respect of PVC. Increase of TPU-2 content in the PVC/TPU-2 blend composition decreases rigidity and significantly increases deformability.

The strength and deformation properties of the PVC/TPU-2 blends upon TPU-2 content are presented in Fig. 4. The decrease of yield strength from 40.5 MPa down to 20.3 MPa when TPU-2 content increases up to 20 wt% was accompanied by decrease of tensile strength about 3 times (from 37.9 MPa to 13.8 MPa). Such behaviour could be explained by the concentration effect of the TPU-2 content in PVC/TPU-2 composition. TPU-2 forms the dispersed phase in the blends and disrupted the dipolar interaction of PVC that results on the decrease of blends strength [27]. On the other hand, the elongation at break of PVC/TPU-2 blends significantly increases from 4.5 % up to 163.8 % as TPU-2 content increases up to 40 wt%. Such change of mechanical properties confirm proposition that TPU-2 acts like the plasticizer for PVC, decreasing tensile strength and at the same time markedly improving deformability.

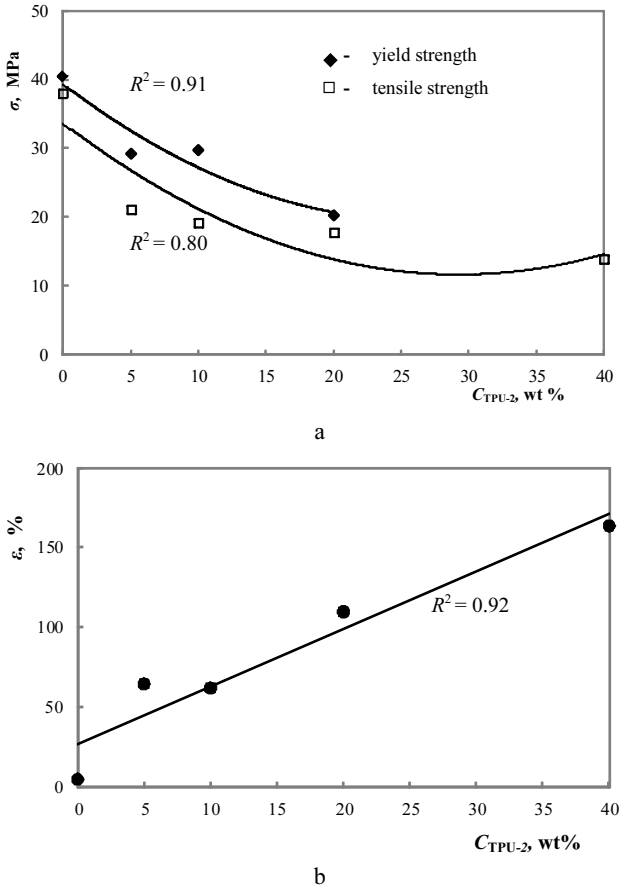

Fig. 4. Influence of TPU-2 content on PVC mechanical properties: a – strength properties; b – elongation at break

4. CONCLUSIONS

Miscibility and mechanical properties of polyvinyl chloride (PVC) and poly-ε-caprolactone-based thermoplastic polyurethane (TPU) waste blends were analysed. According to the calculated miscibility criteria it was found that miscibility of PVC and TPU depends on waste type. The best miscibility with PVC show TPU waste with highest glass transition temperature and strength properties.

PVC blends with TPU (20 – 40 wt%) show predominantly amorphous structure with small crystalline or quasi-crystalline fractions.

TPU acts like the plasticizer for PVC. It forms the dispersed phase in the blends and disrupted the dipolar interaction of PVC. Therefore, increase of TPU content results on the decrease of blends rigidity, strength and increase of the deformability.

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