

Rheology, Morphology and Mechanical Properties for Mixtures of Multicomponent Waste from Polymer Composites as Secondary Raw Materials

Serhii PRYSTYNSKYI^{1,2}, Viktoriia PLAVAN^{1*}, Roman SHULIAK²,
Oleksandr SLIEPTSOV¹

¹ Kyiv National University of Technologies and Design, 2, Mala Shyianovska Street, Kyiv 01011, Ukraine

² Kostal Ukraine Ltd, 2, Pokrovska Street, Pereiaslav 08400, Ukraine

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The article presents a study of the rheological properties and morphology of a mixture of multicomponent waste polymer compositions of polyamide 6 (PA6GF30) and polycarbonate (PC) from automotive parts. A comparative analysis of the melt flow rate of the obtained mixture was carried out depending on the content of its components. The compatibility of the components in the mixture and their distribution in the obtained polymer composition were studied. The effect of the composition of the secondary multicomponent mixture on the physical and mechanical properties is demonstrated. The possibility of reusing multicomponent polymer waste with the predicted main parameters of the technological process of injection molding is shown.

Keywords: multicomponent polymer waste, polyamide 6, polycarbonate, injection molding, secondary processing.

1. INTRODUCTION

Plastic products obtained from polymer materials by injection molding, extrusion, or 3D printing have unique properties that ensure their effective performance characteristics and cost-effective production. The ability of polymer materials to be modified makes it possible to produce products of lower weight with the properties of metals while using simpler and more efficient processing methods. Moreover, it has been noted that some polymer composite materials have several advantages over metal components, particularly in thermal conductivity [1]. Polymer composite materials account for almost half of the weight of the modern Boeing 787 Dreamliner [2], which demonstrates the unique capabilities of plastics to meet the most stringent requirements along with a high level of financial efficiency.

Thanks to their unique properties, polymer materials are widely used in the automotive industry as parts of almost all components and mechanisms, replacing relatively heavy metal parts. The increasing use of plastics in the car structure is justified by the fact that it reduces the unladen weight of the car, reduces fuel consumption and wear of parts, and, accordingly, increases the carrying capacity. Usually, the problem of increasing the durability of parts and assemblies due to the corrosion resistance of plastics is solved, the level of operational noise is reduced, etc. [3].

Polymers can solve complex modern problems thanks to combinations of materials with different chemical groups in the same product [4, 5]. However, it is difficult to imagine the technological processes of polymer processing and the exploitation of finished products without the generation of waste, which consists of defective products, damaged elements during use and technological losses. That is why

the recycling of polymer composite materials remains an urgent task.

Today, every company that manufactures parts and finished products from polymer composite materials is faced with the problem of disposal or reuse of waste, especially multicomponent waste. This is because separation is an expensive process and in most cases is not financially viable, which complicates the process of reusing polymer composite materials.

Even though there are many technologies for the processing of polymer composite materials, injection molding is still widely used [6] to obtain various parts of any complexity from both original polymers and secondary raw materials. European Union (EU) directive already requires car manufacturers to recycle 95 % of waste since 2015, automotive industry companies are facing new challenges related to the possibility of reusing polymers without losing their main properties after recycling plastics by injection molding [7]. In previous studies [8], it was shown the possibility for processing a mixture of waste of multicomponent polymer compositions polyamide 6 (PA6GF30) and polycarbonate (PC) (90/10 wt.%) and the influence of the components on the main parameters of injection molding with the validation of the process within an assessment of the stability of the process itself and the molding machine.

Nevertheless, taking into account that mixtures of polymer wastes can differ in the content of plastics, an important factor in the process of their processing by injection molding is the rheological properties, which have a significant impact on the capability of the technological process and, as a consequence, on the product. Special attention is paid to studying the rheological behavior of the melt of filled polymers with different content of such fillers

*Corresponding author. Tel.: +380-67-2504985; fax: +380-44-2800512.
E-mail: plavan.vp@kmutd.edu.ua (V. Plavan)

as metals, glass, and other organic and inorganic additives [9, 10]. Among the most widely used fillers included in polymer compositions is glass. This material is used in the form of fibers or spheres, which can change the rheological properties and it has an impact not only on the physical and mechanical characteristics but also on the parameters of the technological process of injection molding [11]. That is why the measurement of melt flow of polymer compositions is widely used, which contributes to optimizing technological processes of plastic processing [12]. The rheological properties of not only individual compositions but also mixtures are actively studied, using various research methods [13]. Studying the fluidity properties of secondary polymer compositions (re-granulates or ground-up waste) is equally important because the issue of reusing plastics is becoming increasingly popular.

Mixing polymers is one of the most economical and attractive methods of obtaining new materials with excellent properties. However, most polymers are immiscible, and their mixtures often have a phase-separated morphology, which can be a discrete phase structure (droplets in the matrix) or a co-continuous phase structure depending on the composition and processing conditions [14]. Studies of both rheology and morphology are important for understanding the properties of the obtained mixtures [15] for further evaluation of their influence on the physical and mechanical characteristics of materials [16] and the quality of finished products as a whole. In particular, Younggon Son & Shichoon Lee [17] investigated the compatibility of polyketone/polyamide 6 polymer mixtures to expand the scope of their use, since compatibility can play a key role in the processing of the obtained mixtures. The morphology of the mixture showed good interfacial adhesion. This contributes to improving the impact strength of the polyketone/polyamide 6 mixture and expands the possibilities for practical application.

At the stage of polymer mixtures processing, the supramolecular structure of polymer composite materials is formed under the influence of temperature and pressure, depending on the nature of the initial components of the composite and their mixing parameters, interphase interactions at the filler-polymer matrix interface. It is possible to estimate the phase distribution of components in the mixture, establish factors that will affect the technological compatibility of components in polymer composites, and predict their operational characteristics by studying the peculiarities of the morphology of polymer materials [18].

It is worth noting that despite the availability of modern processing technologies, multicomponent polymer composite materials are still not sufficiently used as secondary raw materials due to the availability of limited data on their processing capabilities. The behavior of a mixture of multicomponent materials has not been sufficiently studied, which determines the need to conduct complex studies of the morphology and rheological properties of multicomponent polymer materials obtained from the waste of the main production process.

Table 2. The main parameters for determining the melt flow rate (MVR)

No	T_{melt} , °C	Loading, kg	Number of slices, units	Cut interval, s	Nozzle height, mm	Load weight, g	D capillary, mm
1	300.0	5.0	10	1.5	8.0	4.0	2.095

The purpose of the research is to select and justify the technological parameters for the reprocessing of the PA6GF30/PC polymer mixture obtained from the technological waste of injection molding to reduce the cost of finished products and improve the environment.

2. EXPERIMENTAL DETAILS

2.1. Materials

In the experiment, defective parts (Fig. 1 a) and technological waste of the PA6GF30 and PC component from automotive parts were ground on a Rapid 200 grinder (Rapid Granulator AB, Sweden). The obtained mixture (Fig. 1 b) with PA6GF30 (90 wt.%) and PC (10 wt.%) was separated into components by sorting. The black element is molded from the glass-filled material PA6GF30. The white element is obtained from PC.

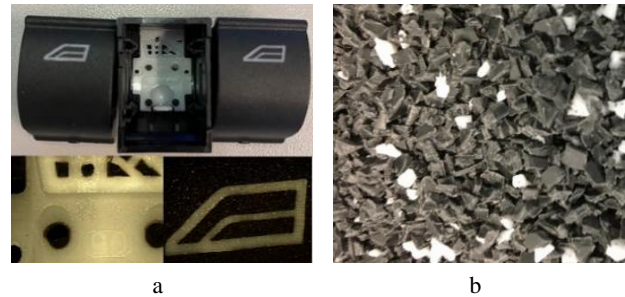


Fig. 1. General view of the material: a – two-component polymer part; b – the ground-up material

The main information about the polymer materials used in the study is presented in Table 1.

Table 1. The main data of polymer materials used in the research

Components	PA6GF30	PC
Properties		
Trade name	Durethan BKV 30 H 3.0 black	Makrolon 2405 white
Chemical group	polyamide 6	polycarbonate
Country of origin	Germany	Germany
Manufacturer	Lanxess AG	Covestro AG
Melt temperature (recommended by the manufacturer)	270-290°C	280°C
MVR, cm ³ /10 min	57.68	60.87

The melt flow rate (melt volume-flow rate (MVR), cm³/10 min was measured using the Melt Flow Indexer MFI-1322 (AMSE S.r.l., Italy) with data processing according to ISO 1133 (Table 2).

2.2. Methods

The melt temperature (Table 1) was chosen based on the recommendations of the material manufacturer (Table 2) and in such a way as to obtain an acceptable tolerance ($\pm 5\%$) for both polymer compositions in the mixtures.

The difference in the processing temperatures of PA6GF30 and PC is 2 % or 5 °C, which made it possible to choose the optimal melt temperature for the obtained mixtures.

The ratio of components in the mixture was changed in steps of 10 wt.% (Fig. 2). The morphology was studied microscopically using a Mitutoyo QS250Z optical microscope (MITUTOYO, Japan).

To determine the properties of any mixture, the rule of mixtures [19] is often used, which was employed to calculate the melt flow index of two-component polymer mixtures, and can be represented in general by the Eq. 1:

$$\text{MVR}_{i+j} = \text{MVR}_i \cdot \omega_i + \text{MVR}_j \cdot \omega_j, \quad (1)$$

where ω_i is the mass fraction of the i -component, ω_j is the mass fraction of the j -component, at the same time $i+j=1$; MVR_i is the value of the melt flow index for the i -component; MVR_j is the value of the melt flow index for the j -component. The calculation results are shown in Fig. 2.

To define the density (ρ , g/cm³) of samples from polymer mixtures PA6GF30/PC, it is necessary to determine their mass in the isopropyl alcohol and air-dry mass. Then, it is calculated according to the Eq. 2.

$$\rho = \frac{m_1}{m_1 - m_2} \cdot \rho_{alc}, \quad (2)$$

where m_1 is the air-dry mass of the sample, g; m_2 is the mass of sample in isopropyl alcohol, g; ρ_{alc} is the density of isopropyl alcohol (0.786 g/cm³).

Evaluation of the mechanical properties of the PA6GF30/PC polymer mixtures was performed using a compression-testing machine EDZ-40 (Germany) via definition compressive strength limit $\sigma_{comp.st}$ (MPa) by the Eq. 3 according to ISO 604:2002 Plastics – Determination of Compressive Properties:

$$\sigma_{comp.st} = \frac{F_{comp.st}}{A_{c.s.}}, \quad (3)$$

where $F_{comp.st}$ is the load force that corresponds to compressive strength, N; $A_{c.s.}$ is the cross-sectional area of the sample, mm.

3. RESULTS AND DISCUSSION

In the process of researching the rheological properties of the obtained mixtures, changes in the behavior of the melt were detected depending on the content of PA6GF30 and PC wt.% (Fig. 2).

It was established that with a gradual increase in the content of PC in the PA6GF30/PC mixtures, the melt flow rate increases until the ratio of PA6GF30/PC is 80/20wt.% in the mixture. With a further increase in the content of PC in the mixture, the melt flow index decreases until the ratio of PA6GF30 and PC reaches 60/40 wt.%.

From the data given in Fig. 2, it can be concluded that the melt flow rate of the PAGF30/PC mixtures, calculated according to the rule of mixtures, increases linearly as the polycarbonate content in the mixture increases. Such different regularities in the change in melt flow index may indicate that during the mixing of two polymers of different chemical natures, there is a mutual influence of the components of the mixture. In this case, a partial chemical reaction occurs, which is accompanied by a change in the

molecular structure or formation of supramolecular structures. The classic method of mixing the immiscible polymers is the addition of a block or graft copolymer or its in-situ formation during melt mixing [20]. Naturally, during the mixing of PA and PC in the melt at certain ratios, their copolymerization occurs, and the resulting product has a higher molecular weight, which is evidenced by an increase in the MVR of the mixture. In addition, nanoparticles of mineral fillers are used for the compatibility of immiscible polymer mixtures [20]. The localization of nanoparticles at the polymer-polymer interface is the key to success. The PA6 glass filler particles may act as a compatibiliser.

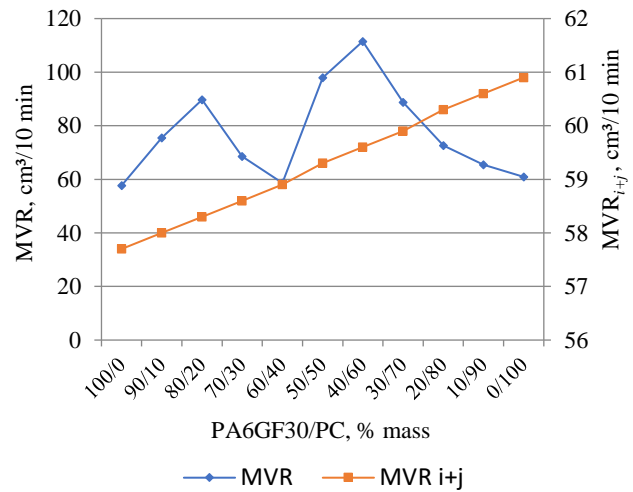


Fig. 2. Dynamics of changes in the MVR melt flow index depending on the content of the components of the PA6GF30/PC mixture

Therefore, with an increase in the content of the PC, the content of glass-filled PA decreases, and the content of the filler in the system as a compatibilizer also decreases, so the properties of the PC prevail. This is confirmed by a decrease in the MVR index of the mixture, and at the same time, the phenomenon of polyamide encapsulation in the polycarbonate structure takes place. Confirmation of this was found because of research on the morphology of mixtures.

Physico-mechanical properties of polymer blends of different compositions obtained from waste mixtures were evaluated by changes in density and compressive strength (Fig. 3). As can be seen from the data in Fig. 3 as the content of polycarbonate in the composition increases, its density decreases; starting from 60/40 wt.% the density of the composition becomes lower than the density of polycarbonate and polyamide separately. A decrease in density may indicate a decrease in the degree of crystallinity of the polymer composition [21].

This can occur as a result of the formation of long-chain polymers because of a high degree of polymerization or via the formation of co-polymers by block copolymerization, which does not lead to an increase in the degree of crystallinity. Thus, our previous assumption is confirmed by the statement that during the mixing of PA and PC in the melt in certain ratios, their copolymerization occurs. The resulting product has a higher molecular weight, which is evidenced by an increase in the MVR of the mixture. The degree of crystallinity also affects the elastic-plastic

properties of the polymer composition. In particular, an increase in polymer crystallinity causes an increase in its strength, which is sometimes accompanied by the appearance of brittleness, and the amorphous part of the polymer is responsible for its plasticity [22].

A crystalline polymer would be too brittle to be used as plastic. Amorphous regions give the polymer the ability to bend without breaking and absorb impact energy, i.e. sufficient strength for practical use. The results of determining the compressive strength limit of the samples fully confirm this.

As can be seen from the given data (Fig. 3), as the polyamide content in the polymer composition decreases, the compressive strength decreases as well.

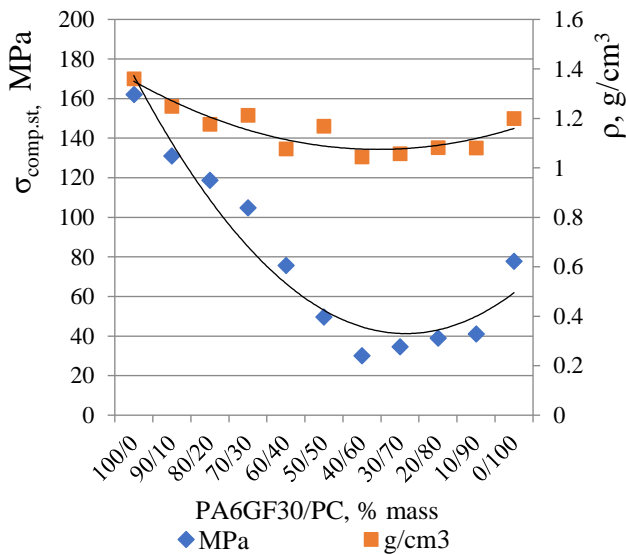


Fig. 3. The results of determining the physical and mechanical characteristics of samples of polymer mixtures depending on their composition

This is consistent with the previously expressed assumption that because of partial chemical interaction, copolymerization of polyamide and polycarbonate can occur, which is accompanied by a decrease in the degree of polymer composition material's crystallinity and is manifested by a reduction in density. With a predominant content of PC in the polymer composition, the influence of PA is neutralized due to the phenomenon of encapsulation of PA in the structure of PC. It is clearly visible during the study of the morphology of the mixtures. The mixture acquires the characteristics of PC, which is accompanied by a gradual increase in the compressive strength limit.

Fig. 4 shows the distribution of materials in the obtained extrudate. Glass-filled polyamide PA6GF30 has a dark color, polycarbonate has a light color.

In the mixture PA6GF30/PC 90/10 wt.% and 80/20 wt.% (Fig. 4 a and b) a uniform distribution of PS in PA6 and the absence of clear boundaries between the components, as well as the absence of cavities, are observed.

Spherical areas of different sizes, similar to cavities, are visualized in the mixtures with a ratio of PA6GF30/PC 70/30 wt.% and 60/40 wt.% (Fig. 4 c and d). During the further addition of PC to PA6GF30 starting from the PA6GF30/PC ratio of 50/50 wt.% and up to 10/90 wt.% (Fig. 4 e–i), spherical areas of different sizes, similar to

cavities, with clear contours are observed. Perhaps part of the polyamide is encapsulated in the volume of polycarbonate along with the presence of cavities.

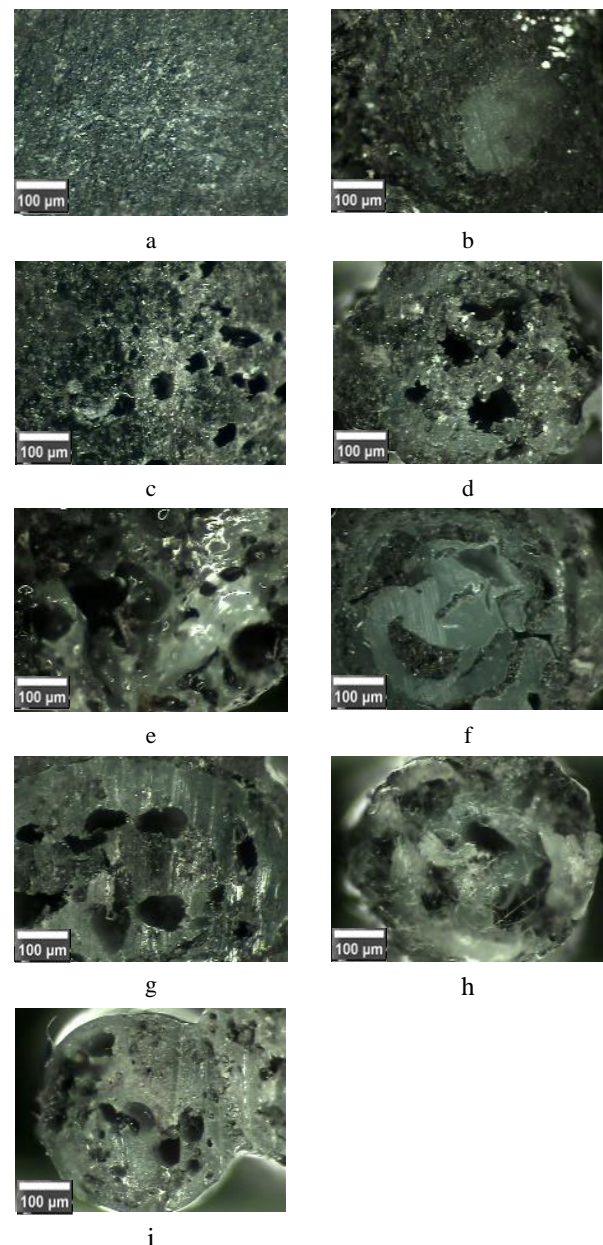


Fig. 4. Microphotographs of the cross-section of the extrudate after the study of the melt flow index from PA6GF30/PC in the ratios, wt.%: a–90/10; b–80/20; c–70/30; d–60/40; e–50/50; f–40/60; g–30/70; h–20/80; i–10/90

Yazdani Sarvestani and co-authors in their research [23] also demonstrated the formation of cavities in the PA6/PC mixture under a PC concentration of 25 wt.% and more. This phenomenon is explained by insufficient interfacial adhesion and the formation of stress concentration between PA6 and PC.

The authors of the work [24] also noted the deterioration of PC distribution in the PA medium at the ratio (PA-6)/PC 70/30 wt.%. At such allocation the existence of cavities at the interface between PC and PA6 was observed and, accordingly, poor compatibility of the components of the mixture.

It is probably the phenomenon of encapsulation that can explain the rheological properties of the PA6GF30/PC mixture at a ratio of 50/50 wt.% and above when a greater influence of polycarbonate on the flow rate of the melt is manifested. Gattiglia E. and co-authors in their work [25] during the study of thermal properties and compatibility of PA6 and PC polymer compositions also observed a clear separation between the two components and explained this as a possible encapsulation phenomenon.

It is worth noting that the processing of a mixture of waste PA6GF30/PC 90/10 wt.% (Fig. 4 a) did not require a significant correction of the technological process parameters of injection molding [8]. It is due to the uniform distribution of the components of the mixture (Fig. 4 a), which determines a sufficient level of homogeneity of the polymer composite material obtained from this mixture.

The possibility of not increasing the processing temperature by injection molding of a polymer mixture is probably explained by the fact that their copolymerization occurs as a result of their partial chemical interaction. The resulting product has a higher molecular weight, as evidenced by an increase in the MVR index of the mixture, while the degree of crystallinity decreases, as evidenced by the lower density of the resulting polymer compositions.

The authors [26] also confirmed the possibility of chemical interaction of PA and PC during the study of magnetic resonance of PA6/PC, and PA6/PPO mixtures. It was found that the addition of PC to PA prevents the effect of plasticization by causing hardening of the mixture, which is explained by the restriction of the mobility of the NH group as a result of the chemical interaction between PA and PC.

It should be noted that the mixture of PA6GF30/PC 40/60 wt.% (Fig. 4 f) probably acquires the properties of PC, although it shows clear boundaries between polyamide 6 and polycarbonate along with the presence of cavities. Nevertheless, the probable chemical interaction, which occurs without the dissolution of PA in PC, allows the resulting polymer mixture to be processed by injection molding.

4. CONCLUSIONS

The obtained results confirm the possibility of processing the multicomponent waste from polymer composite materials without their separation by injection molding in the absence of significant influence on the process parameters.

It is shown that with a predominant PA content in the PA6GF30/PC mixture, partial copolymerization probably occurs because of chemical interaction with PC. Probably, in the case of the use of glass-filled polyamide in a mixture with polycarbonate, there are the particles of the glass filler that perform the function of compatibilizer. A higher MVR value and a lower density of the product formed may indicate a lower degree of crystallinity. This allows the processing of the mixture without increasing the temperature and without deterioration of mechanical properties.

It was determined that with a predominant content of polycarbonate in the polymer composition, the effect of polyamide is neutralized due to the phenomenon of

polyamide encapsulation in the structure of polycarbonate. It is visible during the study of the morphology of the mixtures. The mixture acquires the characteristics of polycarbonate, accompanied by a gradual increase in compressive strength. Based on the findings, it is possible to recommend the secondary mixture PA6GF30/PC with a composition of 90/10, 80/20, and 70/30 wt.% for processing by injection molding. According to the specified ratio of components, the expected physical and mechanical properties of the finished products are preserved in combination with the optimal parameters of injection molding.

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