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Tri-component Recycled PVC/PE-HD Material Composite with Keratin Fibres

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Abstract

Presented in the article are some mechanical and processing properties of composites prepared from two recycled immiscible polymers: poly(vinyl chloride) (PVC) and high density polyethylene (PE-HD), and keratin fibres used as a filler. The simplex-centroid design technique was adopted to fix the make-up of the composites. Results of the investigations are presented in the form of triangular graphs which illustrate the content percentage of singular components in the composite. It was found that the blend of PVC and PE-HD is suitable as a matrix in polymeric composites to hold keratin fibres as the filler. On the grounds of results obtained, the opportunity is presented to adopt a composition of the composite that responds to a complex of desired properties (within assumed limits of the singular components).

Key words: keratin fibres, composites, PE-HD, PVC, mechanical properties, simplex-centroid design.

Introduction

Polymeric composites based on thermoplastic polymers with nature-derived fibrous fillers (natural fibre composites-NFC) are modern construction materials. Properties of the materials depend largely upon how the fibres are purified, and on conditions under which the composites are prepared. There is a chance of bringing the properties of the materials obtained into line with the needs of various applications by adequately selecting the fibre, matrix, auxiliary agents and conditions of manufacturing [1 - 4]. Demands of the economy and environment, as well as the depletion of crude oil resources have inclined scientists to pay more attention to the recycle of waste materials, including plastics [5, 6]. Plastic wastes are increasingly being reused to produce a number of goods, to give only a few examples, floor covering, garden furniture and many technical appliances. Wood flour is another material used as filler in composites made of recycled plastics (Wood Plastic Composites) [7]. PVC/PE compounds in the form of recyclates of various PE content and composites based on the matrix of those mixtures containing 15%, 30% and 50% of wood flour were processed in a Brabender plastographometer. Based on the results of mechanical and thermal studies, it was found that the addition of a wood filler leads to an increase in the homogeneity of the polymer matrix, which positively affects selected mechanical properties. The presence of a wood filler causes an increase in the softening temperature, especially when the matrix contains over

90% of PVC recyclate. This effect allows to extend the scope of applications of these composite materials at high temperature [7]. Promising results of processing and utility properties of wood composites for the matrix of the mixture of PE-HD and PVC, immiscible with each other, indicate the possibility of its use as a matrix of composites with other fillers such as poultry feathers.

Ever more attention is being paid to polymer composites reinforced with natural fibres, which have the advantage of being renewable and constitute a new generation of resources for the manufacture of polymeric materials [1].

In the article [8], results of examinations of composites prepared on the basis of PE-HD are described, while keratin fibres prepared from poultry feathers were used as a filler.

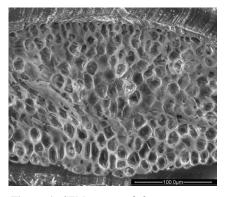


Figure 1. SEM image of the cross-section of a single fibre made from poultry feathers (0.3-5 mm in length).

Table 1. Mass fraction of the components.

No. of sample	X ₁	X ₂	X ₃
1	1	0	0
2	0	1	0
3	0	0	1
4	0.5	0.5	0
5	0	0.5	0.5
6	0.5	0	0,5
7	0.33	0.33	0.33

It was found that the composites containing 15, 30 and 50% of keratin fillers are characterized by a considerable moisture resistance and demonstrate negligible water absorption in comparison with materials containing other natural fillers. Moreover all of the composites are classified as the HB40 category of combustibility, and therefore they can be used as construction elements of common use with increased resistance to fire.

Beneficial properties like low density, silencing, excellent compressibility and elasticity, good insulation and water repellency make keratin fibres an ideal material for use in a variety of polymeric materials. One more asset is their low cost. They are abundant and, in fact, available everywhere, thereby providing a durable and inexhaustible source of raw material supply.

The aim of the investigation presented was the preparation of composites based on two immiscible polymers: PVC and PE-HD, and keratin fibres as a filler, with an assessment of the impact of the content of individual components on the mechanical, thermal and processing properties of the new composite materials. The method of simplex-centroid design was applied to determine the relationship between the composition of composites and their properties.

Materials and methods

Materials

The following materials were used for production of the composites:

 recycled PE-HD (Tipelin BA 550-13 by Tiszai Vegyi Kombinát Nyrt) from waste generated in the manufacture of thin-walled

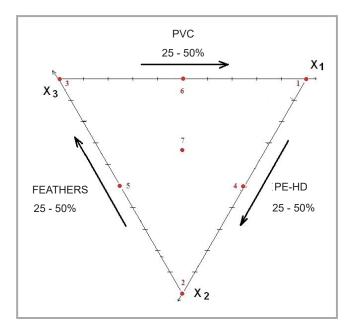


Figure 2. Triangular graph presenting the experimental points for three components.

Table 2. Make up of the PVC/PE-HD/F composites.

No. of	Mayling of the comple	Weight concentration of the components			
sample	Marking of the sample	PVC	PE-HD	FEATHERS	
1	PVC 50/PE-HD 25/F 25	50	25	25	
2	PVC 25/PE-HD 50/F 25	25	50	25	
3	PVC 25/PE-HD 25/F 50	25	25	50	
4	PVC 37.5/PE-HD 37.5/F 25	37.5	37.5	25	
5	PVC 25/PE-HD 37.5/F 37.5	25	37.5	37.5	
6	PVC 37.5/PE-HD 25/F 37.5	37.5	25	37.5	
7	PVC 33.3/PE-HD 33.3/F 33.3	33.3	33.3	33.3	

- pipes (Mass Melt Flow Ratio MFR₁₉₀ \circ C/21 kg/8/2 = 39.2 g/10 min.),
- recycled PVC from waste generated in the manufacture of window profiles (a compound based PVC produced by Anwil, Włocławek, Poland), MFR₁₉₀ °C/21kg/8/2 = 1.2 g/10 min., thermal stability at 200 °C 18 min.,
- filler in the form of keratin fibres derived from poultry feathers: apparent density of 3.5×10^2 g/cm³, content of sulfur -2.9%, nitrogen -15.5%, ash ca. 1% and fat -15.8%.

Figure 1 (see page 73) shows an SEM image of the cross-section of a single fibre made from poultry feathers after pretreatment.

Composition of the composites

The method of simplex-centroid design permits to present in the form of triangular graphs the relationship between a given property (e.g. modulus of elasticity) and the concentration percentage of the components in the composite. Seven composites made up in accordance with the simplex-centroid design and determination of their properties brings about the possibility of reading any composition of the blend and the respective properties of the composite. The relationships can be presented in the form of graphs primarily as fragments of a triangle surface. A big advantage of the simplex-centroid design is that a lot of valuable and reliable information can be derived with just a few experiments made.

Triangular graphs were adopted in this work to illustrate the impact of the composition of PVC/PE-HD/keratin fibre composites upon some properties. The content of each of the three components was varied in the range of 25 to 50 wt.%. Composites made up of the three components: PVC, PE-HD and keratin fibres are hereinafter designated with the abbreviation PVC/PE-HD/F.

The make-up of the composites was adopted on the basis of the simplex-centroid design [9], percentages of the single components X_1 , X_2 , X_3 i.e. PVC, PE-HD and the feathers, falling into the range of 25 - 50%, respectively

Figure 2 presents a triangular graph depicting the plan of the seven design points. Mass fractions of the singular components in the various composites are compiled in *Table 1* and are marked

on the graph. The numbers serve also as a marking of the samples.

Further to the diagram above, the makeup of the PVC/PE-HD/F composites is presented in *Table 2*.

The simplex-centroid design is based on regression analysis of the second order and leads to defining of coefficients of the second degree of polynomial, which determines the relationship between the property analysed and the composition of the composites selected. Three-level experiments were conducted. The X_1 , X_2 , X_3 variables define the basic, minimal and maximal level of each of the components, that is 0.5, 0 and 1, whereby applies the relationship:

$$X_1 + X_2 + X_3 = 1$$
 (1)

The regression equation in this case takes the formula of:

$$y = b_1 X_1 + b_2 X_2 + b_3 X_3 + b_{1,2} X_1 X_2 + b_{1,3} X_1 X_3 + b_{2,3} X_2 X_3$$
(2),

and is a function that presents the relationship between the property examined y and content of the singular components in the given composite, where: $b_{i,j}$ $\langle 1 \leq i < j \leq 3 \rangle$ are coefficients of **Equation 2**.

Preparation of the keratin fibre filler

With impurities separated and screening accomplished, poultry fibres were washed with water containing 3% of the detergent Ludwik® (GRUPA INCO S.A. Poland) at about 40 °C for 1 hour, followed by centrifuging and several water washes to remove the detergent. The fibres were then put into a water bath with the content of 15% of H₂O₂, and after 24 hours they were rinsed with water and centrifuged. The purified, wet feathers were crumbled first by means of a guillotine and next in a plate mill -Sprout – Walorom, with a slot of 0.3 and 0.1 mm (BELOIT, USA). The crumbled feathers were then centrifuged and dried for 3 hours at 80 °C. The dry feathers were crumbled for 10 minutes in a disintegrator (KMW, KARLSTAD, Sweden) to produce keratin fibres 1 - 2 mm long.

Preparation of the composites

Composites made up as presented in *Table 2* were obtained in the crusher of a modular extruder - Zamak Mercator® (Zamak Mercator Sp. z o.o., Poland). First the two plastics PVC and PE-HD

were melted, and after 1.5 minutes keratin fibres were added in small portions. The mass of each of the lots prepared amounted to 70 g. The total softening time was 5 minutes at 185 °C, and the speed of the main rotor - 30 r.p.m. The composite taken out of the crusher chamber was crumbled in a laboratory mill (Home-built). The powdered individual lots after drying for 3 hours at 105 °C were pressed in a hydraulic press (Home-built) at 180 °C and pressure of 8 MPa in the socket within 8 minutes.

Preparation of the samples for testing

From two different moulds, pieces were obtained sized $130 \times 110 \times 4$ mm and $120 \times 120 \times 2$ mm. From these, the following samples were cut out by means of a milling machine - SERON 6090AM® (Poland):

- specimens in the form of a beam sized 80 × 10 × 4 mm for the testing of impact resistance, hardness and Vicat softening temperature,
- specimens in the form of a beam sized 130 × 10 × 4 mm for the testing of flammability,
- paddles 1BA [10] with standarised dimensions of 30 × 5 × 2 mm for the testing of mechanical properties at static extension.

What remained was used in the testing of density, MFR and in thermogravimetric analysis (TGA).

Methods

Unnotched charpy impact test (a_n). Measurements were made in 10 samples of each of the composite according to [11] using a Zwick HIT 5P (Zwick GmbH & Co. KG, Germany).

Mechanical properties at static extension were examined according to [12] by means of a testing machine - Zwick Roell ZO10 (Zwick GmbH & Co. KG, Germany), at ambient temperature and rate of extension of 10 mm/min. Estimation comprised the elasticity module at elongation E_t , tensile strength (σ_M) and relative elongation at break (ϵ_B). Measurements were made in 5 samples of each of the composites.

Flammability by the horizontal burning method was measured according to [13]. The linear rate of burning was calculated for 1.0 MPa of each composite from the formula:

V = 60 L/t in mm/min

where, L – thermal-defected length of the sample in mm, t – burning time of the sample between lines 25 mm and 75 mm in s.

Estimation of the softening temperature was carried out on the basis of standard [14] by the VST B50 method using Vicat apparatus HDT 300A (Chengde Precision Testing Machine Co., Ltd., China).

The mass flow rate (MFR) was estimated according to Standard EN ISO 1133 by means of MFR apparatus type D4004DE (Dynisco, USA).

The density of the composites was measured at ambient temperature with a helium pycnometer - Pycnomatic ATC (Thermo Fisher Scientific, USA).

Thermogravimetric analysis (TGA) was carried out in accordance with standard [15] by means of TG 209 F3 TARSUS apparatus (Netzsch, Germany). Measurements were made in the temperature range from 0 to 700 °C in nitrogen at a heating speed of 10 °C/min. The dependence of the mass loss of the sample on the temperature was registered. From the thermograms prepared, the temperature was read at which mass loss of 5% and 50% had occurred.

Observation of the composite structure by scanning electron microscopy. The structure of the brittle fractures of selected composites was inspected by means of a microscope - Quanta 200 FEI Co. (USA), in a high vacuum at an electron accelerating voltage of 15 kV. The samples were broken in liquid nitrogen, and before being placed in the chamber of the microscope were sprinkled with a thin layer of gold by means of a vacuum sprayer - Q150R S.

Results of the investigation and discussion

Impact resistance by unnotched Charpy method

Figure 3 (see page 76) shows the impact resistance as a function of the variable content of the three composite components. Two points are marked, for example, featuring the following contents of the singular components: one point – 30% PVC, 32.5% PE-HD, 37.5% feathers, and the other – 35% PVC, 37.5% PE-HD and 27.5% feathers.

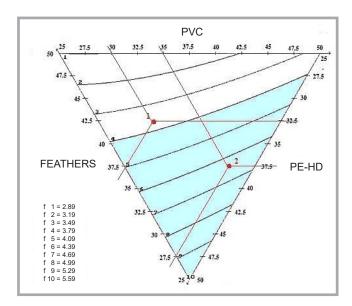


Figure 3. Distribution of impact resistance in kJ/m² of the PVC/PE-HD/F composites as a function of their composition.

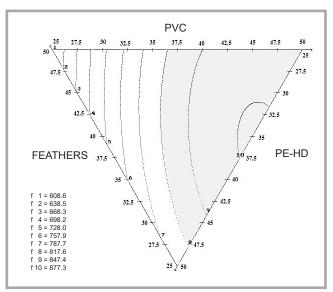


Figure 4. Distribution of modulus of elasticity in MPa of PVC/PE-HD/F composites as a function of their composition.

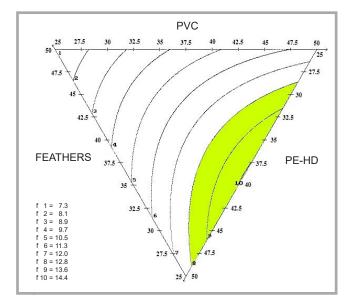


Figure 5. Distribution of tensile strength values in MPa of PVC/PE-HD/F composites as a function of their composition.

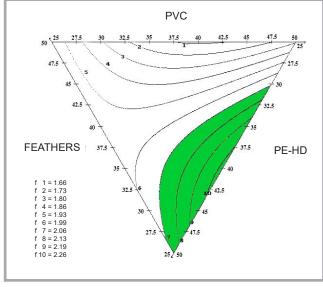


Figure 6. Distribution of relative elongation values in % of composites PVC/PE-HD/F in dependence on their composition.

From the triangular graph, it ensues that the impact resistance is up with an increasing content of PE-HD and falls with an increase in keratin fibres. This may be caused by insufficient adhesion between the feathers and matrix or by the uneven distribution of the filler in the matrix. Composites with a PVC content below 29%, PE-HD above 46% and feathers of no more than 28% reveal the highest impact resistance. Marked in the diagram is the area of impact resistance above 4 kJ/m².

Mechanical properties at static extension

Figures 4 - 6 present the relation between mechanical properties in the form

of triangular graphs: modulus of elasticity, tensile strength and relative elongation at break, and the content of the three composite components: PVC, PE-HD and feathers.

The addition of keratin fibres exerts a distinct impact on the modulus of elasticity of the composites. The modulus goes up with increased contents of PVC and PE-HD and decrease in the filler. The highest values of the modulus of elasticity can be seen in the graph in composites which contain more than 37.5% of PVC, up to 37.5% of PE-HD and no more than 27% of keratin fibres. Marked in the graph is the optimal area of the modulus of elas-

ticity of the composites with values exceeding 800 MPa.

The relationship of tensile strength versus the content of the three components resembles that of the modulus of elasticity. It ensues from the graph in *Figure 5* that the tensile strength of the composites goes up with the increasing content of both polymers and is down when the keratin fibre amount increases. Marked in the graph is the optimal area of tensile strength with the minimum value of 12.5 MPa for composites containing 36 - 37.5% of PVC, 37.5-39% of PE-HD and no more than 25% of feathers.

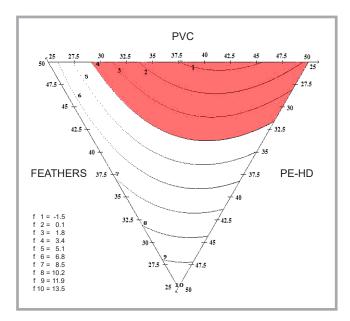


Figure 7. Distribution of the linear value of burning speed in mm/min. of composites PVC/PE-HD/F in dependence on their composition.

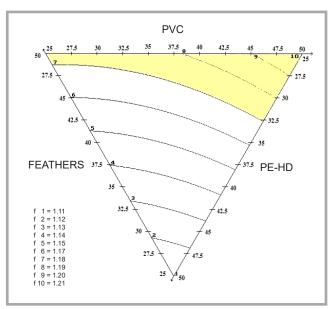


Figure 8. Distribution of density values (g/cm³) of PVC/PE-HD/F composites as a function of their composition.

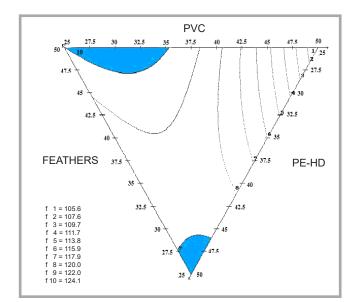


Figure 9. Distribution of softening temperature values in $^{\circ}$ C of composites PVC/PE-HD/F as a function of their composition.

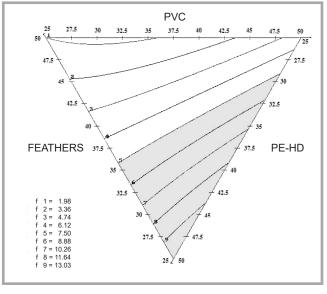


Figure 10. Distribution of MFR values in g/10 min of composites PVC/PE-HD/F as a function of their composition.

It ensues from the relationships presented in *Figure 6* that the relative elongation at break goes up with the increasing content percentage of PE-HD and decreasing content of PVC and keratin fibres. The highest relative elongation at break of more than 2% was recorded in composites that contained from 37.5% to 44% of PE-HD, from 31% to 37% of PVC and no more than 25.5% of keratin fibres.

Estimation of flammability by horizontal burning test

Flammability is amongst the crucial properties of composites that qualify materials for special use. It issues from

the data presented in the triangular graph in Figure 7 that the linear burning speed is up along with the increasing content of PE-HD, and falls when the contents of PVC and feathers decline. The area of lowest linear burning speed depends on the content of PVC, which ought to be in the range from 37.5% to 46% and, at the same time, with up to 25.5% of PE-HD and from 28% to 37.5% of feathers. Marked in the diagram is the area of the composite compositions characterised by a linear burning speed below 4 mm/min. Composites containing more than 32% of PVC, up to 34% of PE-HD and feathers in the amount from 25 to 45% respond to the HB 40 category of flammability

as laid down in Standard PN-EN 60695 -11-10.

Density measured by helium pycnometer

The addition of keratin fibres to the matrix made up of a PVC and PE-HD blend causes a change in the density of the composites obtained. From the data presented in *Figure 8* it may be inferred that the density of the composites is up along with an increasing content of the feathers and declining amount of PE-HD in the mixture. The area of composites with the highest density is in the range of more than 39% of PVC and less than 30% of PE-HD. The lowest density characterises

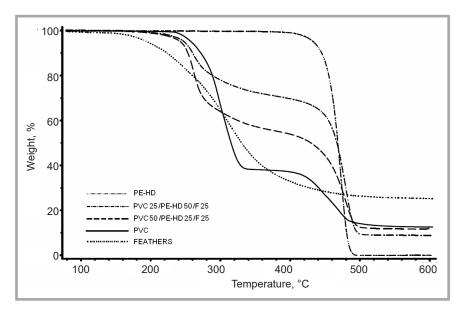


Figure 11. TGA thermograms in the temperature range from 100 to 600 °C.

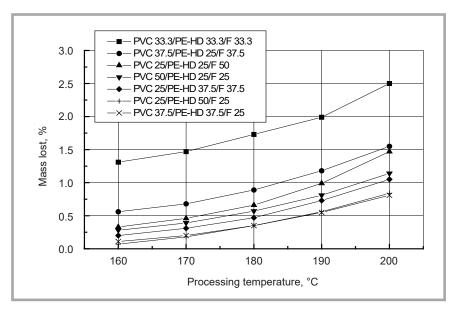


Figure 12. Mass loss of composites PVC/PE-HD/F as a function of the processing temperature.

composites that contain more than 47% of PE-HD and, at the same time, up to 29% of keratin fibres.

Vicat softening temperature

Data presented in *Figure 9* (see page 83) show the highest softening temperatures (above 120°C) in composites that contain from 25 to 35% of PVC and, at the same time, from 45 to 50% of keratin fibres. The area which covers the optimal softening temperatures above 122 °C is marked in the diagram. Composites containing higher amounts of PVC of 41 to 50% with a content of PE-HD in the range of 25 to 41%, reveal softening temperature from 105 to 118 °C.

Mass melt flow rate

Results of measurements made by means of a plastometer, shown in Figure 10 (see page 83), evidence that MFR values of the PVC/PE-HD/F composites increase along with an increasing content of the PE-HD component. The area with the highest MFR i.e. from 10 to 13 g/10 min concerns composites which contain less than 39% of PVC and more than 35% of PE-HD. An increasing content of keratin fibres causes a decline in MFR values. However it, does not witness a worsening of the composite processing behaviour. In fact MFR measurement is related to the flow of polymer particles up to tens of micrometers in size, while the particle size of natural fillers fall into the range of several millimeters. It is a matter of course that a composite with such fillers may not flow at all considering the fact that the standard nozzle of the plastometer is only 2 mm in diameter. Having this in mind, one has to adjust the dimensions of nozzles, pinpoint gates and channels, and the construction of forming heads in injection moulders to the specificity of the plastics with a filler content.

Thermogravimetric analysis

Testing of thermal resistance is crucial in the processing of PVC. The addition of other components to the PVC blends implies examining whether the additives may accelerate the degradation of the polymer. Hence the aim of the TGA analysis is to learn how the three variable components influence the run of TGA thermograms of the PVC/PE-HD/F composites.

The thermograms of two composites with various contents of the two polymer components and constant amount of feathers, and, for comparison, of the three single components: PVC, PE-HD and feathers are presented in *Figure 11*.

The temperature range related to the initial degradation is important in the processing; therefore the temperature at which the 5% loss of mass proceeds was determined from the thermograms of all composites tested. These data are compiled in *Table 3*. TGA analysis was carried out in the temperature range from 100°C up to 600°C; the temperature at which a mass loss of 50% occurs was also defined. Moreover the data shown in *Table 3* may be useful for the possible energy recycling of the composites investigated.

PVC undergoes thermal decomposition in two steps: first in the temperature range of about 200 - 350 °C and then in 350 - 500 °C, with proceeding dehydrochlorination, followed by cracking of the macromolecules [16 - 18]. The thermal resistance of PE-HD is much higher; its decomposition starts at above 400 °C. In keratin, the temperature related to 5% mass loss is 200 °C, while the maximum of the degradation rate appears at about 325 °C [8] and is related to the degradation of proteins in the fibre [19].

The run of thermogram curves of PVC/PE-HD composites with the same con-

tent of the filler depends significantly on the composition of the matrix blend. The temperature at which decomposition starts related to 5% mass loss is in the case of the PVC 50/PE-HD 25/F 25 sample about 10 °C lower in comparison with the temperature of PVC 25/ PE-HD 50/F 25. Moreover both temperature values are lower than the temperature of 5% mass loss of unfilled PVC, which signifies a negative impact of the addition of keratin fibres on the thermal stability of the composites. This is, however, of minor importance since the processing proceeds at a lower temperature below 200 °C. For this reason, in Figure 12 the dependence of mass loss of the PVC/PE-HD/F composite on temperature from 160 to 200 °C (the range of PE and PVC processing temperature), at which the polymers investigated are routinely processed, is presented.

The mass loss registered in the temperature range from 160 to 200 °C amounts from 1.25% to 2.5%, regardless of the content of keratin fibres in the composites based on the PVC/PE-HD matrix and of the content proportion of both polymers. The highest mass loss was registered in the composite PVC 33.3/PE-HD 33.3/F 33.3.

From the TGA analysis it results that the temperature of 5% mass loss of the composites depends primarily on the content of PVC and keratin fibres. The mass loss in the first stage of degradation is related to the presence of PVC and keratin fibres, which are less thermally stable in comparison to PE-HD. In the second stage of degradation of composites the decomposition of PE-HD takes place. The temperature of both the 5% and 50% mass loss rises along with an increasing content of PE-HD and decreasing amount of the filler.

Assessment of the composite structure

Inspection of SEM pictures of composites having a PVC/PE-HD matrix filled with keratin fibres leads to the inference that the filler is uniformly distributed in the polymeric matrix regardless of the dominant content of PVC or PE-HD (*Figure 13.a, 13.c*). The structure of the matrix is in both cases inhomogeneous, which can be clearly seen in *Figures 13.b* and *13.d*, at a magnification of 500×. Seen in the pictures are fragments of feathers in the form of barbules and

Table 3. Characteristics of thermal transformations proceeding during the heating of composites.

Cample	Temperature,°C		
Sample	5% mass loss	50% mass loss	
PVC 50/PE-HD 25/F 25	242.7	429.7	
PVC 25/PE-HD 50/F 25	248.1	467.1	
PVC 25/PE-HD 25/F 50	232.9	431.2	
PVC 37.5/PE-HD 37.5/F 25	245.7	457.3	
PVC 25/PE-HD 37.5/F 37.5	242.1	460.8	
PVC 37.5/PE-HD 25/F 37.5	238.1	431.5	
PVC 33.3/PE-HD 33.3/F 33.3	233.8	449.1	

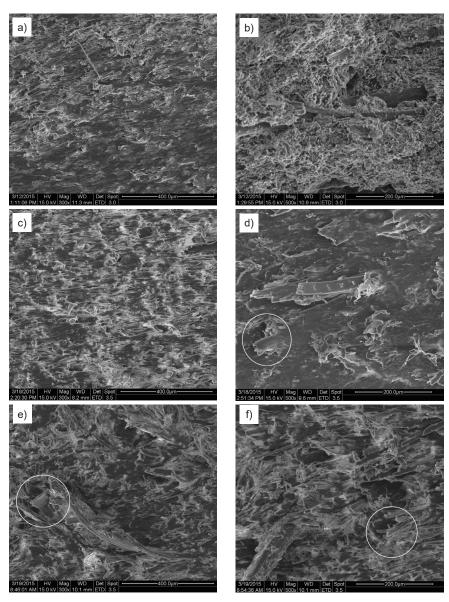


Figure 13. Images of the fracture surface of the PVC/PE-HD/F composites; a) PVC 50/PE-HD 25/F 25, b) PVC 50/PE-HD 25/F 25, c) PVC 25/PE-HD 50/F 25,d) PVC 25/PE-HD 50/F 25,e) PVC 25/PE-HD 25/F 50, f) PVC 25/PE-HD 25/F 50. The circle indicate fragments of special interest.

barbicels which are durably joined with the matrix regardless of its composition.

The image of a fractured surface of the composite with a filler content of 50% (*Figure 13.e, 13.f*) confirms the good ho-

mogeneity of all components. All forms of the feather structure can be seen, from rachis to barbicels. Feather elements like barbs and barbules sized ca. 10 to 20 µm are prevailingly arranged in parallel, which probably results from the flow di-

rection (Figure 13.f). Evidence of good adhesion of the fibre to the matrix is given in the image of the composite in which larger feather elements like rachis and barbs are seen, probably drawn out from the matrix during brittle cracking (Figure 13.d). Kaczmar [20] presented a similar interpretation of the interaction between polymer and hemp fibre. The interaction between the surfaces of the feather elements and the matrix (recounted on the surface unit) is probably analogous, however, the impact resistance may depend on the global friction surface, which stands in relation to the fibre diameter [3].

Summary

A blend of recycled polymers PE-HD and PVC is a suitable material for preparing composites filled with poultry feathers.

The concentration range of the three composite components adopted results from potential uses of the materials. In the recycling business, a lot of polyole-fins may appear in PVC waste and, of course, the reverse too. Screening of such blends is unprofitable.

The concentration of three components including feathers as a filler was varied in the range from 25 to 50 wt%. Disposal of the mix PE-HD and PVC is difficult due to its poor miscibility. Moreover the blend poses a burden to the environment, such as feathers. The preparation of a new useful material from such waste presents a chance for its management.

Owing to the method *simplex-centroid* design applied in this work, the dependence of selected properties on the composition of the composite could be established in the range of concentration of the components adopted. By overlaying of the surfaces of the triangles on each other, which respond to desired proper-

ties, the opportunity presents itself of estimating the optimal composition of the composite, which would provide a desired level of several properties.

On basis of the triangular diagrams, an optimal composition was defined of composites based on a mix of PVC and recycled PE-HD with poultry feathers in the concentration range from 25 to 50% of each of the components, which warrants the best possible mechanical properties at static extension, flame resistance and the melt flow index: 40.5% PVC, 32% PE-HD and 27.5 % fibres of poultry feathers. The concentration of the individual components can, of course, be changed depending upon property requirements.

Moreover the composites containing PVC, PE-HD and poultry feathers in most of their variants belong to category HB40, which marks composites of medium flame-resistance and provides the possibility of their use in the manufacture of elements with improved anti-flame properties.

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