DOI: 10.6060/ivkkt.20226508.6583

УДК: 678.01: 541.68

ФИЗИКО-МЕХАНИЧЕСКИЕ СВОЙСТВА КОМПОЗИТОВ НА ОСНОВЕ ПОЛИЭТИЛЕНА НИЗКОЙ ПЛОТНОСТИ И ТЕРМОЗОЛЫ БЫТОВЫХ ОТХОДОВ

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В статье приводятся результаты исследования влияния концентрации и дисперсности термозолы бытовых отходов на физико-механические свойства композитов на основе полиэтилена низкой плотности. Термозола была получена в термопечи на Балаханском предприятии г. Баку при температуре 1200 °С. Исследовали такие свойства композитов, как предел текучести при растяжении, разрушающее напряжение, относительное удлинения, предел прочности на изгиб. Для изучения влияния дисперсности термозолы на свойства были получены три помола с размером частиц: 80-110 нм, 300-500 нм и 1200-2000 нм. Было установлено, что сравнительно высокими прочностными свойствами (разрушающее напряжение, предел текучести при растяжении, модуль упругости при изгибе) обладают нанокомпозиты полиэтилена низкой плотности с размером частиц 80-110 нм. Показано, что независимо от дисперсности частиц максимальное значение предела текучести при растяжении и разрушающего напряжения достигается у образцов, содержащих 10% масс. термозолы. Максимальное значение предела прочности на изгиб установлено у образцов с 20%масс. содержания термозолы. С целью улучшения совместимости компонентов в смеси полимер-наполнитель использовали компатибилизатор, представляющий собой привитой сополимер полиэтилена с малеиновым ангидридом марки Exxelor PO1040 с 5,6%масс. содержанием малеинового ангидрида. Согласно полученным данным, введение компатибилизатора способствовало существенному улучшению совместимости наполнителя с полимером, что выражалось в повышении прочностных свойств композитов. Дается теоретическое обоснование обнаруженным закономерностям в изменении физико-механических свойств композитов в зависимости от дисперсности и концентрации термозолы. ИК-спектральный анализ композитов показал характерные полосы поглощения термозолы в отдельности и в составе полиэтилена низкой плотности. Смещение полос поглощения в составе полимерной матрицы позволяло утверждать о вероятности физического взаимодействия макроцепей с поверхностью частиц термозолы. Приводятся результаты исследования показателя текучести расплава композитов. Установлено, что введение термозолы в ряде случаев приводит к повышению текучести расплава полимерных композитов.

Ключевые слова: предел текучести при растяжении, разрушающее напряжение, относительное удлинение, теплостойкость, предел прочности на изгиб, термозола

Для цитирования:
Кахраманов Н.Т., Гасанова А.А., Аллахвердиева Х.В., Мустафаева Ф.А., Абдалова С.Р. Физико-механические свойства композитов на основе полиэтилена низкой плотности и термозолы бытовых отходов. *Изв. вузов. Химия и хим. технология*. 2022. Т. 65. Вып. 8. С. 125–133. DOI: 10.6060/ivkkt.20226508.6583.

For citation:
Kakhramanov N.T., Hasanova A.A., Allahverdiyeva Kh.V., Mustafayeva F.A., Abdalova S.R. Physical-mechanical properties of composites based on low density polyethylene and thermal ash of household waste. *ChemChemTech* [*Izv. Vyssh. Uchebn. Zaved. Khim. Khim. Tekhnol*.]. 2022. V. 65. N 8. P. 125–133. DOI: 10.6060/ivkkt.20226508.6583.

PHYSICAL-MECHANICAL PROPERTIES OF COMPOSITES BASED ON LOW DENSITY POLYETHYLENE AND THERMAL ASH OF HOUSEHOLD WASTE

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The article presents the results of a study of the influence of the concentration and dispersion of household waste thermal ash on the physical-mechanical properties of composites based on low-density polyethylene. Thermal ash was obtained in a thermal furnace at the Balakhani enterprise in Baku city at a temperature of 1200 °C. We studied such properties of composites as tensile yield strength, ultimate tensile stress, elongation at break, and flexural strength. To study the effect of thermal ash dispersity on the properties, three grindings with a particle size were obtained: 80-110 nm, 300-500 nm and 1200-2000 nm. It was found that low-density polyethylene nanocomposites with a particle size of 80–110 nm have relatively high strength properties (ultimate tensile stress, tensile yield strength, flexural modulus). It has been shown that, regardless of the dispersion of the particles, the maximum value of the tensile yield strength and ultimate tensile stress is achieved for samples containing 10 wt % of thermal ash. The maximum value of the flexural strength was established for samples with 20 wt % thermal ash content. In order to improve the compatibility of the components in the polymer-filler mixture, a compatibilizer of the Exxelor PO1040 brand was used, which is a graft copolymer of polyethylene with maleic anhydride (content of maleic anhydride 5.6 wt %). According to the data obtained, loading of the compatibilizer contributed to a significant improvement in the compatibility of the filler with the polymer, which was expressed in an increase in the strength properties of the composites. A theoretical substantiation is given for the regularities found in the change in the physical-mechanical properties of composites depending on the dispersion and concentration of thermal ash. IR spectral analysis of the composites showed characteristic absorption bands of thermal ash alone and as part of low density polyethylene. The shift of absorption bands in the composition of the polymer matrix made it possible to assert the probability of physical interaction of macrochains with the surface of thermal ash particles. The results of the study of the melt flow index of composites are presented. It has been established that loading of thermal ash in some cases leads to an increase in the fluidity of the melt of polymer composites.

Key words: tensile yield strength, ultimate tensile stress, elongation at break, heat resistance, flexural strength, thermal ash

INTRODUCTION

As the technique and technology of industrial production improve, research aimed at developing high-quality and low-cost plastic products becomes more and more significant. The good quality of polymeric materials predetermines high strength and thermophysical properties in combination with melt fluidity, which makes it possible to carry out their processing at a satisfactory level by extrusion and injection molding [1-3]. Along with this, research aimed at the development of materials in which high quality is combined with their low cost is becoming relevant [4-9]. In this connection, work aimed at reducing the cost of plastics by using various waste products as a filler began to acquire paramount importance. In Baku, at the Balakhani enterprise, thermal ash is produced in large quantities, obtained in the process of thermal processing of household waste at 1200 °C [10]. Thermal ash is successfully used when laying roads under asphalt. Along with this, recent studies in this direction have shown the promise of their use as a cheap filler for polyolefins [11, 12]. The production of thermal ash nanoparticles made it possible to significantly expand their capabilities in the direction of obtaining nanocomposite polymeric materials.

In this regard, the purpose of the ongoing research was to show new promising possibilities for obtaining high-quality structural materials using the example of low-density polyethylene (LDPE) and thermal ash (TA).

EXPERIMENTAL PART

The object of the study was LDPE grade 108-14, synthesized in SOCAR-POLYMER (Sumgayit city) with an average molecular weight of 125000, density of 927 kg/m^3, ultimate tensile stress of 10,1 MPa, elongation at break of 720%, flexural strength of 19.6 MPa, melt flow index (MFI) – 1.9 g/10 min., heat resistance -85 °C, melting point – 106 °C, degree of crystallinity 57%.

As a filler, thermal ash (TA) was used, obtained at 1200 °C in a thermal furnace of the Balakhani waste processing plant in Baku city [10]. This average thermal ash was taken from the dump accumulated during the year on the territory of the plant. Thermal ash has the following mineralogical composition (wt %): silica – 7.0%, calcite – 54%, anhydride – 15%, illite – 7.0%, NaCl – 4.0%, $Ca_{10}(PO_4)_{5.64}(CO_3)_{0.66}(OH)_3$ – 10%, Fe_2O_3 – 3.0%. The chemical composition of the thermal ash (wt %): Na_2O – 0.42, MgO -0.78, Al_2O_3 – 12.43, SiO_2 – 25.37, P_2O_5 – 2.07, SO_3 – 6.77, K_2O – 1.24, CaO – 41.23, TiO_2 – 0.66, MnO – 0.14, Fe_2O_3 – 5.2, Cr -1.92. From the results of the physicochemical analysis of household waste thermal ash, it can be seen that there are no heavy metals in its composition.

Compatibilizer (PEMA) – functionalized with maleic anhydride (MA) LDPE Exxelor PO1040, intended for loading into the composition of filled LDPE composites. The degree of MA grafting in the composition of LDPE is 5.6 wt. %, MFI is 8.6 g/10 min.

The particle size of the thermal ash was determined using an STA PT1600 Linseiz device (Germany), it was 75-110 nm, 300-500 nm, 1200-2000 nm. Thermal ash nanoparticles were obtained on an A-11 analytical instrument at a rotor speed of 30000 rpm.

Composites based on LDPE and thermal ash were obtained by mixing on a roller at a temperature of 150 °C. After melting LDPE on rollers for 8 min thermal ash was loaded in parts. Plates 2 mm thick were pressed on the basis of the obtained polymer composite at a temperature of 170 °C. The holding time under pressure was maintained at 90 MPa during cooling from 190 to 50 °C.

The melting temperature was determined on a Q-1500d derivatograph, and the heat resistance was determined by the Vicat method on an HDT-Vicat device, which is characterized by high measurement accuracy. The relative error of the experiment is 3-5%.

Ultimate tensile stress, tensile yield strength, and elongation at break of nanocomposites were determined from the results of analytical data (from 5 measurements) in accordance with GOST 11262-80.

When measuring the flexural strength in accordance with GOST-4648-2014, the speed of the movable traverse was 50 mm/min. We used samples obtained by pressing with a size of 80x10x4. Analytical data were determined from 5 measurements. The scatter of readings was no more than 5 rel.%.

IR spectral analysis was carried out on an ALPHA IR-Fourier spectrophotometer in the range of 600-4000 cm^{-1}.

The MFI of polymeric materials was determined on a MELT FLOW TESTER, CEAST MF50 capillary rheometer (INSTRON, Italy) at a temperature of 190 °C and a load of 5 kg. The relative error of the experiment is 5%.

RESULTS AND DISCUSSION

The task of the study was to research the effect of the concentration and dispersion of TA on the main physical-mechanical properties of composites based on LDPE. The concentration of TA was varied within the range of 1.0-40 wt %. To assess the effect of the fineness of TA on the properties, 3 grindings were obtained: 75-110 nm, 300-500 nm, 1200-2000 nm. Of the presented 3 samples of TA, only one with a particle size of 75-110 nm corresponded to the nanoscale level.

Table 1 presents the results of a study of the influence of the concentration and dispersion of TA on the physical-mechanical properties of composites based on LDPE. As can be seen from this table, nanocomposites have relatively high strength characteristics and elongation at break (samples 2-7). By identifying the data given in this table, it can be established that, regardless of the size of the TA particles, the highest values of tensile yield strength and ultimate tensile stress are characterized by composites with a 10 wt % filler content.

Previously, in [12], we showed that in composites based on HDPE, the highest ultimate tensile stress is achieved with the loading of 5.0 wt % TA, while when using LDPE as a polymer matrix, this effect is established at 10 wt % of its content. This circumstance indicates that the crystallinity of the initial polymer matrix has a significant effect on the formation of the structure and, accordingly, the properties of composite materials. The lower the crystallinity of the initial polymer matrix, the more free space is available to fill the amorphous regions of LDPE. It is known that in the process of crystallization and growth of spherulites, solid filler particles are displaced into less dense amorphous regions of the interspherulite space [13-15]. Accumulating in this area, filler particles form certain difficulties for the growth of crystalline structures and at the same time block the mobility of “through” macrochains responsible for the deformation characteristics of composite materials [16-20]. A high concentration of filler particles in the amorphous space contributes to the appearance of brittleness in the samples. Attention should be paid to the fact that, regardless of the fineness of the TA, in the process of a sharp decrease in the elongation at break of the composites to 10% and below, the fragility of the samples increases so much that the values of the tensile yield strength and the ultimate tensile stress are equaled, i.e. in this case, there is no plastic deformation.

Table 1

Influence of the concentration and particle size of thermal ash on the main physical-mechanical properties of composite materials based on LDPE

***Таблица 1*. Влияние концентрации и размера частиц термозолы на основные физико-механические свойства композитных материалов на основе ПЭНП**

№	Composition of composites based on LDPE + thermal ash (TA)	Thermal ash particle size, nm	Tensile yield strength, MPa	Ultimate tensile stress, MPa	Elongation at break, %	Flexural strength, MPa
1	LDPE	-	9.6	10.1	720	12.6
2	99LDPE +1TA	75-110	10.8	11.9	620	13.7
3	95LDPE +5TA		13.6	12.4	185	14.8
4	90LDPE +10TA		15.7	14.3	95	15.5
5	80LDPE +20TA		12.2	11.5	55	16.8
6	70LDPE +30TA		10.9	10.2	35	14.7
7	60LDPE +40TA		9.1	9.1	10	13.1
8	99LDPE +1TA	300-500	10.3	11.7	690	10.5
9	95LDPE +5TA		12.0	12.9	135	11.2
10	90LDPE +10TA		14.9	13.5	85	12.9
11	80LDPE +20TA		11.7	11.2	40	14.0
12	70LDPE +30TA		10.0	9.6	20	13.8
13	60LDPE +40TA		8.8	8.8	10	12.5
14	99LDPE +1TA	1200-2000	9.8	11.0	480	10.0
15	95LDPE +5TA		11.4	12.1	100	11.0
16	90LDPE +10TA		13.3	12.6	65	12.3
17	80LDPE +20TA		11.1	10.6	25	13.5
18	70LDPE +30TA		9.5	9.5	10	12.7
19	60LDPE +40TA		7.1	7.1	-	11.0

Analyzing the nature of the change in the flexural strength, it can be seen that the maximum value of this indicator appears at 20 wt % content of TA. At the same time, the dispersion of TA does not affect the regularity of change in flexural strength.

We have also found that the dependence of the MFI of composites on the concentration of TA is ambiguous. So, for example, with an increase in the concentration of TA nanoparticles (80-110 nm) in the order indicated in table 1, it is accompanied by a change in MFI in the following sequence: 2.5, 3.6, 4.2, 4.0, 3.2, 1.1 g/10 min with a maximum at 10-20 wt % filler content. Comparing the data obtained, it can be established that the loading of TA into the composition of LDPE in an amount of up to 20 wt % contributes to a certain increase in the MFI. There is reason to believe that in the process of thermal combustion of household waste at 1200 °C, in addition to soot, graphite is also formed, which, having a lubricating effect, improves the fluidity of the composite melt.

Fig. 1 and 3 show the IR spectra of TA and a 30 wt % filled composite. Comparing the IR spectra presented in Fig. 1-3, it can be established that the stretching vibrations of TA somewhat change in the composition of the nanocomposite.

So, for example, if the IR spectra of TA are characterized by absorption bands in the region of 1436 cm^{-1}, 1034 cm^{-1} and 874 cm^{-1}, then for nanocomposites these absorption bands are shifted and fixed at 1049 and 873 cm^{-1}. The absorption bands at 874 and 1034 cm^{-1} characterize the asymmetric stretching vibrations of the epoxy group, and the absorption band at 1049 cm^{-1} indicates the presence of phosphorus-containing groups. An intense and wide absorption band at 1436 cm^{-1} is typical for sulfates. These compounds are included in the mineralogical composition of TA.

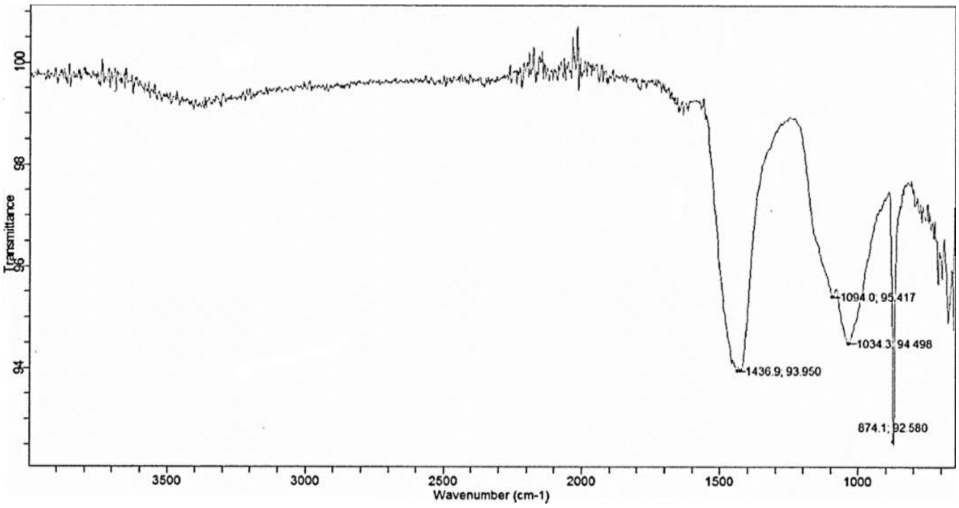

Fig. 1. IR spectrum of thermal ash
Рис. 1. ИК спектр термозолы

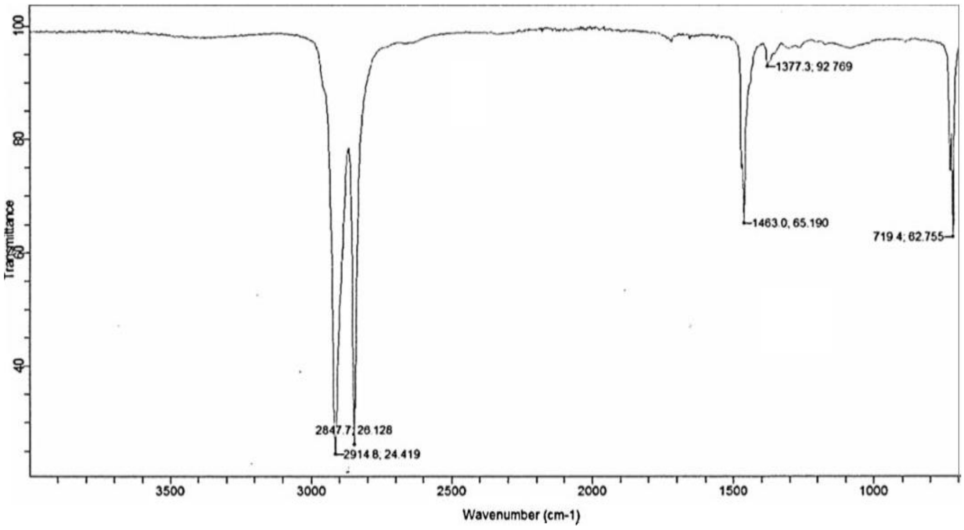

Fig. 2. IR spectrum of initial LDPE
Рис. 2. ИК спектр исходного ПЭНП

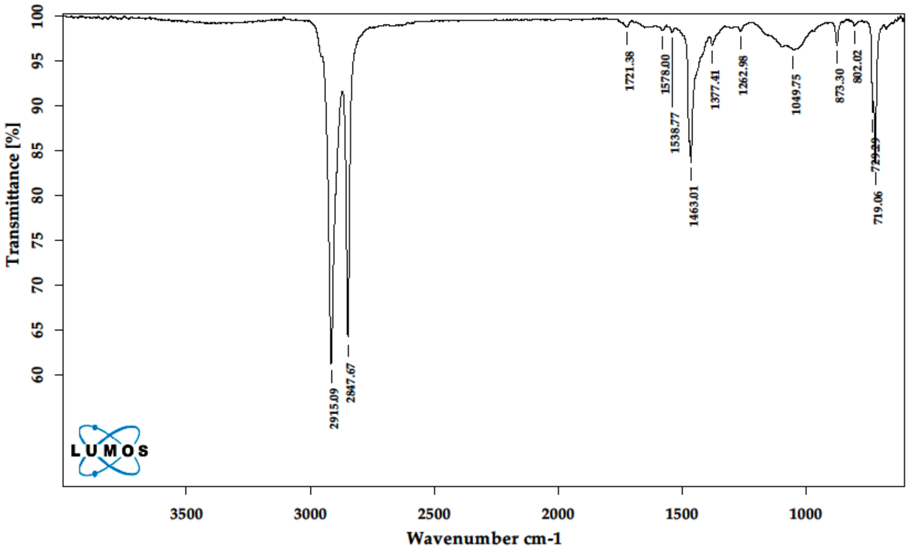

Fig. 3. IR spectra of LDPE nanocomposite filled with 30 wt % thermal ash
Рис. 3. ИК спектры нанокомпозита ПЭНП наполненного 30%масс. термозолы

The very fact of the shift of the characteristic absorption bands of TA in the composition of the polymer matrix from 1034 to 1049 cm^{-1} and from 874 to 873 cm^{-1} clearly indicates a possible physical interaction during the orientation of macrochains on the surface of TA particles. There is reason to believe that such an interaction ultimately promotes the formation of heterogeneous nucleation centers. The simultaneous formation of homogeneous and heterogeneous nucleation centers in the polymer matrix is the main source of the formation of a fine spherulite structure in the polymer matrix [16, 17, 21]. Regardless of the fineness of TA, the latter circumstance is one of the main reasons for the increase in strength characteristics: tensile yield strength, ultimate tensile stress, and flexural strength.

Taking into account that most fillers are poorly compatible with non-polar polyolefins, in this case with LDPE, a compatibilizer (PEMA) was loaded into its composition. It is an LDPE modified with maleic anhydride. A feature of PEMA is that those macrochain segments that do not contain maleic anhydride (MA) units can be incorporated into the LDPE crystal structure. At the same time, if regions of macrochains contain MA units, then they cannot be incorporated into the LDPE spherulitic structure and, therefore, as crystals grow, they will be pushed out into the interspherolitic amorphous region. In other words, an amorphous region is a space filled with filler particles and polar MA groups. It is important to note that an increase in the polarity of the amorphous region favorably affects the improvement of the compatibility of LDPE macrochains with TA particles.

Table 2 shows the results of a study of the effect of PEMA, concentration and dispersion of TA on the physical-mechanical properties of composite materials based on LDPE. Analyzing the data given in this table, it can be established that PEMA contributes to a significant improvement in the properties of composites. The effectiveness of most fillers is more pronounced in the presence of compatibilizers. This fact once again confirms our arguments regarding the role of compatibilizers in improving the quality parameters of polymer composites.

Table 2

Influence of PEMA, concentration and particle size of thermal ash on the main physical-mechanical properties of composite materials based on LDPE

***Таблица 2*. Влияние ПЭМА, концентрации и размера частиц термозолы на основные физико-механические свойства композитных материалов на основе ПЭНП**

№	Composition of composites based on LDPE + TA+PEMA (wt %)	Thermal ash particle size, nm	Tensile yield strength, MPa	Ultimate tensile stress, MPa	Elongation at break, %	Flexural strength, MPa
1	98LDPE+2PEMA	-	9.1	11.5	730	12.0
2	97LDPE+1TA+2 PEMA	75-110	11.8	13.9	620	14.1
3	93LDPE+5TA+2 PEMA		14.5	15.4	215	15.7
4	88LDPE+10TA+2PEMA		17.5	16.6	115	17.6
5	78LDPE+20TA+2PEMA		15.0	13.2	75	18.5
6	68LDPE+30TA+2PEMA		12.9	11.8	55	18.0
7	58LDPE+40TA+2PEMA		9.8	9.8	10	15.3
8	97LDPE+1TA+2PEMA	300-500	11.9	13.0	540	13.8
9	93LDPE+5TA+2PEMA		14.9	14.0	125	15.0
10	88LDPE+10TA+2PEMA		16.4	15.2	100	16.5
11	78LDPE+20TA+2PEMA		13.8	12.0	40	17.2
12	68LDPE+30TA+2PEMA		11.4	10.2	20	16.7
13	58LDPE+40TA+2PEMA		9.3	9.3	10	14.4
14	97 LDPE+1TA+2PEMA	1200-2000	10.4	11.3	280	12.3
15	93 LDPE+5TA+2PEMA		12.3	11.9	95	13.4
16	88LDPE+10TA+2PEMA		14.8	13.2	55	14.8
17	78LDPE+20TA+2PEMA		12.1	11.5	20	14.5
18	68LDPE+30TA+2PEMA		10.5	10.5	10	14.7
19	58LDPE+40TA+2PEMA		8.5	8.5	-	12.8

As can be seen from Table 2, in composites modified with a compatibilizer the maximum values of the tensile yield strength and ultimate tensile stress are also achieved at 10 wt % TA content. At the same time, the maximum value of the flexural strength is fixed at 20 wt % content of TA. It is possible that, under uniaxial tension, the samples are more sensitive to changes in the composition of the composite. This circumstance is due to the fact that a neck is formed in the process of uniaxial plastic deformation of the samples. In this case, the centers of destruction are tears and defects in the crystalline structures arising from an excess of filler particles [18, 22, 23]. The magnitude of the ultimate flexural strength is determined at small deformations that do not contribute to the appearance of a critical point of destruction of the samples. Therefore, the maximum flexural strength values are fixed at relatively high filler concentrations.

Comparing the data presented in Tables 1 and 2, it can be seen that at low concentrations of TA, the elongation at break is characterized by high values (samples-2, 8, 9, 14). Above 200% elongation, the tensile yield strength has values lower than the ultimate tensile stress. However, at an elongation below 200%, the opposite happens: the tensile yield strength has higher values compared to the ultimate tensile stress. And in brittle fracture, the values of both strengths are become equal. The tensile yield strength corresponds to the region of elastic deformation, then the process of recrystallization and necking begins, from this moment the destruction of the samples is characterized by the ultimate tensile stress [15, 16]. At very large deformations, the neck of the samples is stretched to such an extent that, as a result of the orientation of macromolecules, the strength of the samples continues to increase and prevails over the tensile yield strength.

Thus, the conducted studies allow us to assert the possibility of using household waste thermal ash as a filler for polyolefins. The use of a compatibilizer (PEMA) can significantly improve the compatibility of the mixed components of the blend, which manifests itself in a noticeable increase in the strength characteristics of composites based on LDPE and TA. The low cost of waste and the achieved high strength characteristics of composites and nanocomposites based on LDPE give grounds for recommending their use in the production of various structural products for technical purposes by injection molding and extrusion. Primary tests were carried out at the plastics processing enterprises of METAK LLC, where the technological aspects of their processing were worked out in a wide range of temperatures and shear stresses.

CONCLUSIONS

The influence of the concentration and dispersion of TA on the main physical-mechanical properties of composites based on LDPE was studied. We used TA of 3 grindings: 80-110 nm, 300-500 nm, 1200-2000 nm. It is shown that nanocomposites with a particle size of 80-110 nm have relatively high properties.

It was found that, regardless of the fineness of TA, the maximum values of the tensile yield strength and ultimate tensile stress are exhibited by composites with 10 wt % of TA. At the same time, composites with a 20 wt % TA content have the maximum value of flexural strength. The IR spectra of TA and composites are presented, confirming the probability of the existence of physical interaction of macrochains on the surface of filler particles.

The influence of a compatibilizer (PEMA) on the regularity of changes in the above properties was studied. It has been established that the loading of 2.0 wt % PEMA contributes to a significant improvement in all considered physical-mechanical properties of composites based on LDPE.

A theoretical substantiation is given for the processes that characterize the effect of TA particles on the polymer matrix in the interfacial amorphous region.

The authors declare the absence a conflict of interest warranting disclosure in this article.

Авторы заявляют об отсутствии конфликта интересов, требующего раскрытия в данной статье.

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Поступила в редакцию 07.02.2022
Принята к опубликованию 25.05.2022

Received 07.02.2022
Accepted 25.05.2022