Розглянута можливість збільшення об'ємів утилізації відходів буріння шляхом використання їх в якості наповнювача для виготовлення полімерного композиційного матеріалу. В результаті проведених досліджень проведена модифікація вторинного поліетилену високої густини відпрацьованим буровим розчином у вигляді частинок високодисперсного наповнювача. Одержані полімерні композити вторинного поліетилену високої густини, які були наповнені відходами бурового виробництва із вмістом відходів до 30 %. В результаті дослідження виявлені закономірності зміни ударної в'язкості, руйнуючої напруги при вигині і водопоглинання від вмісту твердої фази відпрацьованого бурового розчин у (ТФВБР) у вторинному полімері.

Показано, що при введенні до складу вторинного поліетилену високої густини ТФВБР у вигляді частинок високодисперсного наповнювача відбувається значне підвищення їх міцності без значного погіршення водопоглинання (до 2,9 % при наповненні відходами до 30 %).

Встановлено, що оптимальний вміст відходів бурового виробництва у складі полімерних композитів на основі вторинного поліетилену високої густини складає 20 % мас. При цьому досягаються максимальні значення ударної в'язкості і руйнуючої напруги при вигині для композиції з ТФВБР на основі бентонітової глини до 63,3 кДж/м^2 і 200,1 МПа, а для композиції з солевою ТФВБР до 38,1 кДж/м^2 і 207,4 МПа відповідно. Одержані полімерні композити за своїми експлуатаційними характеристиками перевершують відомі аналогічні полімерні матеріали з використанням таких наповнювачів як тальк і каолін. Це дозволяє рекомендувати сумісну утилізацію відходів буріння і полімерних відходів

Ключові слова: композит полімерний, наповнювач, відпрацьований буровий розчин, модифікація структури, ударна в'язкість, руйнуюча напруга

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IDENTIFICATION OF PROPERTIES OF RECYCLED HIGH-DENSITY POLYETHYLENE COMPOSITES WHEN FILLED WITH WASTE MUD SOLIDS

N. Rykusova
Pastgraduate student
Department of Chemical Engineering and Industrial Ecology**
E-mail: n_rykusova@ukr.net

O. Shestopalov
PhD, Associate Professor*
E-mail: shestopalov.it@khpi.edu.ua

V. Lebedev
PhD, Associate Professor
Department of Technology of Plastics and Biological Active Polymer**

T. Tykhomyrova
PhD, Associate Professor*
E-mail: tatikh@i.ua

G. Bakharieva
PhD, Associate Professor
Department of Occupational Safety and Environmental**
E-mail: baharevaann@gmail.com

*Department of Chemical Technique and Industrial Ecology**
**National Technical University "Kharkiv Polytechnic Institute"
Kyrpychova str., 2, Kharkiv, Ukraine, 61002

1. Introduction

In well-drilling, drilling mud is used, which, after passing the technological cycle, most adversely affects the environment than any other drilling waste. Muds are polydisperse heterogeneous systems, contain both coarse and colloidal particles (more than 90 % of waste mud solids (WMS) represent a fraction of less than 20 microns). According to the composition of the dispersion medium and the dispersed phase, they are divided into water-based muds (clay, carbonate, sulfate) and non-aqueous (hydrocarbon) muds [1]. To achieve the required properties of muds, various chemical reagents are used [2].

After the completion of drilling, waste mud (WM) is discharged in mud collectors (mud pits) and is subject to disposal. When drilling each gas well, an average of about 500 m^3 of WM is formed. Mud collectors or mud pits that are built to store drilling waste also remain potentially environmentally hazardous after well construction.

The issue of the possibility of recycling and treatment of drilling fluids is not fully resolved. The most common method of WM recycling is partial use in the production of building materials [3]. But this method has not found broad application.

One of the promising areas of WM recycling can be the use as a fine filler in the production of polymer composites.

No less urgent problem is polymer waste recycling. It is known that recycled polymers do not fully possess the primary operational properties. At the same time, it is considered that irreversible relaxation processes take place in them during the use of polymer products.

Very relevant today is the production of recycled polyolefin (polyethylene and polypropylene) polymer composites using disperse waste of various productions, both organic and inorganic. By changing the composition and structure of the recycled polymer matrix, it seems possible to regulate the quality characteristics of polymer waste composites within a fairly wide range.

Therefore, the relevant area of research is the development of recycled polymer composites with structural modification using industrial waste, such as WM.

2. Literature review and problem statement

One of the widely used methods of structural modification of the recycled polymer matrix is the introduction of various mineral fillers [4]. In this case, it is possible not only to significantly reduce the cost of the composite, but also to improve a number of physicomechanical characteristics.

The publication [5] shows the possibility to reuse non-metals of recycled printed circuit boards as reinforcing fillers in polypropylene composites. An increase in the strength of composites to 133 % is revealed and the use of no more than 30 % of filler is recommended.

In [6], the possibility of increasing recycling volumes of production waste of alumina – red mud by using as a filler for the polymer composite production is considered. The developed composites with a filler content of 55–75 wt % of red mud had a density within 2,200–2,500 kg/m^3, water absorption within 3.00–5.02 % and compressive strength was over 17 MPa.

However, this method of polymer modification does not always provide the desired results. Most fillers are insufficiently moistened with the polymer matrix, which certainly affects their basic physicomechanical properties [7]. In this regard, the most widespread technique is the introduction of silane coupling agents into composites, intended for targeted improvement of the adhesive interaction at the polymer-filler interface.

Attention is drawn to research on the use of various industrial wastes as fillers of polymer composites. Thus, [8] considers modification of the composite with calcium stearate. According to the authors' recommendations, the filler share in composites is 5–20 wt %, which limits the recycling of waste, especially large-tonnage, in the production of composites.

The publication [9] reviewed the reinforcement of composites with plant waste, such as rice husks and almond shells. It is shown that the greatest improvement of polymer properties is limited by the filler share of 10–15 %, over which there is a deterioration of the mechanical properties and operational characteristics.

In [10], the properties of composites, consisting of unsaturated polyester filled with glass fibers and calcium carbonate, are considered, but the optimal waste concentration to give the best mechanical properties to the composite is not reviewed.

In [11], samples of plastics with organic fillers are studied. It is shown that the properties of the obtained composites depend on fiber composition, adhesion properties, particle size and mass fraction of the filler.

The paper [12] proved the prospects and feasibility of obtaining complex-modified basalt plastics based on corona-treated high-density polyethylene and disperse basalt, since all physicochemical and mechanical properties of polyethylene composites are enhanced. In [13], it is proved that the introduction of disperse basalt and vermiculite into polyethylene makes it possible to increase the whole complex of physicomechanical characteristics and also to improve combustibility indices of the developed composites.

In [14, 15], the high efficiency of kaolin as a filler for mixes of such polymers as polystyrene, low-density polyethylene (LDPE) and polypropylene is also shown. The strengthening effect of such a filler on the complex of physicomechanical characteristics of the obtained composites is demonstrated.

In [16], low-density polyethylene composites filled with talc and chalk are investigated. The optimal filler content of 50 % in the composite is determined.

In [17], the composite based on unsorted crushed waste of thermoplastic polymers – LDPE, HDPE in an amount of 10–50 wt % with the addition of disperse clay with a humidity of 8–12 % is proposed. The technical result of the invention consists in reducing energy consumption, simplifying the method and obtaining material for the production of walling, finishing and road-building composites for civil engineering.

In accordance with [18], thermoplastic (polyethylene, polypropylene, polystyrene, etc.) polymer composite using disperse barium sulfate is developed. By selective surface modification of disperse barium sulfate, the operational properties of the composite can be controlled.

Thus, the creation of composites using industrial waste is a common practice worldwide. At the same time, the creation of composite mixes with the introduction of any filler requires research into the properties of new composites.

From the analysis, it follows that additional information is needed regarding changes in the properties of high-density polyethylene when it is reinforced with solids of different muds.

3. The aim and objectives of the study

The aim of the study was to identify improvements in the properties of recycled high-density polyethylene composites by filling with waste mud solids. This will make it possible to use them instead of virgin polyethylene in the manufacture of critical products for construction and household purposes.

To achieve this aim, it was necessary to perform the following tasks:

– to investigate the effect of waste mud solids on the strength characteristics of recycled high-density polyethylene;

– to identify the optimal waste mud solids content in recycled high-density polyethylene composites.

4. Method of experimental studies of recycled polyethylene and WM composites

4. 1. Method of composites research

As a raw material for the filler production, two types of waste mud, selected at one of the operating gas wells were used. The first sample (WM 1) was selected at the drilling interval (220–2,400 m) and was a polymer-treated clay mud (based on bentonite clay). The second sample (WM 2) corresponded to the drilling interval (2,400–4,145 m) and was a thin clay, polymer, salt mud (based on potassium, sodium and calcium carbonate salts). Studies of mud properties were carried out according to standard methods used in mud quality testing [19].

The chemical composition was determined by the following standard methods. Flint (IV) oxide (with a mass fraction from 1 % to 90 %) was determined by gravimetry and photometry according to DSTU 3305.3-96 (GOST 2642.3-97) using KFK-2 (Russia), iron oxide (III) – by photometry according to DSTU 3305.6-96 (GOST 2642.6-97) using KFK-2 (Russia), sulfur content – by gravimetry according to DSTU 3055-95, loss on ignition – by gravimetry according to GOST 2642.2-86, potassium and sodium oxide content (with a mass fraction from 0.1 % to 5 %) – by plasma spectrometry according to DSTU 3305.11-96 (GOST 2642.11-97), aluminum, magnesium and calcium oxides – by complexonometry.

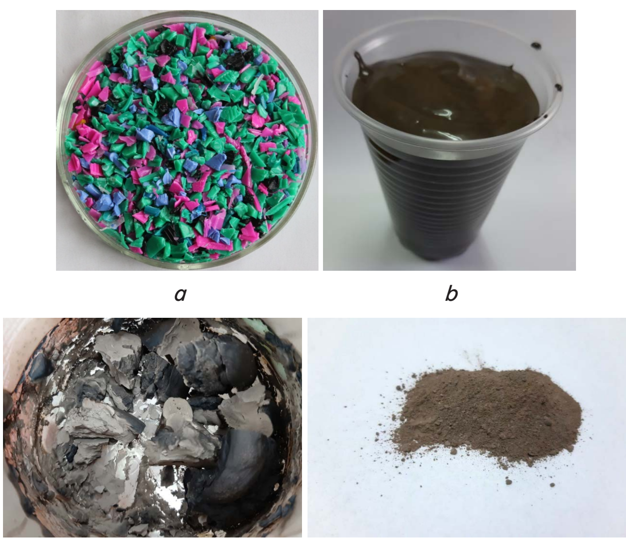
Recycled polyolefins were used as an object of the study, as well as composites on their basis with the addition of inorganic drilling waste. Selection of appropriate polymers for the composite was carried out according to the technological and operational characteristics specified in the technical documentation for the respective grades of polyolefins.

Waste materials of high-density polyethylene were chosen as the polymer matrix for creating composites (Fig. 1, *a*). These materials are crushed, out of service BLUE RAIN water pumps, produced of high-density polyethylene 276-73.

The choice of the above polyolefins was primarily due to the fact that they do not require complex preparation methods. In addition, the products made of these polymers are not subject to significant impacts during use owing to the short life cycle. Before recycling, such waste only needs to be ground and, if necessary, granulated.

Also, the choice of these polymers was facilitated by their rheological characteristics, which determined the possibility of obtaining composites at elevated temperatures with no thermal degradation of the used filler, as well as high-quality mixing of polymers with the filler.

Dried (Fig. 1, *c*) and powdered (Fig. 1, *d*) WM samples (Fig. 1, *b*) were used as a filler.



a *b*

c *d*

Fig. 1. Composite materials: *a* — high-density polyethylene waste; *b* — waste mud; *c* — dried mud; *d* — WMS powder (ready composite filler)

The obtained fine clay fraction (Fig. 1, *d*) was mixed with recycled high-density polymer (Fig. 1, *a*) to the desired filler content from 0 to 30%.

4. 2. Method of obtaining and testing composite samples

Composites were obtained by extruding prepared raw materials in a single-screw laboratory extruder at a temperature of 170–210 °C and screw speed of 0.5–1.5 s^{-1}. The extruder L/D ratio is 25, while to increase the distribution homogeneity of disperse waste in the resulting composites, 3 mass runs were used to obtain finished images. The mass released from the extruder was shaped as a 15×10×1.5–4.5 mm tile.

The study of impact strength and ultimate bending of the obtained samples was carried out on a pendulum pile driver according to GOST 4647 (DIN EN ISO 179-1-2006, DIN EN ISO 179-2-2000) and GOST 9550 (DIN EN ISO 178: 2006), respectively. The choice of these strength characteristics of the studied recycled polyethylene is due to the considerable fragility of this material due to its degradation in use. This can be seen from the decrease in the studied strength properties in comparison with virgin polyethylene 276-73: impact strength decreased from 60 to 18.2 kJ/m^2, breaking stress from 250 to 120 MPa.

For micrographs of filler distribution at the fracture of the samples, the USB Digital Microscope (China) with up to 1600x magnification was used.

Water absorption (wt%) of the samples was determined as the amount of water absorbed by the 15×10×1.5–4.5 mm sample as a result of its stay in distilled water for 24 hours at a temperature of 18–20 °C.

Mathematical processing of experimental results was performed using the Statistica 7.0 software package. In the regression analysis, the task was to search for the functional dependency of the expectation of response $M(Y)$ on the values of the specified factors X: $M(Y)=f(X)$. As a response function, the properties of composites ($Y1$ – impact strength a, kJ/m^2; $Y2$ – breaking stress σ, MPa; $Y3$ – water absorption, %) were considered in the experiments. As an independent variable (X), affecting the response function, mud dosage was chosen.

5. Results of the study of composite properties with the introduction of drilling waste

5. 1. Results of the study of waste mud composition (sample 1 and sample 2)

In this study, muds from different drilling intervals (depth), differing in composition and properties were used as a filler (Table 1).

Table 1

Properties of muds

Parameter	Characteristics of muds	
	WM 1	WM 2
WM sampling depth, m	2,400	4,145
Density, g/cm^3	1.19	1.24
Relative viscosity, sec/quart	90	54
pH	9.5	11.16
Solids, vol.%	22	16
Lubricant (Oil), vol.%	3	4
Sand (solids larger than 75 microns)	0.3	0.2
Chloride concentration, mg/l	2,000	90,000
Total hardness (Ca^{2+}), mg/l	200	360
KCl content, %	–	6
K^+ content, %	–	3.14

Analysis of Table 1 shows that excess chlorides in both samples (especially in WM 2) represent an environmental hazard of contamination of underground aquifers in case of mud pit depressurization. Studies of the chemical composition are given in Table 2.

Table 2

Chemical composition of waste mud samples

Chemical composition, index	Share, %	
	WMS 1	WMS 2
Silicon oxide (SiO_2)	45.3	26.5
Aluminum oxide (Al_2O_3)	16.5	9.0
Calcium oxide (CaO)	6.6	7.6
Magnesium oxide (MgO)	2.84	0.84
Iron oxide (Fe_2O_3)	5.24	4.12
Sodium oxide (Na_2O)	4.20	8.4
Potassium oxide (K_2O)	2.10	9.4
Titanium oxide (Ti_2O)	0.62	0.52
Sulfur oxide (S)	0.90	1.18
Loss on ignition	16.2	32.7

Analysis of Table 2 confirms that these WM samples differ in composition: WM 1 contains more aluminum and silicon compounds, which is typical for bentonite clays, and WM 2 contains more salts of sodium and potassium.

At the same time, of interest is the possibility of binding the solids of drilling waste in the polymer matrix of recycled polymers, for example, as a substitute for other kaolin-based fillers. This will allow not only recycling of drilling waste, but also, possible improvement of operational characteristics of composites obtained from HDPE waste.

5. 2. Results of the study of the characteristics of the obtained composite samples

The results of the study of the obtained composite samples of recycled polymers and dried WM solids are presented in Table 3 and Fig. 2–4.

Table 3

Results of the study of composite characteristics

Filler level, %	Impact strength, kJ/m^2		Breaking stress, MPa		Water absorption, %	
	WMS 1	WMS 2	WMS 1	WMS 2	WMS 1	WMS 2
0	18.2	18.2	120	120	1	1
5	30.2	23.3	156.4	154	1.45	1.2
10	39.4	28.4	188.8	187.6	1.9	1.4
15	54.1	34.5	195	199.3	2.3	1.7
20	63.3	38.1	200.1	207.4	2.6	2.1
25	56.1	34.1	197	189.7	2.75	2.5
30	39	28.7	195.1	166.6	2.9	2.9

As a result of statistical processing of experimental data, equations of approximating curves were obtained, which allow calculating the values of impact strength a (kJ/m^2) depending on the WMS x filler level (wt%) in the composite:

$$a_{\text{WMS 1}}=18.7405+1.1713{\cdot}x+0.1764{\cdot}x^2-0.0064{\cdot}x^3; \quad (1)$$

$$a_{\text{WMS 2}}=18.1357+0.8089{\cdot}x+0.051{\cdot}x^2-0.0022{\cdot}x^3. \quad (2)$$

Graphically, these dependencies are shown in Fig. 2.

Filling of recycled polymer with the disperse phase leads to a significant increase in impact strength with the introduction of both WMS samples relative to the original polymer without filler. Moreover, the bentonite clay WMS filler (WM 1) with a content of 20 % (change in impact strength from 18.2 to 63.3 kJ/m^2) has the highest indices.

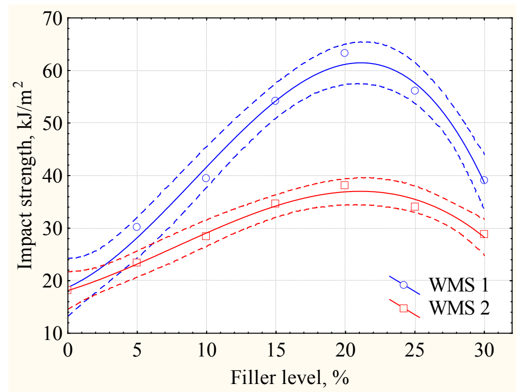


Fig. 2. Dependency of impact strength on waste mud filler level

As a result of statistical data processing, equations of approximating curves were obtained, allowing to calculate values of breaking stress σ (MPa) depending on the WMS x level (wt%) in the composite:

$$\sigma_{\text{WMS1}}=118.9024+10.1506{\cdot}x-0.4088{\cdot}x^2+0.0052{\cdot}x^3; \quad (3)$$

$$\sigma_{\text{WMS2}}=118.9857+8.5068{\cdot}x-0.1725{\cdot}x^2-0.002{\cdot}x^3. \quad (4)$$

Graphically, these dependencies are shown in Fig. 3.

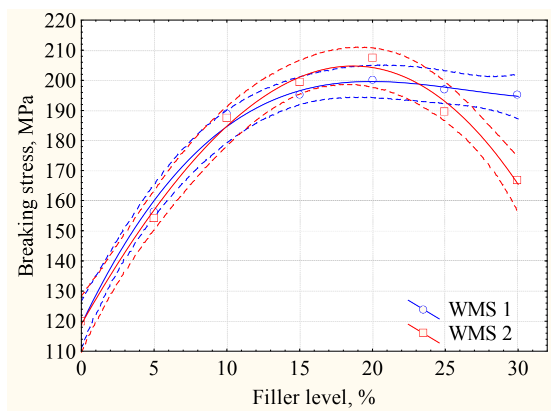


Fig. 3. Dependency of breaking stress on waste mud filler level

Breaking stress also varies significantly: from 120 to 200.1 MPa (with the introduction of 20 % of the WMS 1 filler). Filling of the composite with WMS 2 has a slightly better indicator (up to 207.4 MPa with the introduction of 20 % of filler). The increase in WMS 2 amount leads to a more pronounced drop in strength (up to 166.6 MPa with the introduction of 30 % of filler) than the introduction of WMS 1 filler (up to 195.1 MPa with the introduction of 30 % of filler).

As a result of statistical processing of experimental data, equations of approximating curves were obtained, allowing to calculate water absorption values (%) depending on the WMS x filler level (wt %) in the composite:

$$\textit{Water absorption}_{\text{WMS1}}=0.9738+0.11{\cdot}x-0.0015{\cdot}x^2; \quad (5)$$

$$Water\ absorption_{WMS\ 2}=0.9952+0.0329\cdot x+0.001\cdot x^2. \quad (6)$$

Graphically, these dependencies are shown in Fig. 4.

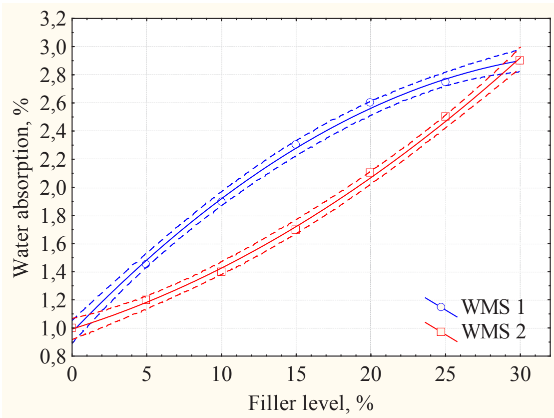


Fig. 4. Dependency of moisture absorption on waste mud filler level

With an increase in the filler share, water absorption increases (Fig. 4), and the dynamics of this indicator in the composite with WMS 1 filler is slightly higher than that of the composite with WMS 2 filler. But with a filling degree over 25 %, water absorption of the samples is close and levels off at 30 %. In general, an increase in water absorption up to 3 % does not significantly affect the operational characteristics of the samples and does not lead to their swelling.

6. Discussion of the results of the study of the developed polymer composites using waste mud

Testing of composites for strength characteristics showed that the removal of inorganic drilling waste can improve the strength characteristics of waste high-density polyethylene. Composites with 20 % of WM filler have the highest strength characteristics, and both impact strength and ultimate bending increase. These results are consistent with the general laws of the processes of filling polymers with disperse fillers. Thus, in [8, 9] it is noted that the introduction of disperse fillers in rather small amounts (up to 10–20 %), as a rule, contributes to the preservation or even some increase of the strength of polymer composites. However, exceeding the specified content additively reduces the strength properties of the composite. This suggests a general boundary degree of filling of polymers with disperse substances to increase their strength properties. It is worth noting that the introduction of WMS into polymer waste increases water absorption from 1 % to 2.9 % with the maximum investigated filling of 30 %. The latter is due to the hydrophilic nature of the studied WMS samples, since clay particles in their composition are prone to swelling.

The main reasons for the strengthening of recycled polyethylene due to the introduction of WMS in the form of fine particles are the growth restriction of polymer microcracks and their branching when meeting with filler particles. It is assumed that due to different particle sizes of the filler, the maximum density of packaging it in the polymer matrix and good homogenization of the filler in the polymer binder were achieved.

These assumptions can be illustrated by the example of fracture micrographs of the samples (Fig. 5). In case of insufficient filling (Fig. 5, *a*), there are unfilled pores and defects. With a filler share of 20 % (Fig. 5, *b*), there is a uniform particle distribution, sufficient filling of the pores and polymer structure that provides the maximum strength. Excess filler (Fig. 5, *c*) decreases the strength of the samples due to the formation of large agglomerates.

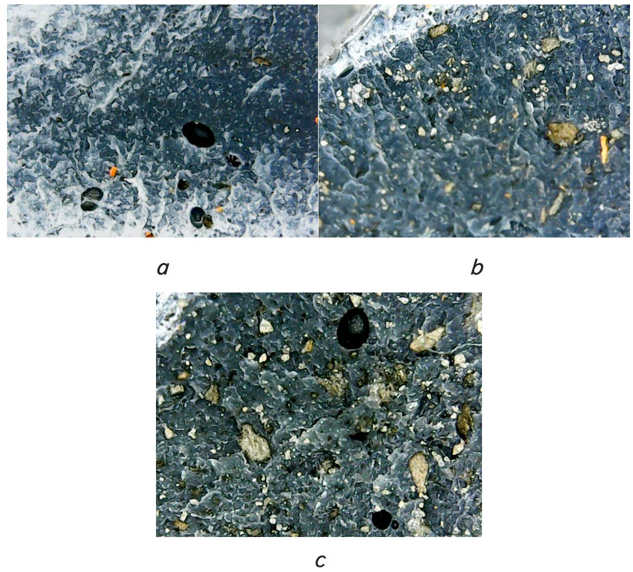


Fig. 6. Photos of fracture of recycled high-density polyethylene composites filled with WMS 1: *a* – 10 % filling; *b* – 20 % filling; *c* – 30 % filling

It should be noted that the obtained composites outperform similar recycled polyethylene polymer composites with the use of disperse fillers of various nature. Thus, [7, 20] recycled polyethylene composites using such fillers as talc, TiO_2 and others have significantly lower operational characteristics than those of the composite samples developed in this work.

The limitation of this study is the insufficient knowledge of the conditions (temperature, changes in the properties of composites during recycling, etc.) of possible recycling of the obtained recycled polyethylene composites filled with WMS to 20–30 %.

Further studies of the developed recycled high-density polyethylene composites using inorganic drilling waste are associated with the structure and morphology of composites. This is important for optimization of recycling conditions depending on the composition and structure of drilling waste used.

In general, modification of recycled high-density polyethylene with the use of inorganic drilling waste (WMS in the amount of 20 %) allows increasing the strength properties (impact strength up to 63.3 kJ/m^2 and ultimate bending to 207.1 MPa) of recycled composites on their basis. This allows using them in the manufacture of critical parts for construction, engineering and domestic purposes.

7. Conclusions

1. Introduction of WMS into recycled high-density polyethylene increased impact strength (up to 63.3 kJ/m^2 after

the introduction of WMS 1) and ultimate bending (up to 207.1 MPa after the introduction of WMS 2) with a slight increase in moisture absorption to 3 %. The obtained recycled high-density polyethylene polymer composites outperform the known similar polymers using such fillers as talc, kaolin, etc. The dependencies are found that allow calculating the impact strength, ultimate bending and water absorption, depending on the waste filling share. These dependencies are regression equations in the form of a quadratic polynomial, from which it follows that the maximum falls on the range of WMS filling of 15–25 %.

2. It is found that the optimal content of WMS in recycled high-density polyethylene polymer composites is 20 wt %. The maximum values of impact strength and ultimate bending for WMS 1 – 63.3 kJ/m^2 and 200.1 MPa, and for WMS 2 – 38.1 kJ/m^2 and 207.4 MPa, respectively, are achieved.

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