

UDC 541; 678; 046.3

DOI <https://doi.org/10.52171/herald.377>

Effect of Filler and Apprete on Polymer Composites Based on Polyethylene and Natural Mineral Rock

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Abstract

This article presents research conducted on adding modifiers containing natural minerals to a polymer matrix as fillers, resulting in higher-quality products when creating next-generation polymer composites. High-density polyethylene was used as the polymer matrix, the natural mineral Kalbajar stone was used as the modifier, and a laboratory-synthesized acrylic acid-hexene-1 copolymer was used as the topcoat. Composite samples were obtained in various component ratios, and a number of physical, mechanical, and thermophysical properties of the resulting materials were studied, yielding satisfactory results. It was established that a number of factors are important for producing products with high performance properties, including the stereoregular structure of the polymer, the dispersion of the filler, and the functionality of the topcoat. The resulting polymer composite materials are considered environmentally and cost-effective.

Keywords: natural mineral rock, polyethylene, polymer composite material, heat resistance, alloy flow index, tensile strength.

Submitted 27 June 2025

Published 16 March 2026

For citation:

V.J. Jafarov, M.A. Manafov, S.A. Bektashi, G.J. Ismailova, G.H. Musaeva, M.J. Rajabova,
S.M. Khalilova, N.A. Alimirzayeva

[Effect of Filler and Apprete on Polymer Composite Based on Polyethylene and Lizardite]

Herald of the Azerbaijan Engineering Academy, 2026, vol. 18 (1), pp. 94-104

ISSN (p): 2076-0515, ISSN (e): 2789-8245

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Polietilen və təbii mineral süxur əsaslı polimer kompozitlərə doldurucu və appretin təsirinin tədqiqi

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Xülasə

Təqdim edilən məqalədə yeni nəsil polimer kompozitlərin yaradılmasında polimer matrisə doldurucu olaraq təbii mineral süxur tərkibli modifikatorların əlavə olunması və daha yüksək keyfiyyətli məhsulların alınması istiqamətində aparılan elmi tədqiqat işi öz əksini tapır. Polimer matris kimi yüksək təzyiqliq polietileni, modifikator kimi Kəlbəcərin təbii mineral süxuru və appret olaraq laboratoriya şəraitində sintez edilmiş akril turşusunun heksen-1 ilə sopolimeri götürülərək komponentlərin müxtəlif nisbətərində kompozit nümunələr alınmış və yaradılan materialların bir sıra fiziki-mexaniki, istilik-fiziki xassələri tədqiq edilmiş qənaətbəxş nəticələr əldə edilmişdir. Müəyyən edilmişdir ki, keyfiyyətli istismar xassələrində malik məhsulların alınması üçün bir sıra amillər zəruri hesab edilir. Məsələn, polimerin stereo müntəzəm quruluşu, doldurucunun dispersliyi, appretin funksionallığı və s. Alınan polimer kompozit materiallar ekoloji cəhətdən və iqtisadi baxımdan səmərəli hesab edilir.

Açar sözlər: təbii mineral süxur, polietilen, polimer kompozit material, istiliyə davamlılıq, ərıntinin axıcılıq göstəricisi, dartılmada möhkəmlik həddi.

Влияние наполнителя и аппрета на полимерные композиты на основе полиэтилена и природных минеральных пород

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Аннотация

В представленной статье отражены научные исследования, проведенные в направлении добавления в полимерную матрицу в качестве наполнителей модификаторов, содержащих природные минеральные породы, и получения более качественных продуктов при создании полимерных композитов нового поколения. В качестве полимерной матрицы использовался полиэтилен высокого давления, в качестве модификатора – природный минеральный камень Калбаджар, а в качестве финишного покрытия – сополимер акриловой кислоты с гексеном-1, синтезированный в лабораторных условиях. Были получены образцы композитов в различных соотношениях компонентов, изучен ряд физико-механических и термофизических свойств созданных материалов, и получены удовлетворительные результаты. Установлено, что для получения продуктов с высококачественными эксплуатационными свойствами важен ряд факторов. Например, стереорегулярная структура полимера, дисперсность наполнителя, функциональность финишного покрытия и др. Полученные полимерные композитные материалы считаются экологически и экономически эффективными.

Ключевые слова: природный минеральный камень, полиэтилен, полимерный композиционный материал, теплостойкость, показатель текучести сплава, предел прочности при растяжении.

Introduction

In conducting scientific research, the issue of carrying out a number of literature studies and, as a result, continuing the work based on a certain plan is one of the important tasks of the researcher. The process of obtaining polymer composite materials and studying a number of their indicators is considered one of the current directions in the field of chemistry. In this regard, in order to obtain high-quality products that contain complex operational properties, fillers selected with appropriate dispersion are included in the system. A number of literature studies have been conducted and reviews have been prepared in this direction.

Analysis of literary sources and the state of the problem

The most famous fiber is carbon. Carbon fibers have exceptionally high heat resistance: when exposed to heat up to 1600-2000°C in the absence of oxygen, the mechanical properties of the fiber do not change, which contributed to their use as a heat-insulating material and heat shields in high-temperature technology. These fibers are resistant to aggressive chemical environments, but oxidize when heated in the presence of oxygen. Their maximum operating temperature in air is about 300-370°C. Applying a thin layer of silicon carbide or boron nitride to carbon fiber allows you to significantly increase the operating temperatures in the air atmosphere. Due to their high chemical resistance, carbon fiber is used to filter aggressive environments, make protective suits, and purify gases. By changing the heat treatment conditions, you can obtain carbon fibers with different electrical properties and use them as electric heating elements. Carbon fiber activation allows

obtaining a material with a large active specific surface area (300-1500 m²/g), which allows increasing adhesion to the matrix. Due to the low density (1.7-1.9 g/cm³), in terms of specific strength (the ratio of strength and modulus to density), the mechanical properties of the best carbon fibers exceed all known heat-resistant fibrous materials. Carbon-carbon composites with high ablative resistance have been obtained based on carbon fibers. However, the specific strength of carbon fibers is inferior to the specific strength of fiberglass and aramid fibers. Structural carbon fiber reinforced plastics are obtained on their basis using polymer binders [1].

In the modern world, the requirements for operational properties of various materials and products obtained from them are constantly increasing, which can be ensured by selecting raw materials and technological production parameters. A significant change in the existing properties of a material and giving it new characteristics is possible by introducing so-called modifying additives, which, along with operational ones, also change the technological properties, facilitating the processing of the material into a product while reducing production costs. Polymers and materials based on them have great prospects in the field of modifying properties. This is due to the large variety of polymer types that differ from each other in properties, and the good compatibility of polymers with various modifying additives. In turn, among polymer-based materials, polymer composite materials (PCM) are of the greatest interest in terms of further modification and the possibility of widespread use. PCMs are based on polymer binders, into which various additives are introduced to modify their properties, facilitating their

processing, increasing resistance to various types of destruction and combustion [2].

The state and prospects of development of production of composites based on various types of multicomponent raw materials (silicon oxide, slag, etc.) and their components are considered. Modern achievements in the field of condensed matter physics of composite materials with mineral matrices and various dimensional levels of fillers are considered. The approaches of leading scientific schools to the creation of composites are analyzed; it is revealed that many issues of obtaining multicomponent composite materials remain open. It is concluded that the optimization of the process of obtaining composites based on multicomponent raw materials should be carried out by changing the target functions and parameters that take into account all types of interaction of components. A method for selecting mineral matrices for the production of composite materials has been developed, the essence of which is to compare the component compositions of raw materials and composite materials, and the search for matrices is performed by the maximum optimal value of intermolecular distances in multicomponent raw materials and composite materials [3].

The results of a study of mixing compositions based on polypropylene and vegetable fillers modified with an enzyme preparation are presented.

During the experiment, the optimal modification time and the ratio of the volume of water to the mass of the vegetable filler during modification were determined.

Physical and mechanical tests revealed that the composites with the modification were superior to the basic composites without the modification [4].

The paper demonstrates the possibility of using fibrous and scaly fillers from rocks such as basalts, silicate waste: substandard impurities, waste from the production of basalt fibers (such as "beads"), scaly materials capable of retaining their properties under concrete hardening conditions in cement compositions. partitions, curbs, berths, etc.) [5].

The issues related to obtaining composite materials based on synthetic polymer matrices reinforced with natural fibers of different classes are considered. The characteristics of the main natural fibers of plant and animal origin used to create polymer composites are given, and the properties of the composites themselves are described. The advantages and disadvantages of using natural fibers to obtain polymer composite materials for various purposes are discussed [6].

Every year the volume of accumulated polymer waste, in particular, household waste, increases, and the issues of their storage and disposal are becoming increasingly relevant. There are many approaches used to process synthetic polymers by mixing with a number of other (both synthetic and natural) polymers. In addition to disposal, the purpose of these studies is also to impart new properties to the resulting materials. However, it is worth noting that it is especially important to search for new approaches to creating biodegradable materials that could decompose under the influence of external factors into substances harmless to nature. One of the polymers of interest is polylactide, obtained from lactic acid. In its properties, polylactide is close to a number of synthetic polymers, but it also has disadvantages, in particular, it is brittle. When creating new materials, polylactide was plasticized using polyethylene glycol (PEG) of

various molecular weights. Mixing was carried out under conditions of high temperatures and shear deformations at different ratios of components. To impart functionality to the materials, biocomposites based on plasticized polylactide with the introduction of stabilized silver nanoparticles were developed. Films were pressed from the obtained biocomposites for further research. Since some of the important characteristics of the materials are their mechanical and thermal properties, they were studied depending on the mass of the PEG used, as well as on the introduction and amount of silver nanoparticles. The results obtained will allow selecting the optimal formulations of biocomposites for their further study [7].

Bio-polymer composites are emerging as a feasible substitute for conventional polymers in various major fields of applications. In the current research, the impact of hybridization of inter and intra-woven natural engineering fibers namely, brown flax, white flax, and jute of different amalgamations along with commingled SCF and novel nano IW filler has been studied. The composites are manufactured by hand layup process and physical and thermomechanical characteristics of the composites are experimentally studied. The composites mingled with IW filler demonstrated better dimensional stability and mechanical properties such as maximum tensile strength of 36.74 MPa, bending strength of 82.53 MPa, and hardness of 84.89 D compared to other composites. However, elastic and flexural modulus are maximum for composite without any filler. The composites mingled with SCF filler demonstrated the highest impact strength, energy absorption, and elongation. Thermogravimetric analysis and thermal conductivity study demonstrated that the composite with SCF filler exhibited better

thermal stability and thermal resistance than the other hybrid composites [8-10].

The presented research work provides for the production of new composites with complex performance properties in various mass. % ratios of components based on isotactic polypropylene and the natural mineral "Demirchi Dam" of the Karabakh region and the study of the effect of fillers on a number of their properties is reflected. In order to ensure and improve adhesion between the polymer matrix and the filler, as well as to obtain composites with higher physical and mechanical properties, a sizing agent is included in the system. Acrylic acid polymer synthesized in laboratory conditions was taken as a coupling agent. The complex and multifaceted properties of polymers greatly facilitate their use as a matrix in obtaining a homogeneous system using them as raw materials. The goal is to realize the possibility of obtaining and using both economically and environmentally competent products for various purposes. It has been established that a number of properties of the resulting polymer composites directly depend on various factors - the structure of the polymer matrix, the amount and dispersion properties of fillers, and the properties of the sizing agent in the interfacial layer. From this point of view, work is considered purposeful [11].

Based on experimental parameters, the optimal amount of filler in the system is determined and used depending on its nature, composition, dispersity, and porosity [12].

Problem statement and solution direction

A number of research works on the path of scientific and technical progress in the specific direction of modern chemical science are indispensable for the development of this

field. In this regard, guided by the urgency of the issue of creating new generation polymer composites distinguished by complex quality criteria, research was conducted in this direction and sufficient results were achieved.

The creation of multicomponent systems and obtaining high results directly depends on a number of factors. Taking into account all of the above, a matrix-filler-finishing system was built and its properties were determined.

Experimental part

Polymer: High-density polyethylene (HDPE)-brand - 10803-020

Filler: Natural mineral rock (53 and 106 mkm dispersion)

Appret: Copolymer of acrylic acid with hexene-1

High-pressure polyethylene is obtained at a pressure of 150-300 MPa and a temperature of 150-320°C. The average molecular weight of the obtained HDPE is $8 \times 10^4 \times 10^5$ and the degree of crystallization is 50-60%.

The natural mineral rock taken as a filler is lizardite, a mineral rock from the Turshsu spring in the village of Zulfugarli, Kalbajar

region, Karabakh territory, and has been determined the chemical and mineralogical composition of the rock.

The difference in the percentage of oxides in white and black mineral rocks and their discharges due to their chemical composition directly depends on the nature of the mineral, its color and, most importantly, its origin. In the black sample of the rock, magnesium oxide predominates in quantity.

In the white sample, silicon 4-oxide (quartz sand) is more abundant. In the lizardite discharge, quartz sand is more abundant than in the white sample. From the analysis of the chemical composition, it can be concluded that the black rock has a richer composition.

Studying the composition of the filler allows us to give it a conditional name:

$*(MgFe)_3Si_2O_5(OH)_4$ – trimagnoferro dissilicookcsi tetrahidrocsolizardite Samples corresponding to the 50 and 106 mkm dispersion of the filler were analyzed by infrared spectroscopic analysis on an Agilent Cary 630 FTIR instrument (wavelength 600-4000 sm^{-1}) from Agilent Technologies.

Table 1(a) – Chemical composition of the filler, %

Conventional name of the sample	Na ₂ O	MgO	Al ₂ O ₃	SiO ₂	P ₂ O ₅	SO ₃	K ₂ O	YTI
Lizardite (black)	0.01	36.9	0.57	36.5	0.01	0.28	0.01	13.3
Lizardite (white)	0.14	9.18	12.9	40.2	0.12	0.43	0.02	4.5
Waste	0.01	34.9	0.53	37.1	0.01	0.11	0.01	13.06

Table 1(b) – Chemical composition of the filler, %

Conventional name of the sample	CaO	TiO ₂	MnO	Fe ₂ O ₃	NiO	Cr ₂ O ₃	Cl	YTI
Lizardite (black)	0.21	0.01	0.12	11.0	0.45	0.40	0.07	13.3
Lizardite (white)	26.4	0.98	0.05	5.0	0.02	0.03	0.01	4.5
Waste	0.30	0.01	0.14	12.5	0.61	0.53	0.10	13.06

Note: VOC indicates the amount of volatile components at 950°C.

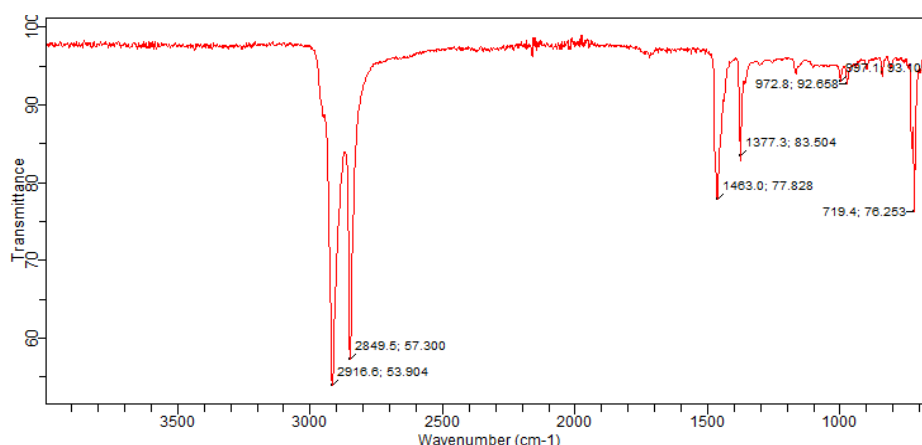


Figure 1 – Photograph of infrared spectra corresponding to a 50 mkm dispersion of the filler

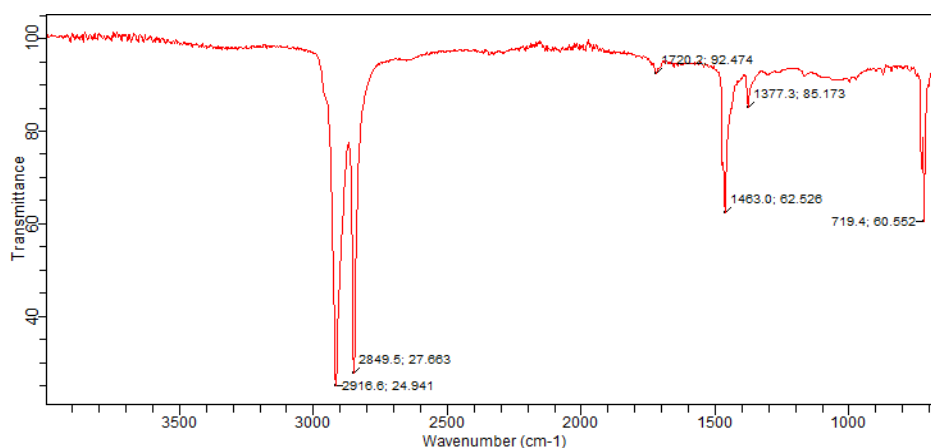


Figure 2 – Photograph of infrared spectra corresponding to a 106 mkm dispersion of the filler

In the spectrum presented in the figures, it was determined that the absorption bands of the filler with a dispersion of 50 mkm had stripes by giving the values.

The interval between absorption bands in the spectra corresponding to a dispersion of 50 mkm of the filler is larger than the interval between absorption bands in the spectra

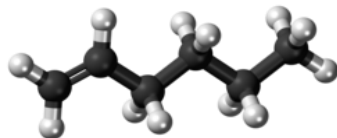
corresponding to a dispersion of 106 mkm. This is explained by the higher reactivity and better compatibility of the filler with the more dispersed state. A copolymer of hexene-1 and acrylic acid synthesized in the laboratory and four organic compounds characterized by different functionalities were taken as the finishing agent.

Table 2 – Mineralogical composition of the filler, %

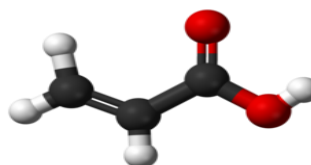
Conventional name of the specimen	(Mg,Al) ₃ ((Si,Fe) ₃ O ₅ (OH) ₄ Lizardite	MgFe ₂ O ₄ (magnezium ferlite)	Other mixtures
Lizardite (black)	88	10	2
Lizardite (white)	-	-	-
Waste	87	13	2

Copolymer of acrylic acid with hexene-1:

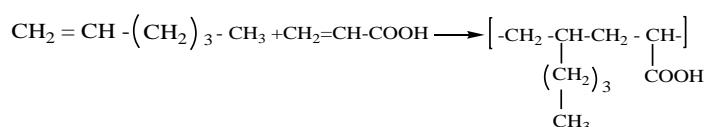
Hexen-1 $\text{CH}_2=\text{CH}-\text{CH}_2-\text{CH}_2-\text{CH}_2-\text{CH}_3$



Acrylic acid $\text{CH}_2=\text{CH}-\text{COOH}$



Reaction scheme:



Results and discussion

Samples of the composite containing HDPE-filler-apprete corresponding to different mass percentages were obtained and their physical-mechanical and a number of properties were studied. Both unsized and sized samples corresponding to the 50 and 106 micron dispersion of the filler were created and their indicators were studied. The following tables 1, 2 present the physical-mechanical indicators of the samples of the composite in different mass ratios. The discussion of the results obtained is a harbinger of the achievement of higher indicators and the effectiveness of the composite samples created.

The interpretation of Table 3 (a), (b), (c), (d) is presented: When looking at the tables, it is observed that the indicators obtained as a result of the influence of a number of factors have lower and higher values. Here, the dispersion dimensions of the filler and the presence of the sizing agent in the system are considered to be the main factors.

Thus, in samples where the filler is more dispersed, the values of the tensile strength limit of each composite sample are higher. Accordingly, the value of the alloy's fluidity index decreases to a minimum in samples with a higher dispersion degree. This is explained by

the corresponding decrease in the fluidity degree in the system due to the influence of the filler. When the sizing agent is present in the system, a slight fluidity is observed as a result of its softness to the system and the provision of homogeneity in the intermolecular pores. Thus, in the composite samples without sizing corresponding to the 50 mkm dispersion of the filler, the tensile strength increases and becomes $\sigma=16.5$ MPa in the ratio of components HDPE/Filler-30/70 mass percent.

Table 3 (a) – Physical and mechanical properties of the composite. (Without apprete)
Filler: 50 mkm

Composition, %	Tensile force, σ , MPa	Relative elongation ϵ , %	AFI, g/10min
HDPE-100	10.8	547.5	2.8
HDPE-70 Filler-30	12.4	30	2.0
HDPE-60 Filler-40	13.8	24	0.7
HDPE-50 Filler-50	13.1	20	0.6
HDPE-40 Filler-60	14.5	20	0.37
HDPE-30 Filler-70	16.5	8	-

Table 3 (b) – Physical and mechanical properties of the composite (with apprete)

Filler: 50 mkm

Apprete: Hexene-1+ acrylic acid (3 %)

Composition, %	Tensile force, σ , MPa	Relative elongation, ε , %	AFI, g/10min
HDPE-70 Filler-30 Apprete	13.8	28	0.9
HDPE-60 Filler-40 Apprete	14.9	22	0.7
HDPE-50 Filler-50 Apprete	15.0	26	0.23
HDPE-40 Filler-60 Apprete	16.0	23	0.07
HDPE-30 Filler-70 Apprete	17.2	8	0.03

Table 3 (c) – Physical and mechanical properties of the composite (without apprete)

Filler: 106 mkm

Composition, %	Tensile force, σ , MPa	Relative elongation, ε , %	AFI, g/10min
HDPE-70 Filler-30	11.5	30	2.5
HDPE-60 Filler-40	12.4	22	0.3
HDPE-50 Filler-50	13.0	21	0.1
HDPE-40 Filler-60	13.5	22	-
HDPE-30 Filler-70	15.0	12	-

Accordingly, the relative elongation at break takes a minimum value and becomes $\bar{\varepsilon}=8$ %. When sizing is added to the system in the appropriate proportions, i.e., HDPE/Filler/Apprete-30/70/3 mass percent, the strength indicator takes a high value and becomes $\sigma=17.2$ MPa. The relative elongation also takes a minimum value and becomes $\bar{\varepsilon}=8$ % in the same way. However, in the presence of sizing,

a slight fluidity is observed in the system. This also finds its explanation in the above data. In the samples corresponding to the 106 μm dispersion of the filler, the values of the strength indicator take relatively low values.

Table 3 (d) – Physical and mechanical properties of the composite (with apprete)

Filler: 106 mkm

Apprete: Hexene-1+ acrylic acid (3 %)

Composition, %	Tensile force, σ , MPa	Relative elongation, ε , %	AFI, g/10min
HDPE-70 Filler-30 Apprete-3	12.8	28	0.9
HDPE-60 Filler -40 Apprete-3	13.0	20	0.7
HDPE-50 Filler -50 Apprete-3	13.8	20	0.23
HDPE-40 Filler -60 Apprete-3	15.0	12	0.07
HDPE-30 Filler -70 Apprete-3	16.5	4	-

This is explained by the large size of the filler and the relatively low homogeneity index of the system, that is, the filler can form in the form of “domains” due to poor mixing in the intermolecular layers. From this it can be concluded that the physicomechanical indicators of the composite take more reasonable values when the filler is more dispersed and in the presence of a finish. These obtained results directly expand the application areas of the composite sample. As a result of the study, the heat resistance properties of the composite samples were studied.

In Table 4 (a), (b) the heat resistance indicators according to Vic. of the composite samples corresponding to the 106 and 50

micron dispersion of the filler, without sizing and in the presence of both sizing, have been studied and the results are presented.

Table 4 (a) – Heat-resistant properties of composite samples. (filler 106 and 50 micron)

Without apprete

Polymer composites, %	Filler, %, mkm 10650	Heat resistance according to Vica, °C
HDPE-100	-	130
HDPE-70	Filler-30	125 120
HDPE-60	Filler -40	135 125
HDPE-50	Filler -50	131 123
HDPE-40	Filler -60	134 128
HDPE-30	Filler -70	138 132

Table 4 (b) – Heat-resistant properties of composite samples (filler 106 and 50 micron)

With a apprete – Hex.-1+ acrilid acid (in each sample, the appret is 3 % by weight)

№	Polymer composites, %	Filler, %, mkm 106 50	Heat resistance according to Vica, °C
1	HDPE-100	-	130
2	HDPE-70	Filler-30	135 131
3	HDPE-60	Filler -40	152 143
4	HDPE-50	Filler -50	155 150
5	HDPE-40	Filler -60	170 163
6	HDPE-30	Filler -70	175 173

According to the results obtained, the heat resistance of the composite sample obtained in the presence of the organosilicon compound corresponding to the 106 mkm dispersion of the filler, in the

HDPE/Filler/Apprete-30/70/3 % system is 175°C, taking a maximum value.

This can also be explained by the fact that the presence of the filler in the form of relatively large grains in the composite creates a more stable state by creating a heavier, “rougher” state in the total mass. As a result of the high functionality conditions and reactivity of the compound in the system created in the presence of organosilicon compounds, the system is characterized by being more resistant to thermal factors.

Conclusion

The issue of creating polymer composite materials with complex operational properties, characterized by a number of specific characteristics of polyolefins and their use, natural mineral resources and their discharges and the presence of fillers, and their use as primary raw materials in various fields, which include high quality criteria, is one of the leading directions of the modern chemical industry. Taking into account these factors, taking HDPE as a matrix, mineral rock, which is a natural mineral rock conditions as a filler, composite samples were created in the appropriate proportions of the components and their physical-mechanical, thermal-physical and a number of properties were studied.

As a result of the study, it was determined that the strength indicators were maximum in the presence of an organic silicon compound and in a more dispersed sample of the filler. However, as a result of the study of the heat resistance properties, it was determined that the system was more heat-resistant with a dispersion of the filler of 106 mkm. The reasons for this are explained in detail above.

Thus, as a result of the scientific research work, polymer composites with high durability,

heat resistance and a number of positive properties are recommended for application in a number of directions of industry and technology. The presence of such different properties directly expands their scope of application and creates a guarantee for their future use in practice. The scientific research

work is considered satisfactory both theoretically and experimentally.

Conflict of Interests

The authors declare there is no conflict of interests related to the publication of this article.

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