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Comparative Study of Various Volcanic Materials as Fillers in Polymer Composites

ABSTRACT

The object of the study was composite materials using rocks of volcanic origin as a filler (60-90 wt.%) and aqueous dispersions of polymers Latex 2012 and Policril 590 as a matrix. The peculiarities of the chemical and mineralogical composition and surface properties of perlite and zeolite as factors of interaction with the binder in the formation of the composite structure are shown. Differences in lyophilicity coefficients and effective specific surface of zeolite and perlite were determined, which are 0.318 versus 0.189 and 11.68 versus 2.20 m2/g, respectively. The influence of a high concentration of fillers on the formation of the pore structure and indicators of physical and mechanical properties of composites is evaluated. The possibility of adjusting the properties of the composites in the following range was established: water absorption in the range of 2.63-14.16 wt.%, open porosity 3.58-21.35 %, residual strain 0.1-0.3, Young's modulus 19.7-677.5 MPa.

Keywords: *composite, filler, perlite, zeolite, concentration, copolymer, porosity, properties.*

INTRODUCTION

The development of scientific and technical principles of polymer composite materials technology is the subject of numerous studies [1-3]. A significant role of composite fillers has been recognised, the use of which can significantly reduce the amount of required binder polymers and improve the properties of materials [4-6].

According to the modern material science concept of the composition structure properties relationship, the characteristics of composites depend on the types of filler and matrix, manufacturing technology and parameters. At the same time, the degree of bonding of the components, their concentration and uniformity of volume distribution determine the structure parameters, general physical, mechanical and special properties of composites.

Natural materials such as chalk, kaolin, and graphite are mainly used as dispersed fillers [7-9]. One of the areas of expansion of the raw material base of fillers is the study and use of by-products of non-metallic materials extraction [10].

In this regard, developments in the use of rocks of volcanic origin, common in theworld and Ukraine, among which perlite and zeolite play a significant role, are of particular interest [11, 12].

Perlite is a rock of volcanic origin. At the edge of a lava flow, at the points of contact between magma melts and the ground, volcanic glass, obsidian, is formed afterthe lava has cooled rapidly. Subsequently, groundwater seeps through the obsidian, undergoing the process of its hydration and the formation of perlite [13].

Perlite is characterised by a fine concentric-shell structure. Perlite differs from other volcanic rocks in that it contains constitutional water (more than 1%). The porosity can range from 8 to 40 % [14].

The use of perlite in composite materials has a number of advantages, such as low density, good thermal insulation properties, resistance to fire, moisture and chemical resistance.

Bituminised perlite is a rather interesting material

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used in the manufacture of roofinsulation. Perlite is first treated with bitumen in the factory. When a solvent is addedto it, it becomes adhesive. This helps to form quite strong insulation layers of various shapes [15].

According to the genetic criterion, all existing varieties of perlite are divided into two large groups: primary hydrated rock and secondary hydrated rock or primary perlite and secondary perlite.

In most cases, primary perlites have a porous or pumice-like texture, with the amount of structural water ranging from 1.5 to 4.5%. Such perlites include those from deposits in Armenia, Georgia, America, Greece, and Turkey.

Secondary pearls can be represented by massive and loose varieties with a water content of 4.5 to 9.5%. These include perlites from deposits in Ukraine, Mongolia, and China.

Zeolite also belongs to rocks of volcanic origin [16-18]. It is known that zeolites are characterised by the development of molecular-sized pores - uniformly sized channels and cavities that create a large specific surface area. In addition to structural features, it is important to note the distribution and significant reserves of natural zeolite deposits, including in Ukraine [19].

Natural zeolites are aqueous aluminosilicates with a skeletal structure that have uniform pores of molecular size.

In Ukraine, the zeolite deposit is located in Sokyrnytsia village (Zakarpattia region). The zeolite of the Sokyrnytsia deposit contains a large amountof clinoptilolite. Sufficient mechanical strength of clinoptilolite, resistance to high temperatures [20], aggressive environments and ionising radiation, selectivity to cations of alkaline, alkaline earth, rare earth, scattered and some heavy metals, absorption capacity and sieve effect - all this leads to the wide use of the mineral.

Developments in the use of new types of raw materials, including zeolite and perlite, require consideration of their physical and chemical composition, impact on the structure formation and characteristics of the systems under study and product properties.

One of the ways to improve the properties of polymers is to fill them with structurally active additives, the addition of which ensures the formation of a given phase and supramolecular structure of the matrix. Currently, layered and skeletal natural minerals, including natural zeolite and perlite, are often used as modifiers of various polymer matrices (polytetrafluoroethylene, polyethylenes, polyester resins, rubbers, etc.).

It is known that the introduction of zeolites into a polymer improves the functional properties of polymer composite materials (PCM). Work [21] shows an improvement in the strength of an epoxy material by 43% when zeolite of natural origin is introduced as a modifier. Various technological methods are used to enhance the adhesiveinteraction at the polymer-filler interface, which leads to a significant change in the structural organisation of a heterogeneous system. For example, mechanochemical activation of the filler can increase the specific surface area of particles by 1.5-2 times.Effect of mechanical impactactivated zeolite was found when it was introduced into polytetrafluoroethylene (PTFE): in the work [22], an increase in deformation-strengthproperties and wear resistance of PCM based on PTFE and activated zeolite was recorded compared to PCM with unactivated zeolite. Structural studies of the PCMs, carried out using scanning electron and atomic force microscopy, infrared spectroscopy and X-ray diffractometry, confirm the transformation of the supramolecular structure of PTFE from lamellar to spherical under the influence of activated zeolite particles. Thus, the dependence of the functional parameters of PCMs on the level of adhesive interaction between the polymer matrix and the filler is obvious, which, in turn, is directly related to such a characteristic of the filler as microporosity.

Perlite has a similar effect. The authors of [23] found a significant effect of perlite nanofiller on the mechanical and thermal properties of composites with a polyethylene matrix, and an increase in the elastic modulus with an increase in the filler concentration was also noted.

A number of research groups have determined the effect of perlite particle size on the mechanical and electrical properties of a polypropylene-based composite and found that a smaller perlite size can lead to an increase in the mechanical properties of polymer composites by reducing shrinkage [24].

It is important that the use of raw materials of different genesis as a filler makes it possible to comprehensively address the issues of composite quality and resource conservation. At the same time, developments in the use of new types of fillers require consideration of the peculiarities of their physical and chemical composition as a factorinfluencing the characteristics of the systems under study and the properties of the composite material. This was the aim of this work in relation to composites based on perlite and zeolite with a polymeric binder.

EXPERIMENTAL

Materials and Research Methods

The object of study was composite materials based on copolymer-filler systems. Perlite of the Berehove deposit and zeolite of the Sokyrnytsia deposit (Transcarpathian region, Ukraine) were chosen as fillers.

Aqueous dispersions of Latex 2012 copolymer and Policril 590 polymer were used as a matrix for the composite (Table 1). At the same time, the choice of binders is based on their presence in a water-dispersed state, which is important for practical implementation in technology, the specified differences in chemical composition and indicators of physical properties.

Table 1 Characteristics of binders

Features.	**Indicators.**	
	Latex 2012	**Policril 590**
Chemical composition	Styrene-butadiene	acrylic
Styrene content, %.	30	-
Physical condition	White aqueous dispersion	White aqueous dispersion
Dry matter content, %.	51.0	53.5-55.0
Particle size, nm	140	200
Viscosity, MPa-s	200	<1000
pH	5.5	5.5-7.5
Temperature (MTU), °C	< 5	0

The IR spectra in the range of 4000-400 cm^{-1} were recorded using a Specord IR-75 spectrophotometer (manufactured by Carl Zeiss, Germany).

The specific surface area was determined by the BET method, which is based on the adsorption of nitrogen vapour at temperature of minus 195 °C by a molecular layer on the surface of the test powder.

The particle size distribution was determined by the sieve method.

The abrasive resistance of the samples was determined in accordance with DSTUB.V.2.7-212:2009 on a Beme-type abrasive wheel, and the mechanical properties - ‘Loading-unloading’ diagrams were studied at room temperature using an automated arrangement consisting of IMASH-20-78, analog-digital converter (ADC), personal computer (PC), and connecting cables. The measurements were performed in a vacuum of 10^{-5} Torr [25].

The technology of manufacturing a composite based on the copolymer-filler system consisted of the following operations:

- Mechanical activation of the filler and binder in a ball mill (20 minutes);
- Forming of blanks (by mould volume) and their maturation (48 hours at room temperature);
- Heat treatment of the workpieces (gradual increase in temperature andholding for 1 hour at 80^{o} C);
- Cold pressing of cylindrical samples with a diameter of 10 mm.

RESULTS AND DISCUSSION

The nature of the filler plays an important role in the creation of polymer composite materials, so it is essential to study their properties first.

The chemical composition of the studied fillers, the results of their X-ray phase analysis, lyophilicity, and the energy state of the surface of the considered fillers are described in our previous work [26].

The experimental fillers have a specific surface area, which is directly related to their mineral phase composition. For example, the specific surface area for zeolite is larger than that of perlite determined by the BET method (11.68 against 2.20 m^2/g) (Table 2).

Table 2 Lyophilicity and energy state of fillers surface

Material	Surface area (BET), m2/g	Coefficient of lyophilicity	Coefficient of wetting during inflow	
			water	benzene
Perlite	2.20	0.189	0.17	0.87
Zeolite	11.68	0.318	0.16	0.52

Perlite has a slightly higher wettability with both water and benzene (0.17 with water, 0.87 with benzene) compared to zeolite (0.16 with water, 0.52 with benzene).

The coefficient of lyophilicity has an inverse relationship and is higher for zeolite (0.318) compared to (0.189) for perlite.

The key parameter that determines the choice of a filler is its dispersion and thepercentage of different fractions, i.e., the particle size distribution (Fig. 1).

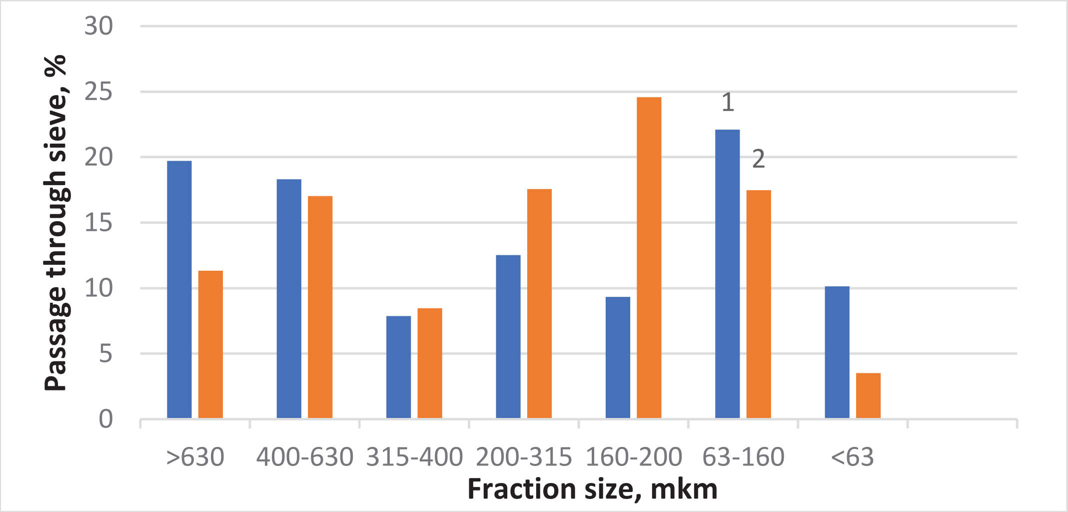


Fig. 1. Particle distribution by fractions of perlite (1) and zeolite (2) samples

It was found that among the studied fillers, the highest content was found in theparticle size fraction of 63-160 μm for zeolite, which is 22.1 %. Perlite has a maximumcontent of 27.58 % in the larger fraction of 200-315 μm.

In order to study the chemical composition of the fillers and their interaction with the binder in more detail, infrared spectroscopy was performed (Fig. 2).

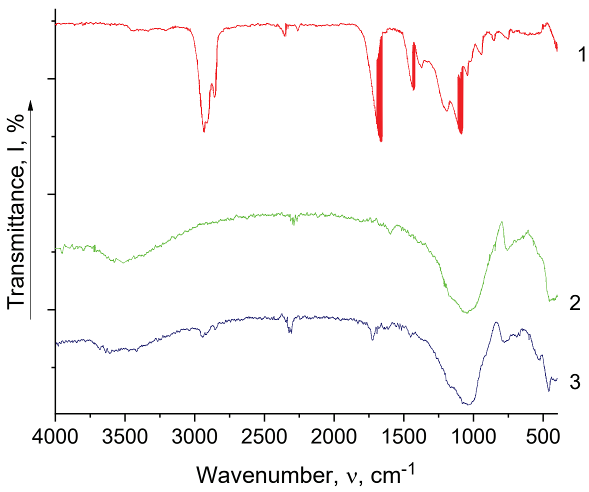


Fig. 2. a. Infrared spectroscopy of samples:
1 - Policril 590, 2 - Perlite, 3 - Perlite + Policril 590

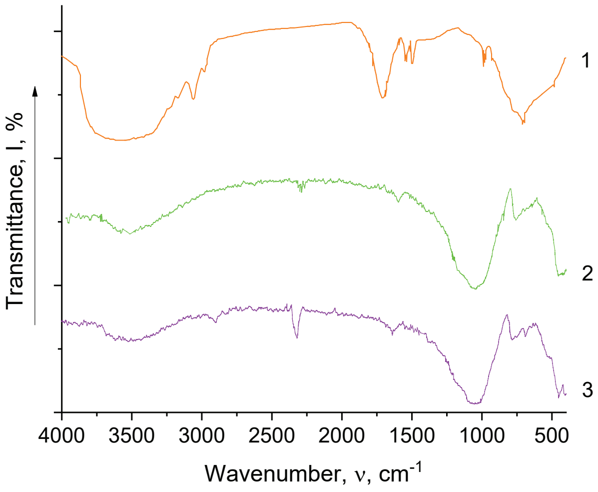


Fig. 2. b. Infrared spectroscopy of the samples:
1 - Latex 2012, 2 - Perlite; 3 - Perlite + Latex 2012

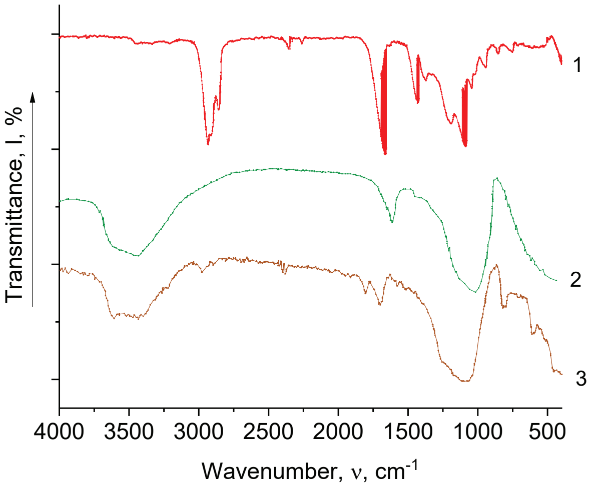


Fig. 2. c. Infrared spectroscopy of the samples:
1 - Policril 590, 2 - Zeolite, 3 - Zeolite + Policril 590

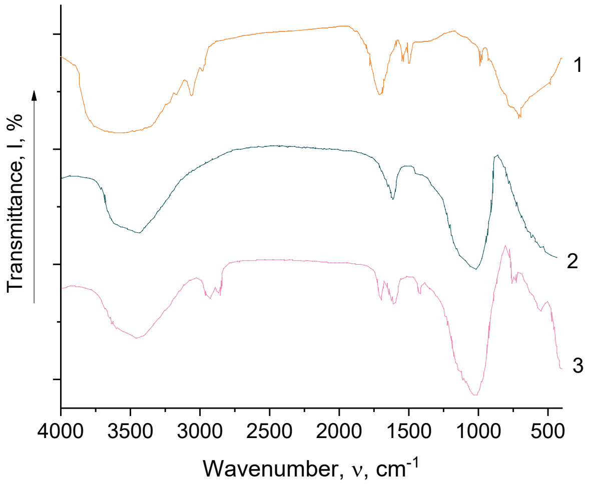


Fig. 2. d. Infrared spectroscopy of the samples:
- Latex 2012, 2 - Zeolite, 3 - Zeolite + Latex 2012

These features of the surface properties of perlite particles correlate with the results of infrared spectroscopic analysis, which showed the presence and characteristics of simple compounds, functional groups, and chemical bonds (Fig. 2. a, curve 2). Among the latter, first of all, it is necessary to note the vibrations of the structure-forming bridging and non-bridging Si-O-Si bonds (valence interval 640-690 cm^{-1}, Si-O-Al (710-780 cm^{-1}). There are also characteristic absorption bands responsible for the deformation vibrations of the Si-O^{-} (440-436 cm^{-1}) and Si-O-Al (507-593 cm^{-1}) bonds.

Analysis of the position of the absorption band maxima characteristic of the valence vibrations of Si-O-Si bonds on the IR spectra showed the presence of a relatively narrow interval (1000-1080 cm^{-1}) for perlite.

It should be noted that there are absorption bands responsible for differential vibrations of adsorbed water at 1620-1640 cm^{-1}. As for zeolite (Fig. 2. c, curve 2), the intense band appearing in the range of 3400-3500 cm^{-1} is the result of the vibration of OH groups of water. The band appearing in the range of 1600-1650 cm^{-1} is the result of molecularly bound water in the structure. The vibration ranges at a distance of 950-1250 cm^{-1} are the result of structural units of the alumina-silicate lattice of Si(Al)-O zeolite. Based on this structural analysis, it is confirmed that this is a nanoporous aluminosilicate material with a defined structure and the presence of free and chemically bound water.

When studying the interaction between the filler and binder using infrared spectroscopy (Fig. 2. a, curve 1), it was found that the acrylic dispersion of Policril 590 is characterised by an absorption band at 1670 cm^{-1}, which corresponds to the monomeric links of acrylate [27] and is the result of asymmetric and symmetric valence vibrations of C=O in the carboxyl group. Vibrational vibrations of the C=C bond correspond to the absorption band at 1435 cm^{-1}, the presence of a CH bond is indicated by the absorption bands at 1100 cm^{-1} [28]. As for the Policril 590 – Perlite system (Fig. 2 a, curve 3), the curve is more consistent with the absorption curve characteristic of perlite, but there are certain nuances: in particular, a slight absorption band at 2280 cm^{-1}, corresponding to the C=C bond, and at 1740 cm^{-1}, characteristic of the C=O bond of the polymer, appeared, but the intensity of these bands is insignificant compared to the original Policril 590 with some shift, which may confirm the presenceof interaction in the system. The same patterns are observed when using zeolite (Fig. 2. c, curve 3).

As for the styrene-butadiene dispersion Latex 2012 (Fig. 2. d, curve 1), the saturation band at 2980 cm^{-1} is responsible for the CH compound in the aromatic ring, and the saturation band at 1525 cm^{-1} is directly responsible for the presence of the ring itself. The absorption band at 2852 cm^{-1} confirms the presence of the CH_2 group, and at 1500cm^{-1} – the presence of the butadiene double bond. [29].

For the composite material based on the Latex 2012 – Zeolite system (Fig. 2. d, curve 3), we assume that physical sorption occurs, as evidenced by the presence of a band of 2920 cm^{-1} corresponding to the -CH bond of the styrene aromatic ring, and a decrease in the intensity of the shift of the band 1525 cm^{-1} C=C characteristic of the butadiene component.

Similar patterns are observed when using perlite (Fig. 2. b, curve 3).

Samples of the composite material were obtained on the basis of binary systemsof the studied fillers (perlite and zeolite) with a binder (Table 3). The concentration offillers with a particle size of ≤ 1 mm varied from 65 to 90 wt.%, and the concentration of binders - from 35 to 10 wt.%, respectively.

Table 3 Composition and properties of the composite material

Composition of the composite	Filler concentration, C, wt. %	Indicators				
		Water absorption after 24 hours, %	Open porosity, %	Total porosity, %	Average density, g/cm^3	Abrasion resistance, g/cm^2
Policril 590 + Perlite	65	2.63	3.58	7.67	1.36	0.026
	75	6.45	9.02	17.70	1.40	0.026
	85	7.08	11.13	30.99	1.57	0.026
	90	8.41	13.55	35.72	1.61	0.034
Latex 2012 + Perlite	65	3.42	4.67	5.42	1.37	0.015
	75	4.16	5.96	13.87	1.43	0.016
	85	7.89	12.67	24.43	1.61	0.040
	90	8.59	14.38	31.73	1.67	0.060
Policril 590 + Zeolite	65	3.35	4.84	11.50	1.44	0.011
	75	3.73	5.64	20.61	1.51	0.013
	85	8.21	13.27	30.01	1.61	0.017
	90	9.28	15.46	34.03	1.66	0.022
Latex 2012 + Zeolite	65	3.91	5.29	7.23	1.35	0.012
	75	6.36	9.32	19.76	1.46	0.013
	85	13.90	20.41	24.70	1.47	0.026
	90	14.16	21.35	28.79	1.51	0.045

According to the test results, the physical and mechanical properties of composites significantly depend on the type and concentration of the filler. When using both fillers, an increase in water absorption and a decrease in the average density are observed with an increase in their concentration. At the same time, the degree of change in these indicators in the specified range of filler concentrations is significantly different. Thus, when comparing composites based on acrylic dispersion (Policril 590)using different fillers, it should be noted that water absorption when using zeolite with an increase in its concentration varies within 3.35-9.28 wt.%, which is slightly higher compared to composites using perlite (2.63-8.41 %). When replacing the binder with Latex 2012, this trend is observed, but the values increase slightly. Thus, for composites with perlite, they range from 3.42-8.59 wt.% against 3.91-14.16 wt.% when using zeolite.

The analysis of the structure of the prototypes showed (Table 3) that there is a linear dependence of the increase in porosity on the concentration of the filler.

Thus, when comparing composites based on Policril 590 dispersion using perlite,it should be noted that the open porosity with an increase in its concentration varies within 3.58-13.55 %, which is slightly lower compared to composites with zeolite (4.84-15.46 %). When replacing the binder with Latex 2012, this trend persists, but the values increase slightly: 4.67-14.38 % for perlite versus 5.29-21.35 % for zeolite.

For composites using perlite, when binders are replaced with Policril 590 by Latex 2012, the initial values of the total porosity increase from 7.64-35.72 % against 5.42-31.73 %. When using zeolite and replacing the binders from Policril 590 with Latex 2012, the values of the total porosity decrease from 11.50-34.03 % to 7.23-28.76 %, respectively.

Obviously, these features indicate differences in the pore structure of the studied composites, since the analysis of the sample structure showed (Fig. 3) that the use of abinder with a higher viscosity (Policril 590) at the same filler concentration leads to an increase in the porosity of the composite samples.

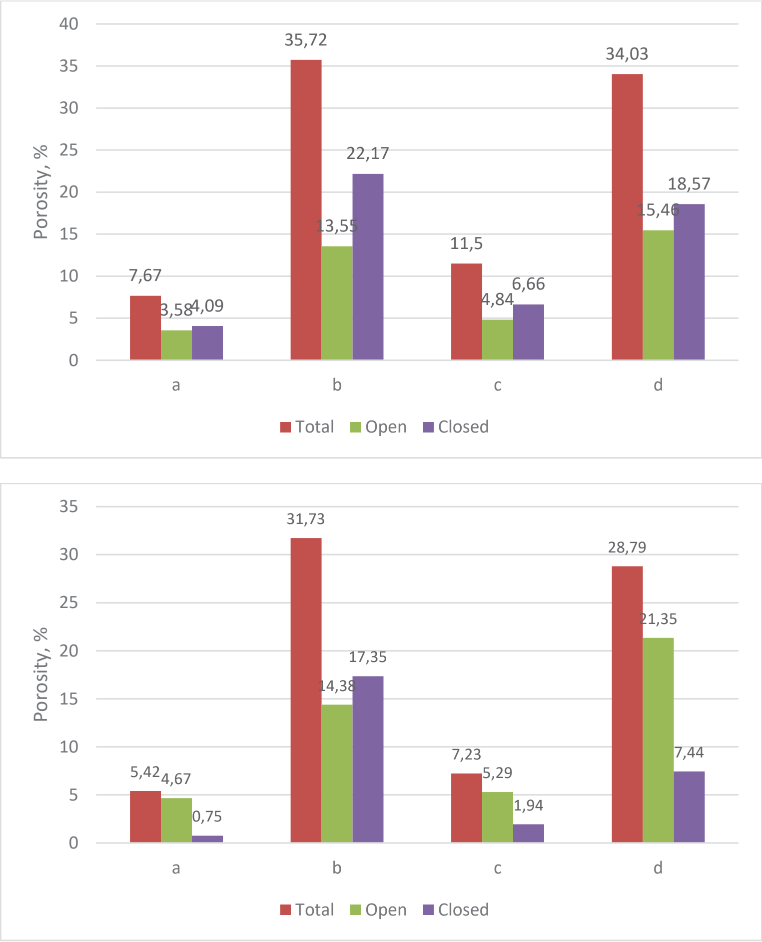


Fig. 3. Porosity of composites based on Policril 590 (1) and Latex 2012 (2) at the concentration of perlite 65.0 (a), 90.0 wt.% (b) and zeolite 65.0 (c), 90.0 wt.% (d)

Among the performance characteristics of the studied composites, the abrasion indicators indicate increased abrasion resistance of the material.

We also studied the mechanical properties of composites using polymeric binders and two types of fillers - perlite and zeolite - under uniaxial compression of thesamples. Figs. 4-5 show the load-strain diagrams for composites with perlite filler using two types of binders, Policril 590 and Latex 2012.

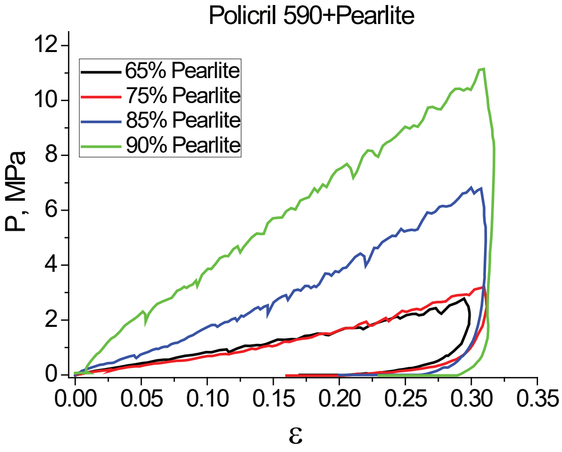


Fig. 4. Dependence of deformation on load for CM system Policril 590 + Perlite

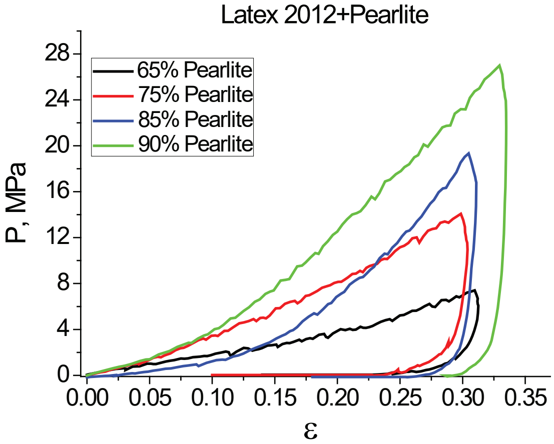


Fig. 5. Dependence of deformation on load for CM system Latex 2012 + Perlite

The elastic ε_{el} and residual (plastic) ε_{pl} strains and Young's modulus $E=P/\varepsilon_{el}$ were determined from the load-strain diagrams, the data are presented in Table 3. For a comparative analysis of the mechanical properties of different types of CM samples, the values of the ultimate load were chosen so that the total deformation of the sampleswas approximately the same, $\varepsilon_{total} = \varepsilon_{el} + \varepsilon_{pl} \approx 0.30$.

As can be seen from the data presented, with an increase in the content of perlite in composites of both types (with Policril 590 and Latex 2012 binders), the value of the ultimate load increases, which indicates an improvement in the strength characteristics. It is worth noting that these composites have rather high residual strains (ε_{pl}), the value of which increases with increasing perlite content in the composites, since the ultimate load for high composites filling increases.

Similarly, as can be seen from Table 4, the increase in the perlite content in the composites materials also increases the Young's modulus, despite the rather significant residual deformations in these composites materials. Moreover, the Young's modulus is much higher for the composites materials with Latex 2012 binder, increasing from 41.9 MPa for the perlite content of 65 wt% to 677.5 MPa for the composites materials with 90 wt% perlite.

Table 4 Strength and elastic characteristics of samples using Perlit

Perlite content, wt.%.	Maximum load, P MPa	εtotal	εpl	εel	Young's modulus, E, MPa
CM system Policril 590 + Perlite					
65	2.77	0.29	0.17	0.12	23.1
75	3.18	0.31	0.16	0.15	20
85	6.85	0.31	0.20	0.11	62.3
90	11.20	0.31	0.23	0.08	140.0
CM system Latex 2012 + Perlite					
65	7.12	0.31	0.14	0.17	41.9
75	14.1	0.30	0.10	0.20	70.5
85	19.30	0.30	0.18	0.12	160.0
90	27.1	0.33	0.29	0.04	677.5

Similar results of the study of mechanical characteristics for the CM with zeolite filler are presented in Figs. 6-7 and Table 5. For these composites, there is also an increase in Young's modulus with increasing filler content, but this dependence is not monotonic: for Policril 590 + 90 wt.% Zeolite composites, a decrease in both the ultimate load for deformations ε_{total} ≈0.30 and the effective Young's modulus was observed, which may indicate a deterioration in the strength characteristics of composites with such a high filling.

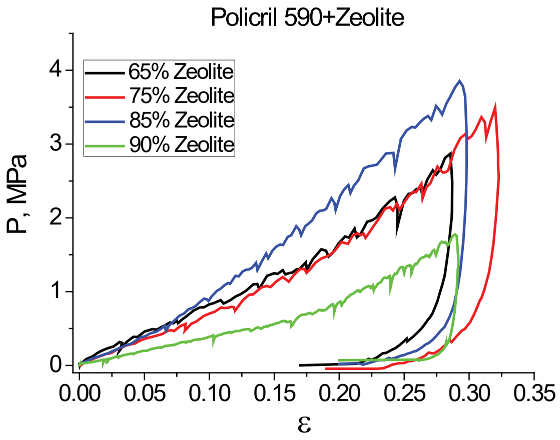


Fig. 6. Dependence of deformation on the load for CM system Policril 590 + Zeolite

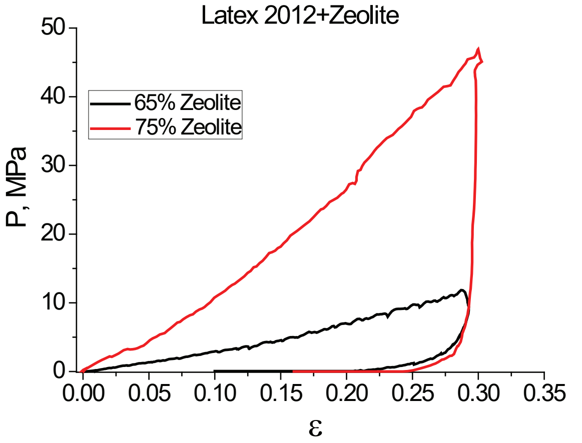
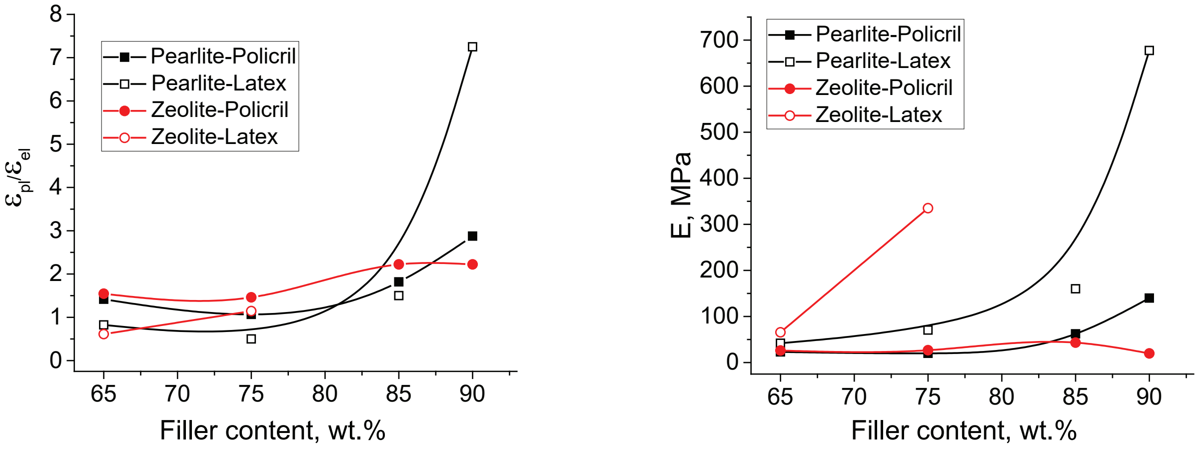


Fig. 7. Dependence of deformation on load for CM system Latex 2012 + Zeolite

Table 5 Strength and elastic characteristics of samples using Zeolite

Zeolite content, wt.%.	Maximum load, P MPa	εtotal	εpl	εel	Young's modulus, E, MPa
CM system Policril 590 + Zeolite					
65	2.88	0.29	0.17	0.11	26.2
75	3.48	0.32	0.19	0.13	26.7
85	3.90	0.29	0.20	0.09	43.3
90	1.77	0.29	0.20	0.09	19.7
CM system Latex 2012 + Zeolite					
65	11.8	0.29	0.11	0.18	65.6
75	46.9	0.30	0.16	0.14	335.0

Fig. 8 shows comparative graphs for the ratio between plastic and elastic deformations ε_{pl} /ε_{el} at the maximum ultimate load and the effective Young's modulus E for all the studied CM samples depending on the filler concentration and type of polymer binder.



a) *b)*

Fig. 8. The ratio between plastic and elastic deformations and effective Young's module versus filler content for CM systems Binder + Filler

As can be seen from Figures 8 a, b, with increasing filler content, the value of $\varepsilon_{pl}/\varepsilon_{el}$ gradually increases, which correlates with an increase in the ultimate load P_{max}, and this increase is most dramatic for the Latex 2012 + Perlite system. The concentration dependence of the effective Young's modulus *E* for these systems is fully correlated with the data for $\varepsilon_{pl}/\varepsilon_{el}$, since $E \sim 1/\varepsilon_{el}$: the maximum effective Young's modulus was observed for CM Latex 2012 + 90 wt.% Perlite.

Comparing all the studied systems with each other, it can be concluded that themain role in determining the mechanical characteristics of composites is played by the type of binder and filler concentration. For example, CM with Latex 2012 binder have a significantly higher Young's modulus compared to composites based on Policril 590, which increases with the increase in filler content (perlite or zeolite) and indicatesan improvement in strength characteristics.

CONCLUSIONS

1. The developed composite materials, intended primarily for use in the field of construction, differ from the well-known high concentration of fillers, which are by-products of the extraction of volcanic rocks, which contributes to a comprehensive solution to the issues of expanding the raw material base for the production of polymer composites and resource saving.
2. The peculiarities of using perlite and zeolite as fillers in the concentration of 65-90 wt. % for the manufacture of composite materials using Latex 2012 copolymer and Policril 590 polymer as a matrix are studied.
3. The differences in the chemical and mineralogical composition and surface properties of perlite and zeolite as factors influencing the formation of the structure and indicators of physical and mechanical properties of the developed composites aredetermined.
4. The influence of high concentration of fillers and binder types on the pore structure and properties of the composite material is considered. The possibility of providing increased abrasive resistance of composites at controlled water absorption in the range of 2.63-14.16 wt.%, open porosity of 3.58-21.35 %, residual deformation of 0.1-0.3, and Young's modulus of 19.7-677.5 MPa was noted.

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IZVOD

KOMPARATIVNO PROUČAVANJE RAZLIČITIH VULKANSKIH MATERIJALA KAO PUNILA U POLIMERNIM KOMPOZITIMA

Predmet studije bili su kompozitni materijali koji koriste stene vulkanskog porekla kao punilo (60-90 tež.%) i disperziju Latex 2012 i Policril 590 kao a matrica. Osobenosti hemijskih i mineraloških sastava i površinska svojstva perlita i zeolita kao interakcija sa faktorima u formaciji prikazane su u kompozitnim strukturama . Utvrđene su razlike u koeficijentima liofilnosti i efektivne specifične površine zeolita i perlita, koje su 0,318 prema 0,189 i 11,68 prema 2,20 m^2/g, respektivno. Uticaj visoke koncentracije punila na formiranje strukture pora i indikatora i sastava i mehaničkih i fizičkih svojstava je procenjeno. Mogućnost prilagođavanja svojstva kompozita u sledećem opsegu je uspostavljena: voda apsorpcija u 4,16 mas. %, otvorena poroznost 3,58-21,35 %, rezidualna deformacija 0,1-0,3 , Youngov modul 19,7-677,5 MPa.

Ključne reči: *kompozit, punilo, perlit, zeolit, koncentracija, kopolimer, poroznost, svojstva.*

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