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POLYMER-SILICATE COMPOSITES WITH MODIFIED MINERALS

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Abstract. New polymer composites on the basis of epoxy resin and different fine dispersed mineral powders (andesite, bentonite, diatomite, liquid glass and quartz sand) were obtained and their mechanical (ultimate strength), thermal (temperature dependence of the softening) and water absorption properties were investigated. It was established that all properties of these materials were essentially improved, when the same fillers modified by tetraetoxysilane (TEOS) were used. It was experimentally shown that composites containing binary fillers - diatomite and andesite at definite ratio - are characterized with so called synergistic effect - the increase in maximal physical or chemical properties. Separately the experimental data on coefficients of reagents diffusion, sorption and penetration, mechanical strengthening, coefficient of heat conductivity and stability to sulfur acid have been investigated. It was shown that exploitation properties of these materials were essentially better in comparison with well known silicate composites. Created materials were characterized by low shrinkage and also low internal stress after hardening. Using the method of mathematical modeling of experiment the optimization of the composition of obtained materials has been performed. Experimental results were explained in terms of structural peculiarities of polymer materials.

Keywords: epoxy resin, mineral filler, polymer composite, mechanical and thermal properties, water absorption.

1. Introduction

The polymer composites attract great attention of scientists and engineers. High monitoring of the exploitation properties in wide interval, high durability,

stability to aggressive media, lightness, easy technology of obtaining and finally low cost are the fundamental characteristics of these materials. The noted factors lead to the high competitiveness to such tradition materials as metals, ceramics, wood and skin. Currently the polymer (synthetic or natural) composites with different mineral fillers are widespread [1-7]. Thanks to these fillers many properties of the composites are improved: the durability and rigidity increase, the shrinkage during the hardening process and water absorption decrease, thermal stability, fire proof and dielectric properties increase and finally the price of composites is reduced [3-5]. At the same time it must be noted that the high content of mineral fillers leads to some impair of different physical properties of composites. Therefore the attention of the scientists is attracted to the substances, which would remove the mentioned leaks. It is known that silicon organic substances (both low and high molecular) as "soft materials" reveal hydrophobic properties, high elasticity and durability in a wide range of filling and temperatures and consequently introduction of these materials to the polymer blends, in general, can increase compatibility of ingredients and respectively increase the mineral filler concentration in the composites [6, 7]. Since the price of mineral fillers is commonly very low than that of polymer (binder), the main investigation in the field of composite technology is directed to creation of high filled composite materials. If we foresee that the composites may be obtained on the basis of different natural or artificial wastes, the application of these materials will be connected with great economical effect.

Application of the method of polymerization filling (polymerization on the filler particles) as a rule leads to obtaining of the composites with high mechanical properties due to formation of the chemical bonds on the filler surfaces between the polymer and active groups on the particles surfaces [3, 4]. This method consists of several stages and is relatively expensive.

At the same time the polymer layered silicate nanocomposites (PLSN) are rather popular [5-7]. There are three main methods of PLSN synthesis: in situ solution intercalation, polymerization, and melt processing (or melt blending). The fourth method, using sol-gel techniques, can in principle be applied, but it is not

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often utilized because of the high temperatures and the tendency of inorganic components to be aggregated. Each of three main approaches may yield exfoliated, intercalated, or mixed exfoliated and intercalated structures. The degree of exfoliation *versus* intercalation depends on a host of experimental conditions, such as monomer type, solvents, temperatures, *etc.*

The purpose of the presented work is the investigation of the effect of modifying the minerals andesite, bentonite, diatomite, quartz sand and liquid glass by tetraetoxisilane. On the other hand, we attempted enhancing the composites with the use of the effect of synergistic effect (enhancing composite characteristics at definite proportion of two or more fillers types) on some physical properties of composites based on epoxy resin.

2. Experimental

The organic solvents were purified by drying and distillation. The purity of starting compounds was controlled by LKhM-8-MD gas liquid chromatography; phase SKTF-100 (10%, the NAW chromosorb, carrier gas He, 2 m column). Chemical analysis of obtained products was conducted on the spectrometer FTIR. Spectra were recorded on Jasco FTIR-4200 device. For modifying the minerals two types of silicon-organic compounds - ethylsilicate (ES) and tetraetoxisilane (TEOS) - were used. The silanization reaction of minerals surface with ES and TEOS was carried out by means of three-necked flask supplied with a mechanical mixer, thermometer and dropping funnel. To obtain the product modified by 3 wt % of modifiers a solution of finely grind mineral powder (50 g) in 80 ml of anhydrous toluene was added to the solution of modifier (1.5 g, 0.0072 mol) in 5 ml toluene. The reaction mixture was heated at the boiling temperature of the used toluene. Than the solid reaction product was filtrated, the solvents (toluene and ethyl alcohol) were eliminated and the reaction product was dried up to a constant mass in vacuum. Other products modified by 5 % modifier were produced by the same method.

The composites based on epoxy resin of type ED-20 with different content of a filler were obtained after careful wet mixing of components in a mixer. To the blend of ingredients the hardener (polyethylene - polyamine) was added and placed in the spatial (in accordance with ISO standards) forms for hardening at room temperature during 24 h. After these procedures the samples were exposed to a temperature treatment at 393 K during 4 h. The concentration of fillers was changed in the range of 10–60 wt %.

Following characteristics of the obtained composites were defined: ultimate strength (on the

stretching apparatus of the "Instron" type), temperature dependence of the softening (using Vicat method) and water absorption (using standard method).

3. Results and Discussion

3.1. Composites on the Basis of Epoxy Resin and Bentonite

From mechanical parameters of the composites with bentonite the ultimate strengthening was investigated. The curves presented in Fig. 1 allow us to make a conception that this parameter increases to the maximal values and then decreases. The position of the maxima is defined with the content of the composite. The curve 1 corresponds to the composite containing the filler without a modifier. The shape of this curve reflects the well known dependence for filled polymer composites. The mechanical strengthening of this composite with unmodified bentonite has the maximum at relatively low concentration of the filler (10-20 wt %), while this maximum for analogical composites modified with TEOS (3 and 5 wt %) is observed at higher concentrations of the filler (Fig.1, curves 2 and 3). This result has practically important meaning - the higher is mineral filler concentration in the composite the lower is its cost. The *curve* 3 of this figure shows that the maximum in this case must be at filler concentrations higher than 40 wt %.

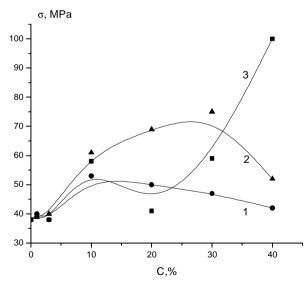


Fig. 1. Dependence of the ultimate strength of the composite ED-20 + bentonite on the concentration of the filler: unmodified bentonite (1); bentonite modified with 3 wt % of TEOS (2) and bentonite modified with 5 wt % of TEOS

The curves of mechanical strengthening dependence for the composites with modified and

unmodified bentonite show that modifier molecules displaced on the filler particle surface increase their activity (expressed with enhancing composite material due to the chemical reactions between active groups of ingredients) till definite concentrations of the filler. At higher values the mechanical characteristics of the composite decrease. One of the main reasons of this phenomenon is the formation of a number of structural defects (mechanical cracks, empties) due to the formation of the surfaces non reacted with organic part of the binder, the amount of which increases with the increase in filler concentration. It is due to the so called effect of high filling (or the Rebinder effect).

In the composites with modified fillers the characteristic maxima on the curve of ultimate strengthening dependence on the filler concentration the modifier molecules enhance the interaction between heterogeneous phases on the one hand and absorb the mechanical stresses in composite body at hardening on the other one. Therefore the maxima on the curves for the composites with modified fillers are shifted to higher concentrations of the filler. However at following increasing of filling content in the composite the modifier phase increases, which is usually a soft phase and plays the role of structural defects. Increasing of this phase leads to softening or decreasing of composite mechanical strengthening.

The phenomena described above appear at investigation of the composite thermal mechanical properties.

From the curves presented in Fig. 2 it is seen that TEOS modified bentonite as a filler in the composite to a definite extent affects the thermal stability of the composite. Namely the softening of the composites containing TEOS modified bentonite begins at relatively high temperatures compared with those containing

mu(t-)01Xrl 3

Fig. 2. Dependence of the softening on the temperature for the composite modified by 5 wt % (1) and 3 wt % of TEOS (2), and for the composite with unmodified filler (3). Concentration of the bentonite in all samples 30 wt %

T,K

360

420

-6

300

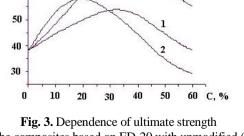
unmodified filler. This result is in a good agreement with those obtained at investigation of the mechanical properties of corresponding materials.

Enhancing of the compatibility of the ingredients in the TEOS modified composites is also reflected in the characteristics of the composites water absorption. In accordance with Table 1 the composites with modified filler show higher water stability than analogues with unmodified fillers. It can be proposed that the composites modified with filler with higher concentration of the same modifier will be more hydrophobic than presented samples, because the named composites with high concentration of modifier as high hydrophobic component will be more waterproof.

3.2. Composites on the Basis of Epoxy Resin, Diatomite and Andesite

The dependence of ultimate strength on the content of diatomite (modified and unmodified) presented in Fig. 3 shows that it has an extreme character. However the positions of corresponding curves maxima essentially depend on amount of modified agent TEOS. The general view of these dependences is in full conformity with well known dependence σ –*C* [8]. The shift of the maximum at the curve for the composites containing 5 % and 3 % of modifier is due to increasing amount of the bonds between filler particles and macromolecules at increased concentration of the filler.

Investigation of composite softening temperature was carried out by the Vicat apparatus. Fig. 4 shows the temperature dependence of the indentor deepening to the mass of the sample for composites with fixed (20 wt %) concentration of unmodified and TEOS modified.



S, MP:

80

70 60

Fig. 3. Dependence of ultimate strength of the composites based on ED-20 with unmodified (1) and modified by 3 wt % (2), and 5 wt % (3) of TEOS diatomite

Table 1

The water absorption of the composites containing unmodified and TEOS modified bentonite with different concentrations of TEOS

Concentration of bentonite %	Concentration of TEOS, %	Water-absorption, %
10	0	0
20	0	0.41
40	0	0.56
10	3	0.108
10	5	0.102
20	3	0.131
20	5	0.118

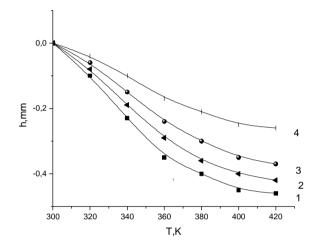


Fig. 4. Temperature dependence of the indentor deepening in the sample for composites containing 0 (1); 20 (2); 20 wt % modified by 3% TEOS (3), 20 wt % modified by 5wt % TEOS (4) diatomite

Based on the character of curves (see Fig. 4) it may be proposed that the composites containing diatomite modified by TEOS possesses higher thermo-stability than in the case of analogous composites with unmodified filler. Probably the presence of increased interactions between macromolecules and filler particles due to modify agent leads to increasing the thermal stability of composites with modified diatomite.

Effect of silane modifier on the investigated polymer composites is also observed in the water absorption. In accordance with Fig. 5 this parameter increases with the increase of filler content. However if the composites contain diatomite modified by TEOS this dependence becomes weak.

The investigation of binary fillers effect on the properties of the composites with the same polymer basis (ED-20) has been conducted. Two types of minerals – diatomite and andesite with different ratios – were used as fillers. It was interesting to establish the effect both of the fillers ratio and effect of ES modifier on the same properties of the polymer composites.

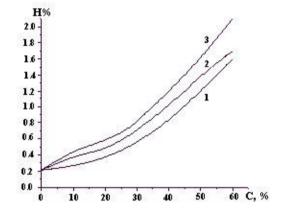


Fig. 5. Dependence of the water-absorption on the concentration of filler in the composites based on epoxy resin containing diatomite modified by 5 wt % (1) and 3 wt % (2) TEOS and unmodified (3) one

The curves presented in Fig. 6 show the effect of TEOS modifier on ultimate strength. The maximum of noted effect corresponds to the diatomite/andesite ratio of about 20/30. Probably the microstructure of such a composite corresponds to an optimal distribution of filler particles in the polymer matrix at minimal inner energy of statistical equilibration, at which the concentration of empties is minimal because of dense disposition of the composite components. It is known that such structures consists of minimal amount both of micro and macro structural defects [8].

Such approach to a microstructure of composites with optimal ratio of the composite ingredients allows supposing that these composites would possess high mechanical properties, thermal stability and low water-absorption. Moreover, the composites with the same concentrations of the fillers modified by ES possess all the noted above properties better than composites with unmodified by ES binary fillers, which may be proposed early (Figs. 6-8). Indeed the curves in Figs. 6-8 show that the maximal ultimate strength, thermal stability and simultaneously hydrophobicity correspond to composites with the same ratio of fillers to which the maximal density corresponds.

Fig. 6. Dependence of the ultimate strength on the concentration of diatomite in binary fillers with andesite: unmodified filler (1) and modified by 5 wt % of TEOS (2) for composites based on epoxy resin. Full concentration of binary filler in the composites is 50 wt %

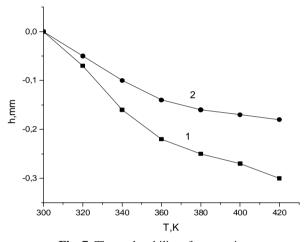


Fig. 7. Thermal stability of composites with binary fillers at diatomite/andesite ratio equal to 20/30

The obtained experimental results may be explained in terms of composite structure peculiarities.

Silane molecules displaced on the surface of diatomite and andesite particles lead to activation of them and participate in chemical reactions between active groups of ES (hydroxyl) and homopolymer (epoxy group). Silane molecules create the "buffer" zones between the filler and the homopolymer. This phenomenon may be one of the reasons of increasing the composites strengthening in comparison with composites containing unmodified fillers. The composites with modified diatomite display higher compatibility of the components than in the case of the same composites with unmodified filler. The modified filler has a stronger contact with polymer matrix (due to silane modifier) than unmodified diatomite. Therefore mechanical stresses formed in the composites by stretching or compressing forces are absorbed effectively by relatively soft silane phases, i.e. the development of micro defects in the carbon

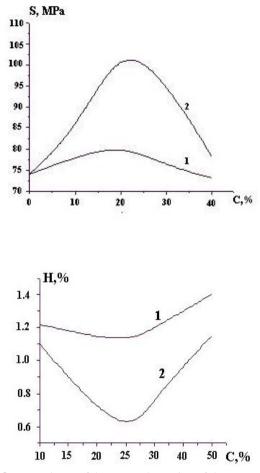


Fig. 8. Dependence of the water-absorption of the epoxy resin based composites on the concentration of diatomite in binary fillers with andesite: unmodified (1) and modified by 5 wt % of TEOS (2) fillers. Total concentration of binary fillers in composites is 50 wt %

chain polymer matrix of composite districts and finishes in silane a part of material the rigidity of which decreases.

The structural peculiarities of composites also influence thermo-mechanical properties of the materials. It is clear that softening of TEOS modified composites begins at relatively high temperatures. This phenomenon is in good correlation with mechanical strength of the corresponding composites. Of course the modified filler has stronger interactions (due to modifier) with epoxy polymer molecules, than unmodified filler.

The amplified competition of the filler particles with TEOS macromolecules influences also the characteristics of water absorption. In general loosening of micro-structure because of micro empty areas is due to the increasing of filler content. Formation of such defects in the microstructure of composite promotes the water absorption processes. Water absorption of composites with modified diatomite is to some extent lower than that of the composite with unmodified filler. The decreasing of water absorption of composites containing silane compound is the result of hydrophobic properties.

Composites with binary fillers possess so called synergistic effect – non-additive increasing of technical characteristics of composites at definite ratio of fillers, which is due to creation of the dense distribution of ingredients in composites.

3.3. Acid Proof Polymer Silicate

Composites

Firstly polymer-silicate composites stable to inorganic acids were obtained on the basis of liquid glass and furan compounds [9, 10]. They were characterized by rather high stability to inorganic acids. The furan resin composites containing additionally polyethylene show higher physical and chemical characteristics [11]. Many technical characteristics of the polymer-silicate composites are better than for different acid stable lubricants – protective coatings [12, 13].

We attempt to improve acid stable properties of existed polymer-silicate composites by selection of ingredient types and their concentrations in the composites.

The liquid glass with module M = 2.8-2.9 with hardener Na₂SiF₆, epoxy-resin of ED-20 type with polyethylene-polyamine hardener and wide spread in Georgia minerals: andesite and quartz sand were used for obtaining of the polymer-silicate composite. Detail description of the composite obtaining method is given in [14]. Experimentally it was established that the right selection (optimization) of ingredients and their concentration is the main way to ensure the obtaining of the composites with best physical and chemical properties. Thanks to the mathematical modelling method [15] we got the composites, containing the ingredients with optimal concentrations (Table 2).

The properties of the composites including the ingredients in accordance with Table 2 are presented in Table 3.

The data of Table 3 show that the numerical values of the composites, developed by us, are essentially better than those for the existed analogues.

Testing of the new polymer-silicate materials with optimal content of ingredients shows that the stability to sulfur acid of the materials with concentrations of 0.5-10 % equals to 0.7-0.85 % and when concentration of this acid is equal to 30-50 %, this parameter is not more than 0.95-1.1 %. Moreover, it was shown by us that the penetration of the aggressive agents (acids, alkalis, air) in the polymer-silicate materials is better in comparison with existed acid-proof silicate materials even in the case of diluted sulfur acid. These indexes are, naturally, rather high and indicate to the high chemical stability of the composites proposed in this work.

The adhesion forces of new PSM to different metals were defined. It was shown that this parameter reaches 3.5–4.5 MPa and the shrinkage for PSM is 1.5 times lower than for the analog noted above. Development of the inner stress forces in time is shown in Fig. 9.

Table 2

Ingredient	Weight part	
Liquid glass	100	
Epoxy-resin ED-20	23–25	
Polyethylene polyamine	2.5–2.7	
Na ₂ SiF ₆	10–12	
Andesite flour	180–200	
Quartz sand	180–200	

Optimal composition of the polymer-silicate composites

Table 3

Some characteristics of polymer silicate composite and silicate lubricant material

Characteristics		Polymer-silicate composite	Silicate lubricant material
Density, g/cm ³		1.65	1.8
Ultimate strength, MPa	at compressing	72–77	25–27
	at elongation	6–7	2.5–3
	at bending	20–23	4–5
Thermal coefficient of expanding, 10 ⁻⁶ grad ⁻¹		5–6	8
Thermal conductivity, Wt/m		0.7–0.8	0.7–1
Water absorption, %		3–5	12–15

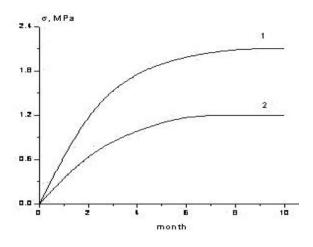


Fig. 9. Development of the inner stress forces in time in the existed acid-proof silicate material (1) and PSM (2)

The curves presented in Fig. 9 show that in both cases inner stresses at hardening of the composites are developed at first intensively, but later they are saturated. However, the residual stresses in the PSM are almost two times lower than in the analog.

PSC described above may be used both in the building engineering and in thermoelectric plants as the coatings preventing the surfaces of different constructions from aggressive liquids or air action.

3.4. Effect of Microstructure of the Composites Based on Epoxy Resin on their Properties

Experimental material presented above allows to express some concepts about considered composite microstructure and its influence on the material properties.

It is well known that after hardening of diane epoxy resin the network with 3D dimension is formed,

which is not soluble, as a rule, in organic solvents (gelfraction is formed). The process of hardening is connected with the formation of the mechanical stresses in the final products, under action of which different types of cracks, micro- and macroempties are formed in the hardened products. These products provide the decrease of all operational practically properties (mechanical strengthening, thermal stability, water and other aggressive media absorption). So the microstructure of hardened epoxy material may be presented as three dimension network of polymer matrix filled with various small or big defects, mainly the empties. Such conception can be presented schematically as the picture in Fig. 10.

The mechanical strengthening of the composite on the basis of different minerals and epoxy resin increases at increasing the filler concentration (till definite values). Practically the dependence of the composite mechanical strengthening coincides with the maximum of the polymer composite with inorganic (e.g. mineral) filler. When we introduce the high dispersed mineral modified with tetraetoxisilane, the changes are appeared at relatively low concentration of modifier. The analogues composite containing bentonite modified by 3 wt % TEOS the maximum on the curve of ultimate strength dependence on the filler concentration shifts to relatively high concentration of bentonite. This phenomenon occurs due to increasing the composite ingredients compatibility. TEOS decreases fragility of composites and increases at the same time the compatibility of ingredients, decreases the formation of such defects, as empties (Fig. 11). At high concentrations of bentonite the so called effect of high filling appears, which is decreased under influence of the modifier. The modifier molecules at low concentrations (till formation of self phase) envelop the fillers particles and form the buffer zone between polymer matrix and filler (Fig. 12). At higher concentrations of modifier the formation of self phase of the modifier takes place.

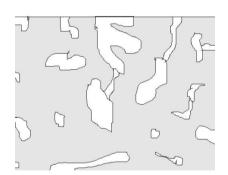


Fig. 10. Model imagination of the hardened pure epoxy resin. White areas – empties, grey area – polymer matrix

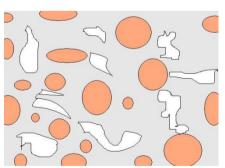


Fig. 11. Model of imagination of microstructure of composite ED-20 + unmodified bentonite. The circles – bentonite, white areas – empties, grey areas – polymer matrix MM

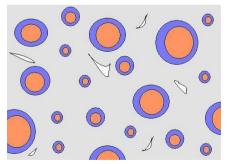


Fig. 12. Model imagination of the composite ED-20 + bentonite +TEOS (till formation of the self-phase of TEOS). Circles – bentonite, rings – TEOS, white areas – empties and grey area – polymer matrix

4. Conclusions

Comparison of the density, ultimate strength, softening temperature and water absorption for polymer composites based on epoxy resin, unmodified and modified by tetraethoxysilane mineral nanofillers, such as bentonite, diatomite and andesite leads to the conclusion that modifying agent stipulates the formation of heterogeneous structures with higher compatibility of ingredients and consequently promotes to enhancing technical characteristics. On the basis of liquid glass, epoxy resin and some high dispersive minerals (andesite, quartz sand) we obtained the composites with high resistance to sulfur acid, which is better in comparison with well known analog - silicate composite. With the use of the mathematical modelling method the optimization of composition of the multi-component systems was conducted, thanks to which the materials with high physico-chemical properties (breaking points at compression, elongation and bending, thermal stability, water and acid resistivity) are obtained.

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ПОЛІМЕР-СИЛІКАТНІ КОМПОЗИТИ З МОДИФІКОВАНИМИ МІНЕРАЛАМИ

Анотація. Одержані нові полімерні композиційні матеріали на основі епоксидної смоли і різних дрібнодисперсних мінеральних порошків (андезит, бентоніт, діатоміт, рідке скло і кварцовий пісок). Досліджені їх механічні (межа міиності), теплові (температурна залежність пом'якшення) властивості та здатність до водопоглинання. Встановлено, що всі властивості цих матеріалів були істотно поліпшені при використані однакових наповнювачів, модифікованих тетраоксисиланом (ТОС). Експериментально показано, що композити, які містять бінарні наповнювачі – діатоміт і андезит при певному співвідношенні – характеризуються так званим синергетичним ефектом – підвищенням максимальних фізичних або хімічних властивостей. Також досліджені коефіцієнти дифузії, сорбції та проникнення реагентів, механічна міцність, коефіцієнт теплопровідності і стійкість до дії сульфатної кислоти. Показано, що експлуатаційні властивості цих матеріалів є істотно кращими у порівнянні з відомими силікатними композитами. Створені матеріали характеризуються низькою усадкою, а також низьким внутрішнім напруженням після затвердіння. За допомогою методу математичного моделювання експерименту проведено оптимізацію складу отриманих матеріалів. Експериментальні результати пояснені з точки зору структурних особливостей полімерних матеріалів.

Ключові слова: епоксидна смола, мінеральний наповнювач, полімерна композиція, механічні та термічні властивості, водопоглинання.