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Influence of Annealing Temperature on Electrical Properties of Unmodified Bentonite

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Abstract: In this work, influence of annealing temperature on the electrical properties of unmodified bentonite has been investigated. It is concluded that, by the increase of annealing temperature, the values of permittivity and conductivity are decreased, but the electric resistance and the density of the bentonite are increased It is also shown that the basic reason for this is the presence of water molecules and alkaline oxides such as Na_2O, K_2O in bentonite.

Keywords: Polymer, Bentonite, Composite, Montmorillonite, Nanocomposite, Conductivity, Resistivity

I. INTRODUCTION

In recent years, extensive research processes of composite materials based on polymers and natural layered silicates have been conducted. This class of new nanocomposites has synergistic properties of the initial components. The singularity of their architectural design is due to the ability of organic phase to capture the nanoparticles into a kind of "traps" or polymer network.

At the same time, many natural silicates (known as smectites), which include, for example, hectorite and montmorillonite (mica-type structure) are comprised of alternating layers of cation and a negatively charged layer silicate. Such layers of "master" together with controlled systems of percolation pores and channels easily form inclusion compounds that is also true in the case of monomer molecules "guests" [1-7]. At the same time, there are almost no materials in the literature devoted to the electrophysical characteristics of bentonite. This work is devoted to the study of the influence of annealing temperature on the electrical properties and IR spectra of unmodified bentonite.

II. EXPERIMENTAL METHODS

The test samples were obtained from the finely divided bentonite in the form discs (washers) with 1.5 mm thickness and 7 mm diameter. The discs were extruded at room temperature without heating under pressure (1 GPa). Then four of the discs were subjected to a thermal annealing at 400°C, 600°C, 800°C and 1000°C. Annealing of the discs was conducted in an air atmosphere furnace such a RH15/15. Disc annealing was performed with the help of a special program. To investigate the electrical characteristics, both sides of the pressed discs were coated by measuring electrodes of silver paste.

For the study Dashsalahlinsky (Azerbaijan) bentonite was used. VAC dielectric properties and electrical resistance were tested on all samples. The measurements of capacitance and dielectric loss factor D (tgδ) were carried out by means of digital gauges immittance E7-20 (at frequencies of $25\text{-}10^6$ Hz). The sample was applied to a measuring voltage of 1V. The measuring instrument selects automatically the characteristics of the reactivity of equivalent circuit of the samples when the accuracy measurement ε and D (tgδ) was 3, and 5%, respectively. The values of the real and imaginary parts of the permittivity (ε' and ε''), as well as the value of conductivity of the samples were determined from the measurement results of capacitance C and the dielectric loss factor using the formulas

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$$c = \frac{\varepsilon\varepsilon_0 S}{d}, \varepsilon^{'} = \frac{\varepsilon}{\sqrt{1+D^2}}, \varepsilon^{"} = \frac{\varepsilon}{\sqrt{1+D^2}} D \qquad \sigma = 2\pi f \ \varepsilon_0 \varepsilon' D$$

Where, D-dielectric loss factor, ε'-real part of the dielectric permittivity, ε "- imaginary part of the dielectric constant, C -capacitance of the capacitor, $\varepsilon_0 = 8.85 \times 10^{-12}$F / m.

The results of calculations are shown in Figs. 1-6. From analysis of the graphics the following conclusions can draw: Independently of the annealing temperature, the change of the dielectric constant (ε) from the frequency has a relaxation character, which is expressed by a monotone decrease of (ε) Fig. 1, with increase of frequency. This character of the variation corresponds to the dipole and the migration polarizations.

The maximum value of dielectric permittivity is observed on no annealed sample (50000) and the lowest value for the annealed sample at 1000°C (39pF), For no annealed sample, by increasing frequency D sharply decreases, reaches the minimum value at f=100kHz then with increase of frequency monotonically increases. For the sample annealed at 500°C with the increase of frequency D increases, reaching the maximum value at f=100 kHz, and then it monotonically decreases (Fig. 2).

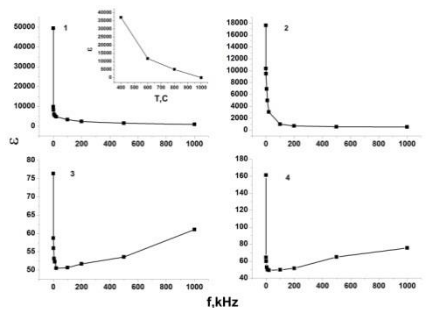

Fig. 1. Dependence of the permittivity on the frequency of the alternating field: 1-unmodified, 2- T=500°C, 3-T=800°C, 4-T=1000°C. 5-ε=f(T).

For the samples annealed at 800°C and 1000°C dependences D = f (F) have the same, namely, particulate character Recession D = f (F) depends on the frequency which is explained by delay of dipoles and reduce of the number of particles participating in polarization.

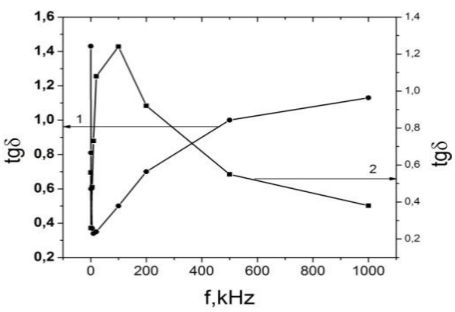

Fig. 2. Dependence of dielectric loss for unmodified bentonite on frequency: 1-T = 25°C, T = 500°C.

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From Fig. 3 it is seen that the dependence of the value of current (VAC) on the applied voltage observed on the investigated samples has complex character.

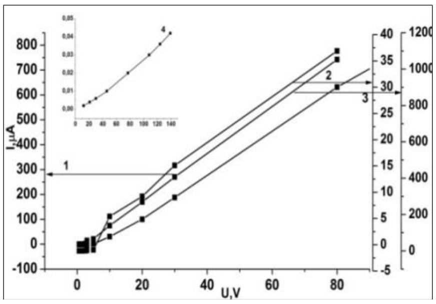

Fig. 3. Dependence of electric current on voltage.

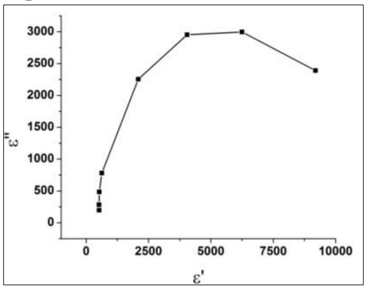

Fig. 4. The Cole-Cole diagram for unmodified bentonite at an annealing temperature at T = 500°C.

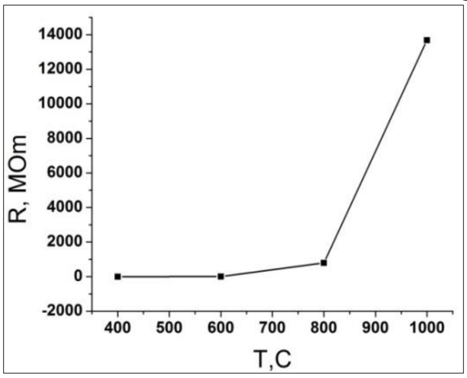

Fig. 5. Dependence electrical resistance of the annealing temperature.

Thus, for no annealed sample VAC is non-linear, in other words with increase of applied voltage the value of current increases and changes in the order of 2-5, for annealed samples VAC has linear character. It should be noted that the observed dependence ε '' = f (ε ') (semicircle) Fig. 4. at annealing temperature at T = 500°C that is typical for dielectric of low conductivity, is not the same for ion-exchange materials [7]. The value of electrical resistance increases rapidly with increase of annealing temperature (Fig. 5).

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III. DISCUSSION OF THE EXPERIMENTAL RESULTS

Note that, the production of polymer compositions with special physical properties are largely depend on the nature of the filler, the shape, size and character of the particle distribution as well as the degree of interaction between the components.
The nature of the aggregation of filler particles, conditions of crystallization and other factors change the morphology of the polymer matrix, and as the result the composites obtained on their basis acquire unique properties, which leads to an increase in their practical application possibilities. This is due to the fact that, unlike traditional non-linear devices, they contain symmetrical current-voltage (I-V).

The development of physical chemistry and technology of composite materials, which are in most cases filled with linear and cross-linked polymers, requires clarification of our ideas about the nature of their nearby orderliness and their above molecular order.

Besides that, there is not sufficient knowledge of contact phenomenon, the change the electro physical and electro active properties of the heterogeneous systems polymer-filler, especially interfacial phenomena. Before discussing the experimental results, we should note the following features of the crystal structure of clay minerals. According to these papers [4-6], the clay mineral includes two layers of silicon-oxygen tetrahedral, facing each other by their peaks, that on both sides covered by the layer of Aluminohydroxyl octahedra, which in general appears to be a three-layer packet (2: 1).

The thickness of the elementary packet is 0.96 nm. If you look at the structure of montmorillonite, it can be seen that the nanoscale related only to the interplanar distances, whereas the length and width of these layers can be up to a few microns. A characteristic feature of the montmorillonite structure is that the molecules of water and other polar molecules, for example some organic molecules, can penetrate between the structural layers, causing the expansion of the lattice.

Montmorillonite clays possess a certain capacity for cation exchange. This indicator determines the amount of exchangeable cations in the layered silicate (mg-equivalent), capable of being replaced by cations of another type in terms of 100 grams of clay.

Montmorillonite has the largest cation exchange capacity (up to 90-120 mg.eq/100 g of dry clay) among layered silicates. From the above statements the experimental results can be explained by the following way. The large value of dielectric constant (or electrical conductivity) and the dielectric loss in the initial bentonite is due to the fact that under the influence of thermal motion the weakly bound ions of alkali metal oxides (Na_2O, K_2O)can come off from the floor fixing points and move from one cell spatial grid to another.

At the same time, ion-relaxation polarization is enhanced, as a result of this, the number of ions involved in the electrical conductivity increases. Moreover, when smaller the ion radius is the greater mobility, and the greater their contribution to the conductivity. Since the sodium ion has a smaller radius than the potassium ion, respectively, it will have more influence on the electrical conductivity of bentonite.

According to the work [6,8] the presence of water molecules in the bentonite also greatly affects the electrical properties of the bentonite. In the works [6,8] it has been shown experimentally that with increasing annealing temperature, the number of water molecules is greatly decreased and as a result of which the dimensions of the mineral lattice is reduced to 98nm.

Note also that, the annealing temperature also affects the density of the samples, namely, by increasing annealing temperature, the density of the investigated samples is increased (Fig. 6).

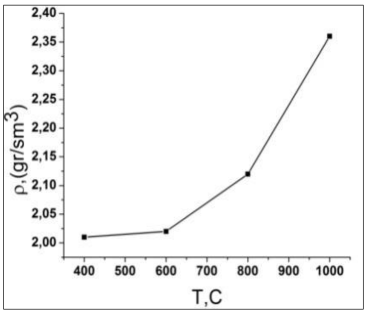
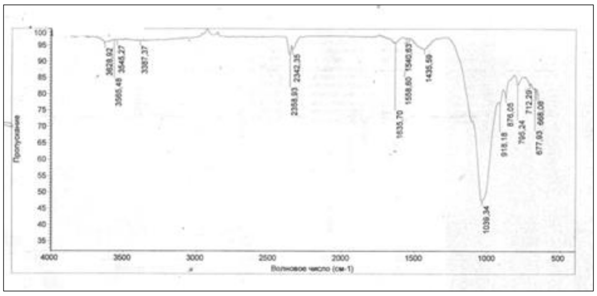
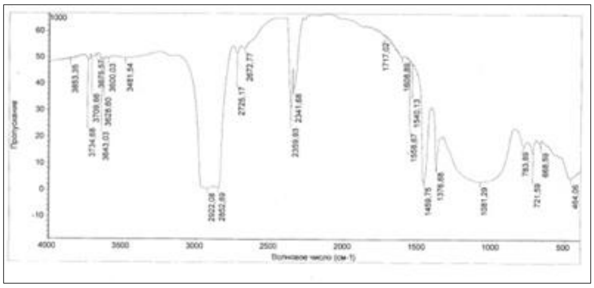


Fig. 6. The dependence of the density of bentonite on the annealing temperature.

In this way, by the thermal annealing the material with a high resistance (10^{10} ohms) is obtained, that can be successfully used in various fields of the electronic industry.



a)



b)

Fig. 7. Infrared spectra: a) unburned bentonite, b) nnealed bentonite.

From the analysis of the IR spectrum of Fig. 7, the presence of various structural groups in bentonite, namely, stretching vibrations of OH groups in the 3400-3600 range, stretching vibrations of Si-O-Si and Si-O-Al at 1037, and deformation vibrations of H_2O at 1635, And Al-OH-Mg at 843, etc. We note that the IR spectrum data are well combined with the data of [9,10]. From a comparison of the IR spectra (Fig. 7) it is clear that at the annealing temperature T = 1000°C there are no reflexes associated with water molecules and OH groups. Taking into account the fact that water has a high conductivity, its absence at the annealing temperature T = 1000°C leads to a decrease in the conductivity of bentonite. This, in turn, according to the formula leads to a decrease in the dielectric constant, dielectric losses and to the growth of electrical resistance.

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It should be noted that for materials used in high voltage installations, especially in high-voltage and high-frequency devices, it is important that these materials have small values of dielectric loss and dielectric constant. The fact is that the presence of large dielectric losses in an electrically insulating material causes strong heating as a result of which the material can be subjected to thermal destruction. In addition, if the dielectric is used in the oscillatory circuit, then dielectric losses prevent the achievement of high Q, since with increasing equivalent resistance of the loss loop, damping of the oscillations in the circuit is amplified.

Taking into account the above, the experimental results obtained make it possible to assert that annealed bentonite can be used with great success for practical application, namely, for manufacturing high-voltage and high-frequency devices. Actually, as seen from Fig. 7, in the samples studied, the dielectric constant and dielectric losses decrease with increasing annealing temperature. At the same time, the permittivity value is 1300, and the dielectric loss is 3 times, respectively. In addition, the value of the electrical resistance increases by 4 order, the quality factor increases by100 times.

IV. CONCLUSION

The influence of the annealing temperature on the electrophysical parameters and IR spectra of unmodified bentonite was determined experimentally. It is established that as the annealing temperature increases, the dielectric constant, electrical conductivity and dielectric loss coefficient decrease, and the electrical resistance increases. The reason for the established facts, along with other factors, is a reduction in the percentage of polar compounds (H_2O, OH groups, etc.) participating in the conductivity with increasing annealing temperature.

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