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SYNTHESIS OF ORGANOMODIFIER BASED ON MONTMORILLONITE

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ABSTRACT
The process of modification of Na-form montmorillonite with a monomeric quaternary salt based on isohexyl monochloroacetate with triethylamine has been studied. The structure of the obtained organomodifier was determined using IR spectral and electron microscopic analyzes. The IR spectra of the modified samples of montmorillonite with a quaternary salt show that the molecules of the quaternary salts interact with the exchangeable cations of the clay. Growth electron microscopy showed that volumetric aggregates are formed on the surface of the modified clay.

KEYWORDS: synthesis, montmorillonite, quaternary ammonium salt, isohexyl monochloroacetate, triethylamine.

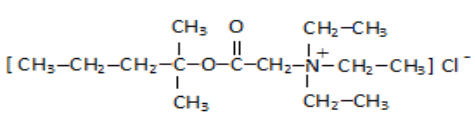
INTRODUCTION

The filler for various (co) polymers is clay minerals capable of swelling - smectites. Many researchers use montmorillonite layered silicates for this purpose [1,2]. The use of montmorillonite clays (MMT) as polymer modifiers plays an important role in improving the complex of physical and mechanical properties of the material. To make the polar layered silicate material compatible with non-polar or weakly polar polymers, the surface of montmorillonite clays is organomodified. A study of the literature shows that the main type of organomodifiers for MMT are quaternary ammonium salts (QAS), the molecules of which include alkyl hydrocarbon chains of various lengths and functional groups. Polyolefins are the largest scale industrial polymers with easy processability, chemical resistance, low cost and a wide range of applications. The development on their basis of new nanocomposite materials with the indicated complexes of properties is of scientific and practical interest. However, for nanocomposites based on polyolefins, it has not yet been possible to achieve the same significant effects of changing the entire complex of mechanical and functional properties as in the case of polar polymer matrices, which is associated with the limited compatibility of non-polar polymers with aluminosilicates and the difficulty of realizing a high degree of separation of particles of a layered filler into single nanolayers in a polyolefin matrix, which primarily determines the efficiency of improving the complex of properties of polymer nanocompositions. Therefore, at present, the efforts of researchers are aimed at a systematic search for new organomodifiers.
The aim of this work was to obtain organomodified clays and polymer materials based on them.

EXPERIMENTAL PART

In the work, we used layered silicate based on the clay mineral montmorillonite (MMT) of high purity of the Navbakhor deposit.
A quaternary ammonium salt (the product of the reaction of isohexyl monochloroacetate with triethylamine) was used as a clay modifier. Its structure and properties are shown in the table.

Table. Structure and characteristics of QAS

Modifier structure	MM	Tmelt., °C
	279,5	140-142

Synthesis of organomodified clays**.** A suspension of the composition was prepared by dispersing 2.5 g of carbonate-palygorskite clay in 250 ml of distilled water with vigorous stirring for 24 h at room temperature. The modification of the clay mineral with a quaternary salt (QAS) was carried out according to the method proposed in [3].

A solution of the quaternary salt in water was prepared at a temperature of 50-60 ° C with constant stirring. Then the hot modifier solution was added in predetermined portions to the montmorillonite suspension with stirring. The time to reach equilibrium in the systems was two days. The modified clay was filtered on a Buchner funnel, washed with distilled water, and dried at room temperature.

IR SPECTROSCOPY

IR spectra were recorded on an FSM 1201 IR Fourier spectrometer with a computer control system, manufactured by AO SPb Instruments (St. Petersburg). The frequency range of research is 400-5000 cm-1. Solid samples for scanning their absorption spectra are prepared in two ways: in the form of a suspension in vaseline oil, or in the form of tablets in potassium bromide.

RESULTS AND THEIR DISCUSSION

The optimal conditions for the reaction of synthesis of nanocomposites were determined. To prepare composites, a sufficiently concentrated bentonite suspension was used. In this concentration range, an important role is played by the forces of attraction between the edges and planes of crystallites. With mechanical stirring, the edge-plane contact is destroyed.

The main factors affecting the process of obtaining a composite based on montmorillonite and QAS are the monomer concentration and temperature. The results of a study of the duration of the QAS reaction to montmorillonite show that the efficiency of the modification process reaches its maximum values in 240 minutes.

To clarify the structure of the modified clay, the method of IR spectroscopy was used. Were taken IR spectra of unmodified and modified montmorillonite (Fig. 1.2).

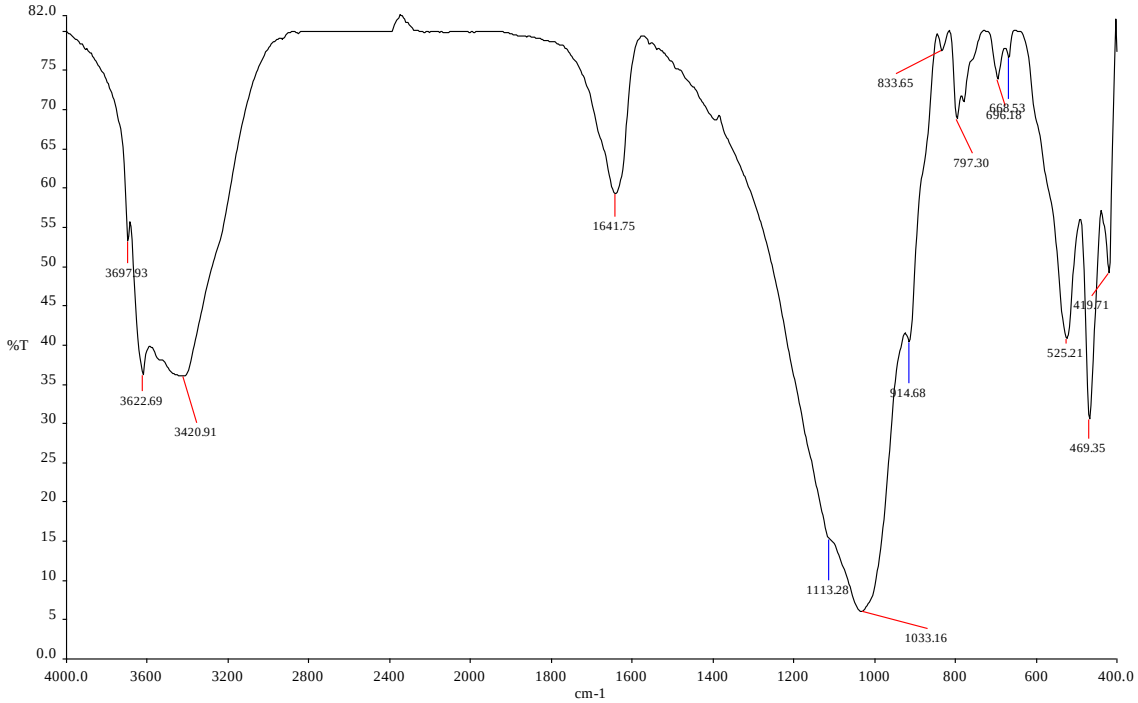


Figure: 1. IR spectrum of the Na-form of palygorskite montmorillonite (attapulgite).

As can be seen from Fig. 2, changes are observed in the IR spectra of organomodified MMT with the indicated quaternary ammonium salt (OMG). A characteristic feature of the interaction of the quaternary salt with the basal surfaces of montmorillonite can be considered the appearance in the IR spectra of maxima at 1577 cm – 1, corresponding to the coordination-bound carboxyl. Also in the regions of 2700-2900 cm-1 additional absorption bands are observed.

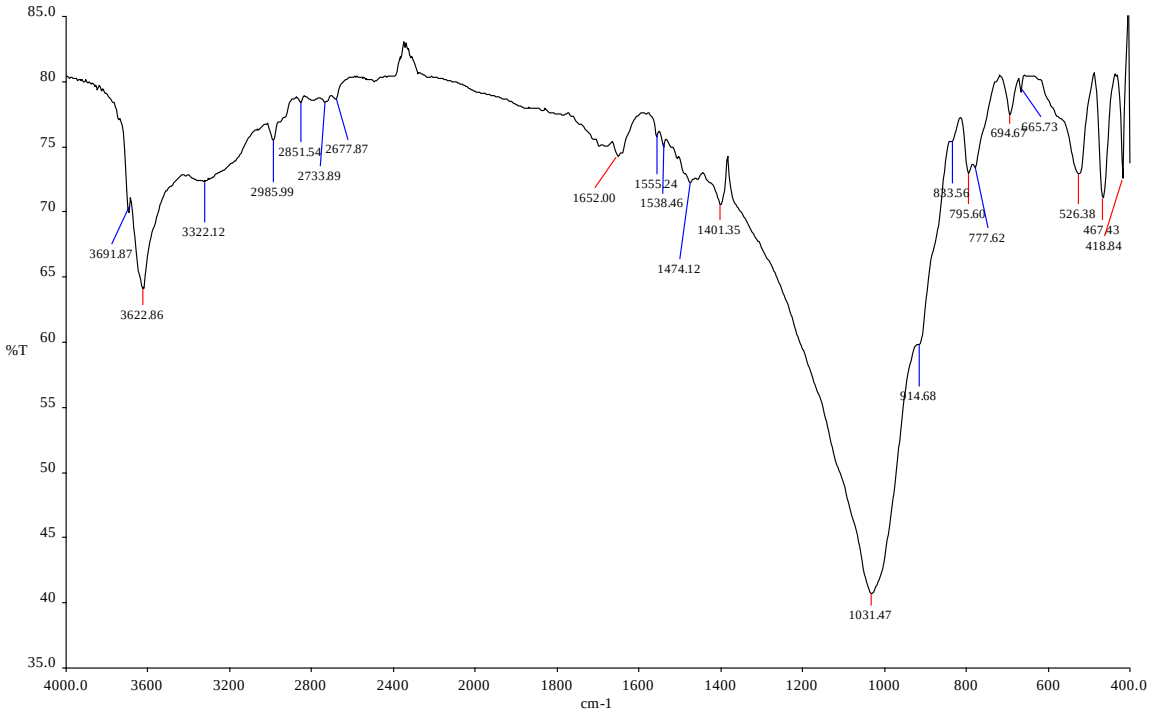


Figure: 2. IR spectrum of montmorillonite modified by QAS.

Investigation of the surface of MMT and its modified forms by rostrum electron microscopy showed a change in morphology after organomodification. Figure 3 shows the ordered formation of organomodifier domains on the surface and in montmorillonite layers. These micrographs from Figure 3 show that bulk aggregates are formed on the surface of the modified MM clay. This indicates the intercalation of QAS molecules into the interlayer spaces of MMT and the formation of nanocenters. During organomodification of MMT, quaternary ammonium salts interact with a negatively charged surface of aluminosilicate, sodium ions from montmorillonite are replaced by ammonium ions and form a loose hybrid structure, increasing the distance between silicate plates.

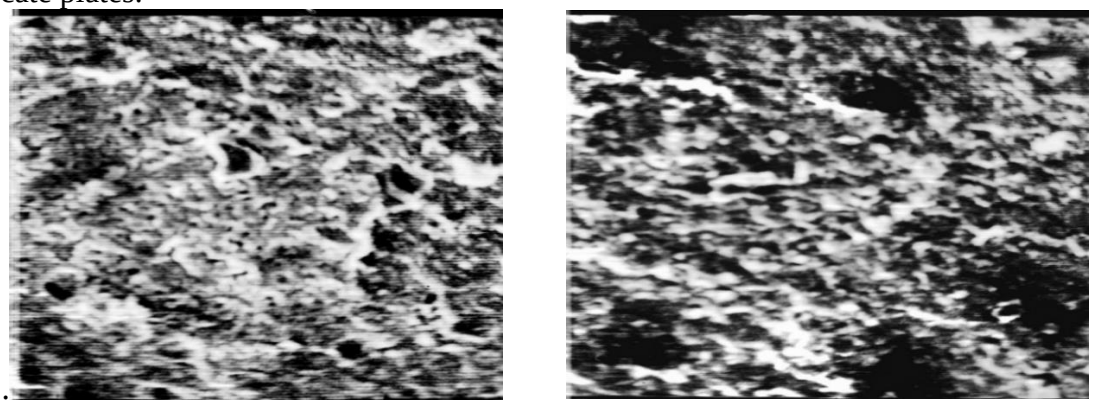


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Fig. 3. Micrographs of unmodified Na-montmorillonite (a) (x-250) and modified QAC (b) (x-250).

CONCLUSIONS

Organomodified MMT based on the monomeric quaternary salt of isohexyl monochloroacetate with triethylamine was obtained. The IR spectra of the modified samples of montmorillonite with a quaternary salt show that the molecules of the quaternary salts interact with the exchangeable cations of the clay. Growth electron microscopy showed that bulk aggregates are formed on the surface of the modified MM clay.

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