



Research article

UDC 625.068.2

DOI: 10.34910/MCE.128.9



## Structure of clay minerals of road soil-cement during complex modification

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**Keywords:** kaolinite, montmorillonite, dodecyltriethoxysilane, complex modification, polycarboxylate ester, intercalation, splitting, chemisorption, soil-cement

**Abstract.** Previously obtained positive results of studies of physical, mechanical, technological and operational properties of road soil-cement at their complex multifunctional modification with organosilicon compounds and polycarboxylate superplasticizers have shown the effectiveness of the use of *strengthened* soils in the construction of roads. However, the influence of complex modification on changes in the structure of clay minerals of strengthened soils in road construction remains insufficiently studied. Using X-ray phase analysis, comprehensive studies of the structure of modified clay minerals were carried out to reveal intercalation processes and changes in the size of their particles as a result of splitting processes. Chemical structure of the studied modifiers, nature of their interaction with clay minerals and organomineral bonds were confirmed using IR spectroscopy. The combined influence of modifying effect of dodecyltriethoxysilane and polycarboxylate ester on kaolinite and montmorillonite clays was established, which led to the manifestation of synergistic mechanism at strengthening of clay soils. During the modification, a multifunctional effect was noted, characterized by an increase of integral intensity of diffraction maxima on the diffraction patterns of modified clay soils, associated with an increase of interplanar distances in their structure as a result of intercalation processes. The positive effect of complex modification of clays is associated with splitting and reduction of mineral particle sizes. It has been established that the consequence of complex modification is chemical interaction (chemisorption) with the formation of organosilane bonds, which provide increased efficiency in strengthening of clay soils in road construction.

**Citation:** Vdovin, E.A., Bulanov, P.E., Stroganov, V.F., Morozov, V.P. Structure of clay minerals of road soil-cement during complex modification. Magazine of Civil Engineering. 2024. 17(5). Article no. 12809. DOI: 10.34910/MCE.128.9

### 1. Introduction

It is known that soil-cements are the building material type obtained by mixing and subsequent compaction of soils, Portland cement and water (modifiers as necessary) [1, 2]. Their demand is ensured by a number of advantages: cost-effectiveness, reduction of negative load on the environment and workability during construction [3].

In road bases and surfaces construction various types of soils and local materials are used, especially in areas with a lack of durable stone materials [4, 5]. However, these materials (especially clay soils strengthened by Portland cement) in humid conditions [6] are susceptible to the climatic factors effects, which contributes to development of damage processes the road pavements structural layers and the roads service life reduction [7].

Clay soils have a wide range of mineralogical composition and consist mainly of small particles – hydrous aluminosilicates and other minerals. Clays are mainly inorganic materials [8, 9]. Clay minerals are a diverse group of hydrous aluminosilicates that usually display layered or fibrous structures encompassing polymeric sheets of silica tetrahedra linked with octahedral sheets [10, 11]. Clay minerals with silica tetrahedral and alumina octahedral sheets are called 1:1 clays. Similarly, clay minerals with one octahedral aluminate sheet sandwiched by two silica tetrahedral are called 2:1 layer clays [12].

The most common clay minerals in soils are kaolinite and montmorillonite. Kaolinite is a relatively stable mineral found in fairly large quantities in many clay soils ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ). The second, most common clay mineral, montmorillonite ( $2\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 4\text{H}_2\text{O}$ ), is formed under alkaline conditions in continental and marine sediments and in the weathering crust. It belongs to the layered silicates subclass and has the ability to swell strongly [13]. According to their mineral composition, clay soils are usually divided into monomineral (kaolinite, montmorillonite, illite, etc.) and polymineral, consisting of various clay and relict minerals [14, 15].

It is known that one of the methods for improving the soil-cement quality and durability is hydrophobization, which gives the mineral particles and their aggregates surfaces the ability not to be wetted by water [16, 17]. The most effective modifiers for reducing water absorption and increasing frost resistance of strengthened soils are organosilicon compounds (silanes, siloxanes, etc.) [18, 19].

Molecules of organosilicon compounds consist of two parts, opposite in nature and properties. At one end of the molecules there are hydrophilic (polar) groups (OH, CHO, COOH,  $\text{NH}_2$ , etc.), which are a source of strong molecular interaction and therefore are highly soluble in water. At the other end of the molecules, there are hydrophobic parts formed by one or several rather long chains saturated with hydrocarbon radicals ( $\text{C}_n\text{H}_{2n+2}$ ), incapable of hydration and insoluble in water [20–22].

Hydrocarbon chains of hydrophobic molecules tend to repel each other, causing them to form a brush-like structure on the absorbent surfaces (Langmuir palisade). Such structures presence promotes the adjacent particles separation and reduces friction between them, which contributes to the soils mixing and compaction processes when they are treated by surfactants. It has been established that the hydrophobic films effect can manifest itself in different ways: they prevent the water molecules penetration to the mineral particles contact points [23]. In addition, the enveloping process the soil capillaries walls contributes to the reverse curvature menisci formation in them, which prevent the movement of capillary and gravitational moisture. The process of mineral particles enveloping with hydrophobic films protects them from swelling, dissolution and destruction [24].

The greatest effect of hydrophobization processes is shown, as noted above, by organosilicon compounds, in the molecules of which silicon atoms are connected to alkyl (methyl, ethyl) and aryl radicals (phenyl) [25, 26].

Another effective direction of increasing operational and technological (workability) characteristics is the soil-cement plasticization [28, 29]. The most promising modifiers among plasticizing substances are polycarboxylate esters, which, unlike known superplasticizers, according to [30], contribute to a positive change in the hydrate new formations morphology and the ettringite crystals size decrease. The possibility of the stable organomineral phases formation due to the polymer modifier incorporation into the calcium hydroaluminate lamellar structure is also noted [31].

The previously obtained positive results of modified soil-cement by polycarboxylate plasticizers [28] are explained by the influence processes on both the soil clay minerals and the cement system. Theoretically, the existence of two main interactions between clay minerals and polycarboxylates is assumed [29, 30]:

- electrostatic interaction between positively charged clay surface areas and negatively charged plasticizer groups;
- plasticizer side chains intercalation between clay minerals aluminosilicate layers as a result of deprotonation processes.

The action mechanism when modified by substances described above was previously considered mainly on cement systems. It is known that plasticizers and water repellents are used mainly in the modification of cement concrete mixtures, in which inert material is used as a filler. However, in strengthened clay soils there is a diverse minerals amount, including clay minerals, which have high cation exchange and specific surface area [12, 32]. These minerals presence in strengthened soils causes their strength and frost resistance decrease [33]. Previously carried out studies on the strengthened clay soils modification made it possible to identify the most effective modifiers for increasing the soil-cement strength and frost resistance: polycarboxylate superplasticizers [27, 33, 34] and water repellents – dodecyltriethoxysilane and others [35, 36]. However, in the works carried out there are no studies of these modifiers influence on the clay minerals structure.

In connection with the above, the purpose of the work is to study the joint modification mechanisms of soil-cement with a polycarboxylate superplasticizer and an organosilicon water repellent (dodecyltriethoxysilane), the structural characteristics change, and, as a consequence, the level of clay minerals properties in road construction:

- to study the modifiers intercalation processes in clay minerals using X-ray phase analysis;
- to determine the chemisorption possibility of the studied modifiers with clay minerals using IR spectroscopy;
- to determine the magnitude of the region of clay minerals particles coherent scattering after modification by X-ray phase analysis;
- to identify the influence mechanisms of complex modification on structural changes that ensure an increase the indicators level of soil-cement compositions properties in road construction.

## 2. Materials and Methods

For the research, kaolinite clay produced by NPP Industrial Minerals LLC (Samara Region, Russia) with kaolinite mineral content of up to 95 % and montmorillonite clay from the Biklyansky quarry of the Tukaevsky district of the Republic of Tatarstan with a montmorillonite mineral content of up to 70 % were used.

The following modifiers were used:

- dodecyltriethoxysilane  $C_{18}H_{40}O_3Si(OC_2H_5)_3$ . Hubei Co-Formula Material Tech Co., Ltd. (China);
- Ready-Mix 304 – plasticizing additive based on a composition of polycarboxylate ethers. JSC TechnoNIKOL (Russia).

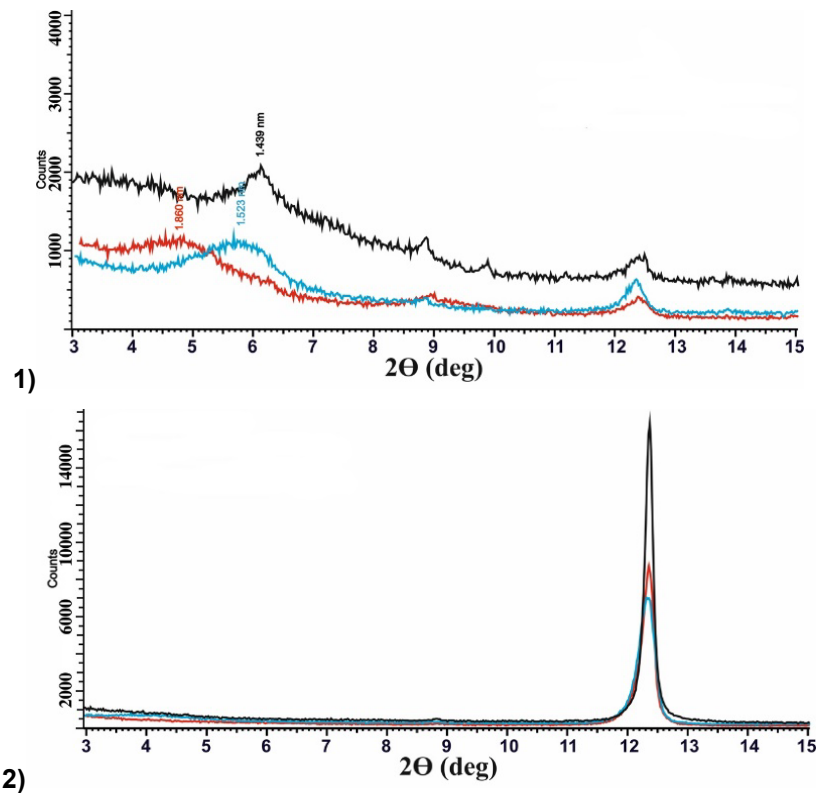
X-ray diffraction analysis was used to determine the interplanar spacing of clay minerals during modification. Experiments were carried out on a Bruker D2 Phaser automatic X-ray diffractometer with a linear coordinate detector.  $CuK\alpha$  radiation, monochromatic ( $\lambda(Cu-K) = 1.54184 \text{ \AA}$ ), X-ray tube operating mode 40 kV, 40 mA were used. Experiments were performed at room temperature in Bragg-Brentano geometry with a flat sample. Construction and analysis of diffraction patterns to determine the mineral composition were performed using the DIFFRACplusEvaluationPackage – EVA, Search/Match, Bruker Diffrac Eva program. The degree of crystallinity was assessed (determining the size of the coherent scattering region) based on the width of the diagnostic diffraction maxima using the Scherer formula.

IR spectroscopy was used to determine the chemical structure of modifiers and their nature of interaction with clay minerals, as well as to determine the covalent bonds between clay minerals and additives. Data were collected using a Bruker Vertex 70 FTIR spectrometer with a single reflection, germanium crystal ATR accessory (MIRacle, PIKE Technologies) purged under dry air to remove atmospheric water vapor. Background spectra of 64 scans at a resolution of  $2 \text{ cm}^{-1}$  were subtracted from sample spectra.

## 3. Results and Discussion

In accordance with the set goals and objectives, a set of studies was carried out to establish the mechanisms and features of the modification processes influence on the interaction between polycarboxylate ester and dodecyltriethoxysilane and soils clayey components.

In the presented diffraction patterns (Fig. 1), there is an integral intensity increase of the diffraction maxima by 29 % (for montmorillonite clay modified by polycarboxylate ether) and 5.8 % (for montmorillonite clay modified by dodecyltriethoxysilane). This is explained by an increase of clay mineral interplanar distance as a result of the polymer radicals intercalation with montmorillonite [30]. A multifunctional effect was noted, characterized by an increase of diffraction maxima integral intensity on modified clay soils diffraction patterns, associated with an interplanar distances increase in their structure as a result of intercalation processes. When kaolinite clay was modified by polycarboxylate ether and dodecyltriethoxysilane, no changes was observed in the interplanar distance.

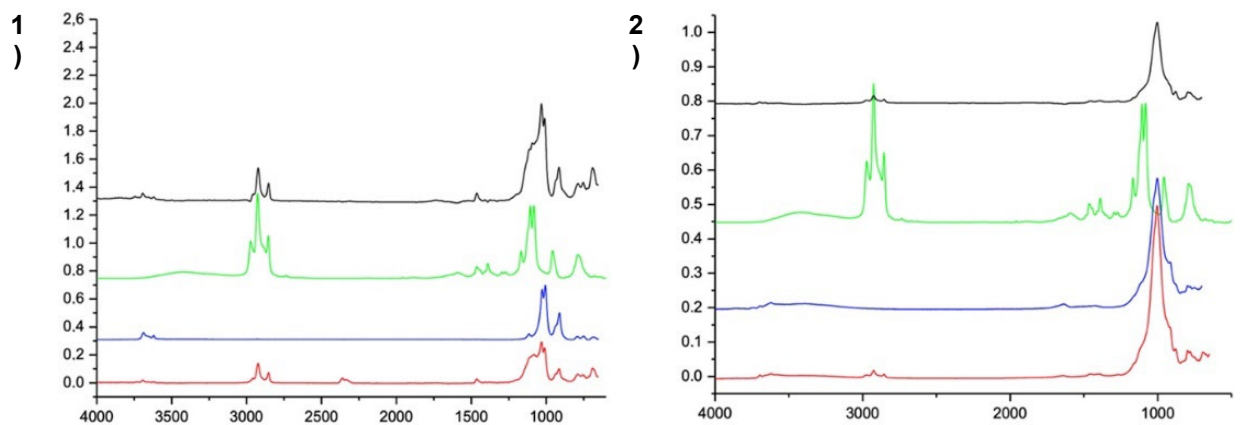


**Figure 1. X-ray diffraction patterns of studied clays: — — unmodified clay; — — clay modified with polycarboxylate ether; — — clay modified with dodecyltriethoxysilane: 1 – montmorillonite clay; 2 – kaolinite clay.**

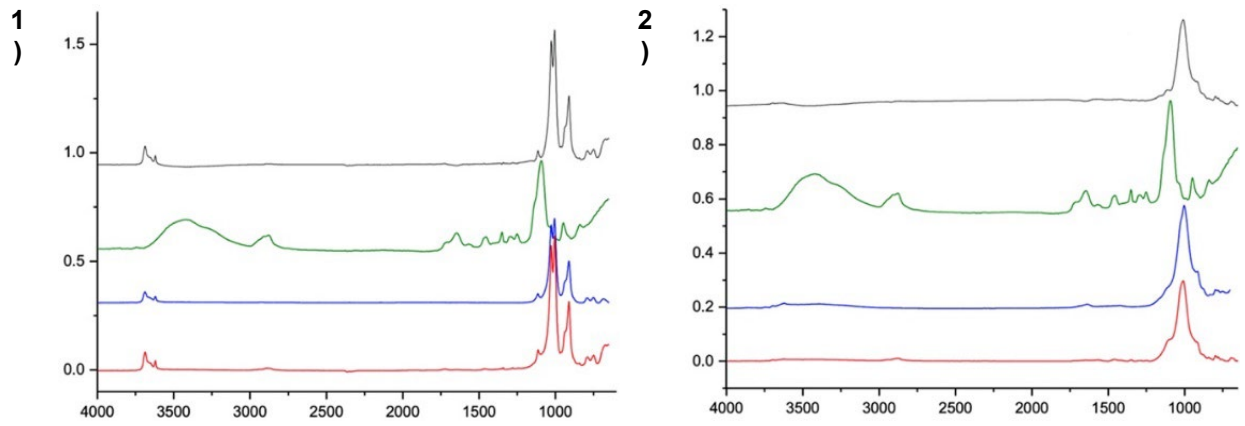
**Table 1. Results of determining the region of clay mineral particles coherent scattering.**

Name of materials studied	LVol-IB, nm
Kaolinite clay	37.72
Kaolinite clay modified by polycarboxylate ester	25.85
Kaolinite clay modified by dodecyltriethoxysilane	22.62
Montmorillonite clay	18.89
Montmorillonite clay modified by polycarboxylate ester	9.65
Montmorillonite clay modified by dodecyltriethoxysilane	8.47

Using the X-ray phase analysis method, the values of coherent scattering region (CSR) of clay minerals particles were calculated (Table 1). The studies performed suggest that modifiers cause the minerals shape and size changes (as a result of their splitting). The positive effect of complex clays modification is associated with a decrease in size of kaolinite clay mineral particles by 31.5 % and 40.0 %, and in montmorillonite clay by 48.9 % and 55.2 %, respectively.



**Figure 2. IR spectra: — — difference spectrum; — — dodecyltriethoxysilane; — — clay; — — clay modified by dodecyltriethoxysilane: 1 – kaolinite; 2 – montmorillonite.**



**Figure 3. IR spectra: — — difference spectrum; — — polycarboxylate ester; — — clay; — — clay modified by polycarboxylate ester: 1 – kaolinite; 2 – montmorillonite.**

It has been established that the complex modification consequence is chemical interaction (chemisorption) with the organosilane bonds formation, which ensure increased efficiency of the clay soils strengthening technology in road construction.

Based on the results of the IR spectroscopic analysis method (Fig. 2 and 3), it was determined that the band at  $3700\text{ cm}^{-1}$  in the studied clays indicates the intrasurface OH groups presence located on the octahedral layers opposite tetrahedral oxygens surfaces, and the band at  $3621\text{ cm}^{-1}$  may be attributed to internal OH groups located in a plane common to octahedral and tetrahedral layers [38]. Analysis of the given IR spectra shows that the main bands in them relate to the valence bonds of silicon with oxygen and hydrogen with oxygen. Oxygen atoms can be connected to two silicon atoms via Si-O-Si bridging bonds, or to one via non-bridging Si-O bonds. In particular, the bands at  $1032\text{ cm}^{-1}$  and  $1009\text{ cm}^{-1}$  are attributed to the stretching vibrations of Si-O-Si(Al) bridging bonds in the kaolinite clay crystal lattice and  $1090\text{ cm}^{-1}$  and  $990\text{ cm}^{-1}$  in montmorillonite clay [38, 39].

The chemical structure of modifiers dodecyltriethoxysilane and polycarboxylate superplasticizer was confirmed. In the dodecyltriethoxysilane spectrum stretching vibrations of bonds correspond to an intense peak at a frequency of  $1093\text{ cm}^{-1}$  with a shoulder of  $1120\text{ cm}^{-1}$  and peaks of ethyl groups stretching and bending vibrations at frequencies of  $2990, 2910, 2840, 1450\text{ cm}^{-1}$ , corresponding to the aliphatic  $\text{CH}_2$  and  $\text{CH}_3$  groups of the monomer radical.

In the polycarboxylate superplasticizer spectrum the mainly contains the stretching vibrations in the region of  $2840\text{--}2970\text{ cm}^{-1}$  and bending vibrations in the region of  $1450\text{--}1470\text{ cm}^{-1}$  associated with aliphatic  $\text{CH}_2$  and  $\text{CH}_3$  groups of polyethylene glycol. The C-O-C ether bond stretching vibrations corresponds to an intense peak at  $1125\text{ cm}^{-1}$ .

It has been established that the complex modification consequence is chemical interaction (chemisorption) with the organosilane bonds formation, which provide increased strengthening clay soils efficiency in road construction. When montmorillonite clay is modified by dodecyltriethoxysilane, the intense peak at a frequency of  $1093\text{ cm}^{-1}$  with a shoulder of  $1120\text{ cm}^{-1}$  disappeared, but the bands corresponding to the monomer radical remained. Analysis of the difference spectrum and spectrum of kaolinite clay modified by dodecyltriethoxysilane showed that no chemical interaction occurs.

When kaolinite and montmorillonite clays modified by polycarboxylate ether, the ether C-O-C bond complete disappearance was noted, which indicated the polymer chemical interaction (chemisorption) with clay minerals.

## 4. Conclusions

1. The joint influence of the dodecyltriethoxysilane and polycarboxylate ether modifying effect on kaolinite and montmorillonite clays has been established, which led to the manifestation about synergistic mechanism in strengthening clay soils. During the modification, the polyfunctional effect was noted, manifested in the processes of intercalation, chemisorption, splitting and reducing the clay minerals particle size. The completed research results will ensure the effectiveness of using soils with a clay minerals high content in road pavement structures.
2. A multifunctional effect was noted, characterized by an increase of diffraction maxima integral intensity on the modified clay soils diffraction patterns, associated with an interplanar distances increase in their structure as a result of intercalation processes. When kaolinite clay is modified

with polycarboxylate ether and dodecyltriethoxysilane, no changes are observed in the interplanar distance.

3. The positive effect of complex clays modification has been established, which consisted in reducing the kaolinite clay mineral particles size by 31.5 % and 40.0 %, and in montmorillonite clay by 48.9 % and 55.2 %, respectively.
4. It has been established that the complex modification consequence is chemical interaction (chemisorption) with the organosilane bonds formation, which ensure an increase the strengthening clay soils efficiency in road construction.

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Received: 23.03.2024. Approved after reviewing: 18.06.2024. Accepted: 22.06.2024.