

Review article

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## Structure formation in the «clay soil – carbide sludge» dispersed system

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### ABSTRACT

**Introduction.** Clay soil is a multiphase, multicomponent aluminosilicate dispersed system with specific properties determined not only by its composition but also by the formation of coagulative and transition contacts (binds) between the soil particles. One of the methods of changing soil properties is the introduction of active mineral additives that promote the formation of phase contacts (binds) between soil particles as a result of the pozzolanic reaction. The effectiveness of using carbide sludge, which is a multi-tonnage lime-containing waste (the content of active calcium oxide reaches 56%) as an additive, has been proved. However, to date, the proposed mechanism of interaction in the «clay soil – carbide sludge» system is based only on the literature data and has not been experimentally verified. **The purpose of this research** is to study the mechanism of structure formation in the «clay soil – carbide sludge» dispersed system. **Methods and materials.** A soil model has been created by mixing saponite-containing material with sand, which corresponds to the composition and properties of sandy loam. The carbide sludge in the form of a suspension was selected from the sludge collector, dried to a constant mass and sieved. Microstructural analysis, differential thermal analysis (DTA), and X-ray phase analysis were used to study the mechanism of structure formation. **Results and discussions.** Based the results of the differential thermal analysis, there is a decrease in the intensity of the endothermic effect in the range of 460 to 470°C associated with the decomposition of calcium hydroxide in the treated sample. Additionally, an endothermic effect is observed at 750°C, which indicating the decomposition of calcium silicate hydrate. The results of differential thermal analysis are confirmed by X-ray phase analysis, which shows the presence of tobermorite group hydrosilicates in the reaction medium. The study of the microstructure of the analyzed mixtures revealed a decrease in the specific volume of pores with a diameter of 4–5 nm in the modified clay soil. This is associated with gelling from particles of new hydrate formations. Besides that, the volume of pores with a diameter of more than 6 nm increased, which indicates the process of contraction. **Conclusion.** The mechanism of structure formation in the «clay soil – carbide sludge» system has been established.

**KEYWORDS:** dispersed system; clay soil; active mineral additive; carbide sludge; structure formation; phase contacts.

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### INTRODUCTION

An analysis of the condition of regional and local roads in the Northern Part of the European Russia, in particular, in the Arkhangelsk region, showed that more than 50% of these roads do not meet regulatory requirements [1]. The lack of well-developed, stable and safe transport system hinders the socio-economic, historical and

cultural development of the region in question, which is included in the list of priority geostrategic territories of the Russian Federation. One of the possible causes of road damage is the weak roadbase, which is composed of clay-based soils that are prevalent in the northern and Arctic territories. These soils include sandy loams, clay loams, and clays, with inclusions of gravel, pebbles, and boulders [2].

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Clay soil is a classic subject for colloidal chemistry both theoretically and practically, which is explained by the clarity and convenience of studying the nature of the interaction of nano- and microparticles using clay dispersions as an example, as well as its wide application in various sectors of human economic activities [3–13]. The soil is a multiphase multicomponent aluminosilicate polydisperse system consisting of solid, liquid and gaseous components that can act both as a dispersed phase and a dispersion medium. In addition, the soil is a capillary-porous body in which the solid frame and pore system are continuous, which meets the main characteristic of the dispersion medium. The solid component of clay soil is composed of various minerals, among these, the presence of clay minerals (up to 20%) gives specific physico-chemical properties characteristic to the soil as a colloidal system: weak water resistance (soaking, high hydrophilicity, ability to swell and shrink), heaving properties, stickiness, plasticity, cation-exchange capacity, thixotropy and increased adsorption capacity [14–18]. This is due to the particle size distribution and structural characteristics of clay rocks. They have a small particle size, typically ranging from 1 to 100 microns, and a highly developed specific surface area (from 100 m<sup>2</sup>/kg for sandy loams and clay loams to 100,000 m<sup>2</sup>/kg for clays) [19]. Clay minerals have a layered structure with a rigid or sliding crystal structure in which two two-dimensional tetrahedral silica-oxygen grids surround two-dimensional octahedral alumohydroxyl grids on both sides [20].

In addition, coagulative and transition contacts have a significant effect on the properties of clay soils. These contacts form between mineral particles due to long-range molecular, magnetic, electrostatic and valence forces and are characterized by low strength and reversible nature of destruction [19]. The mechanism of formation of point (transition) contacts in clay dispersed systems related to the phenomenon of «self-assembly», which occurs due to the redistribution of nanoparticles and crystallization of water-soluble salts within the contact gap, which leads to an increase in the area of interaction between the particles [21]. Less often, high-strength phase contacts form between clay particles due to an increase in the contact area (crystallization) or the development of a new phase at the contact (cementation). This contributes to the improvement of the physico-chemical and physico-mechanical characteristics of clay soils [19].

It is known that one of the most effective and widely used methods for creating phase contacts between mineral particles of clay soils is the introduction of substances called active mineral additives [22–27]. Various industrial waste can be used as additives: pulverized fuel ash, hydraulic removal ash, sewage sludge combustion ash, granulated blast furnace slag, cement and lime dust, dolomite lime, ceramic waste, rice husk ash, waste paper, phosphogypsum and palm oil production ash.

These materials are characterized by a high content of calcium hydroxide, which enters into chemical interaction (pozzolanic reaction) with active siliceous and aluminous components of the soil to form cementing compounds or gels – calcium silicate hydrate (CSH) according to scheme 1. Additionally, calcium aluminate hydrate (CAH) and calcium aluminosilicate (CASH) may also form.



In [28–30], the effectiveness of using carbide sludge as an active additive was proved. This is a multi-tonnage lime-containing waste, formed annually in amounts ranging from about 18 to 70 million liters as a result of quenching calcium carbide in the production of acetylene. The introduction of carbide sludge into the clay soil in an amount of up to 9% from the soil mass on dried basis increases the strength, water resistance and frost resistance of the soil. Besides that, the study of the particle size distribution and mineral composition showed that clay soils are characterized by the pozzolanic activity, which is the ability of active clay components in the soil to interact with calcium hydroxide [31]. However, to date, the proposed mechanism of interaction in the «clay soil – carbide sludge» system is based only on the literature data and has not been experimentally verified.

Thus, the purpose of this research is to study the mechanism of structure formation in the «clay soil – carbide sludge» dispersed system.

## METHODS AND MATERIALS

### Materials

Due to the remoteness and inaccessibility of the northern and Arctic territories, as well as due to the restriction of economic activity in some territories, a model system of clay soil was created based on bibliographic and archival research by mixing the clay component and sand in a ratio of 1:6, respectively. We selected the river poly-mineral sand from the «Krasnoflotsky-Zapad» deposit with a fineness modulus of  $M_f = 1.1$ . Saponite-containing material was used as a clay component, which is a multi-tonnage waste (up to one million tons per year) of industrial enrichment of kimberlite ores from the diamond deposit named after M.V. Lomonosov. The developed soil model according to the mineral composition, particle size distribution and physical properties presented in [31] corresponded to sandy loams as one of the characteristic types of the soils in the Northern part of the European Russia.

The carbide sludge was collected in the form of a suspension from the sludge collector at the Carbide and Ferroalloy Plant, in Inta town (Komi). It was then dried to

Table 1

## Chemical composition of the carbide sludge

Component	Ca(OH) <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaCO <sub>3</sub>	CaSO <sub>4</sub>	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>
Content, %	79.6	0.2	14.9	1.0	3.0	1.3

Table 2

## Characteristics of the carbide sludge

Property	The aggregate state	Hazard class	Humidity, %	Activity level (content of active CaO), %
Value	Other dispersed systems	4	91.40	56

a constant mass at a temperature of  $60\pm 5^\circ\text{C}$  and sieved to eliminate conglomerates that formed during the drying process. The chemical composition and characteristics of the carbide sludge are presented in the Tables 1 and 2 respectively. Based on the fact that clay minerals are the most active component of clay soils, we decided to exclude sand from the model system to study the mechanism of structure formation in the «clay soil – carbide sludge» dispersed system. We then focused on the interaction between «saponite-containing material – carbide sludge».

## Methods

Initially, two groups of samples were created:

1) The dry initial components: saponite-containing material  $S_1$ , carbide sludge  $CS_1$  and a mixture of saponite-containing material and carbide sludge ( $S-CS_1$ );

2) The dry initial components were mixed with water (5% of the dry mixture weight, which corresponds to optimal conditions of structure formation [30]), dry-rodged and hardened under normal conditions (at a temperature of  $20\pm 2^\circ\text{C}$  and relative humidity  $95\pm 5\%$ ) for 28 days: saponite-containing material  $S_2$ , carbide sludge  $CS_2$  and a mixture of saponite-containing material and carbide sludge ( $S-CS_2$ ).

The rational ratio of components in the  $S-CS$  mixture for groups 1 and 2 of the samples was assumed to be the same, and it was 7:1, respectively [31].

To study the mechanism of structure formation in the «clay soil – carbide sludge» dispersed system, a set of scientific research methods were used, including microstructural, differential thermal and X-ray phase analysis.

The microstructural features of the samples were examined using scanning electron microscopy (SEM) with a Carl Zeiss Sigma VP scanning electron microscope (Carl Zeiss, Germany). The morphometric parameters of the microstructure (specific adsorption surface area and porosity) were determined by the sorption method on an

Autosorb-iQ-MP analyzer (Quantachrome instruments, USA) and calculated using the Brunauer–Emmett–Teller (BET) method.

The amount of reduction in the relative pore volume was calculated using the formula:

$$-\Delta P = \frac{P_2 - P_1}{P_1} \cdot 100 \%, \quad (2)$$

where  $P_2$  – the pore volume in the sample of the group 2,  $\text{cm}^3/\text{g}$ ;  $P_1$  – the pore volume in the sample of the group 1,  $\text{cm}^3/\text{g}$ .

The “–” sign indicates a decrease in the relative pore volume.

Differential thermal analysis (DTA) was performed on a thermal analyzer Discovery SDT 650 (TA Instruments, USA) in alumina crucibles in a nitrogen atmosphere (consumption 50 ml/min) at a heating rate of  $10^\circ\text{C}/\text{min}$ .

X-ray phase analysis was performed on a Shimadzu XRD-7000 S X-ray diffractometer (Shimadzu, Japan) on a standard holder, with a rotation of 30 rpm. Operating parameters of the X-ray tube: accelerating voltage 40 kV, current 30 mA, target material – Cu. The scanning range at an angle of  $2\theta$  is from  $10$  to  $150^\circ$ , the scanning speed is  $2 \text{ deg}/\text{min}$ , the measurement pitch is  $0.02^\circ$ . Similarity matching was carried out using the PDF-2 database (Powder Diffraction File™ PDF-2 Release 2010, International Centre for Diffraction Data), taking into account the elemental composition of the sample.

## RESULTS AND DISCUSSION

The results of the specific adsorption surface area, presented in Table 3, indicate that the values of this parameter for the samples from the group 2 are lower than those for the samples from the group 1. This difference can be attributed to the different manufacturing processes. However, the additivity law is not followed for either group of samples, confirming the structure formation in the  $S-CS$  system.

Table 3  
Specific adsorption surface area of the samples

Sample	S <sub>1</sub>	S <sub>2</sub>	CS <sub>1</sub>	CS <sub>2</sub>	(S–CS) <sub>1</sub>	(S–CS) <sub>2</sub>
Specific surface area, m <sup>2</sup> /kg	41 255	18 530	7609	1328	21 933	8870

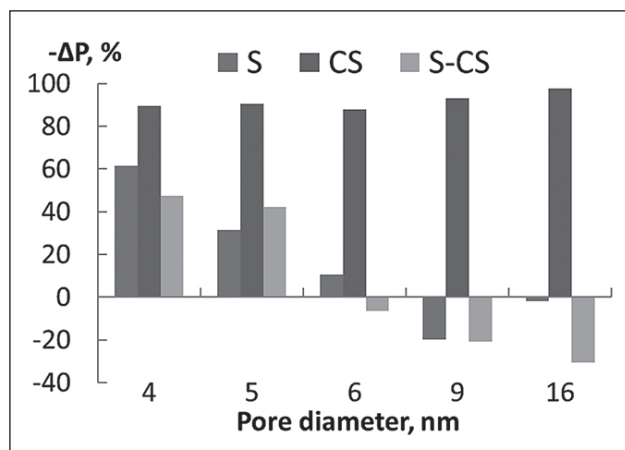


Fig. 1. Dependence of the decrease in the relative volume of pores on their diameter for the samples: S – saponite-containing material; CS – carbide sludge; S–CS – modified clay soil

An analysis of the morphometric features of the microstructure of the samples (Figure 1) showed that the specific volume of pores with a diameter greater than 9 nm increased in the saponite-containing material (S samples).

This is due to the formation of reversible coagulative and transition contacts (binds) between clay particles facilitated by a thin equilibrium liquid film up to several tens of nanometers in thickness, which is partially removed in a degassing at a temperature of 100°C. In the carbide sludge (CS samples), the specific volume of pores with a diameter greater than 4 nm decreased due to compaction of the mixture during sample forming and the physico-chemical processes of structure formation during carbonate hardening. In the modified clay soil (S–CS samples), the specific volume of pores with a diameter of 4–5 nm decreased, which is associated with gelling from particles of new hydrate formations, and the volume of pores with a diameter of more than 6 nm increased, which, in our opinion, indicates the process of contraction.

According to the results of the differential thermal analysis (Figure 2), the thermogram 3 ((S–CS)<sub>2</sub> sample) shows a decrease in the intensity of the endothermic effect in the range of 460 to 470°C, which is associated with the decomposition of calcium hydroxide. This indicates that the carbide sludge is interacting with the active components of the saponite-containing material. In addition, there is an endothermic effect at 750°C, characteristic of the decomposition of calcium silicate hydrate (CSH).

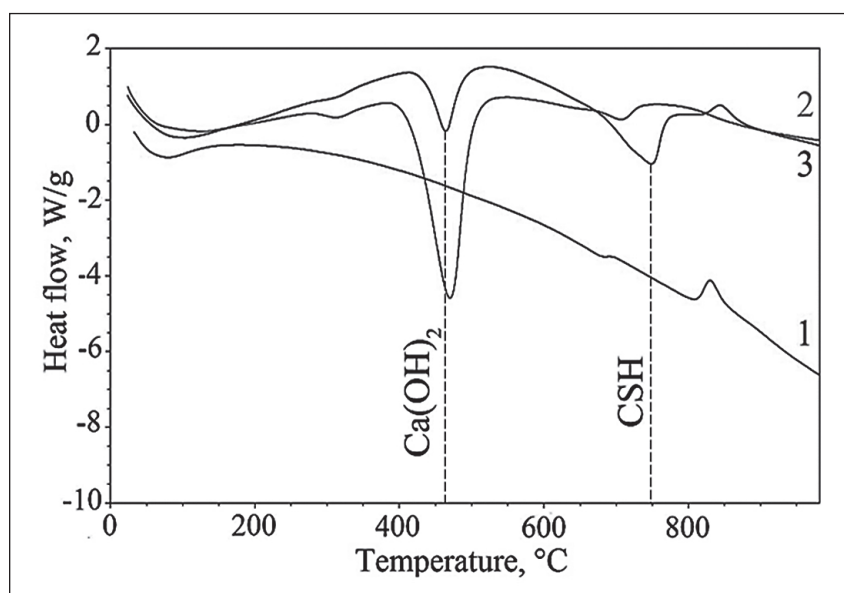


Fig. 2. Differential thermal analysis (DTA) for the group 2 of the samples: 1 – saponite-containing material (S<sub>2</sub>); 2 – carbide sludge (CS<sub>2</sub>); 3 – a mixture of the saponite-containing material and the carbide sludge (S–CS)<sub>2</sub>; CSH – calcium silicate hydrate



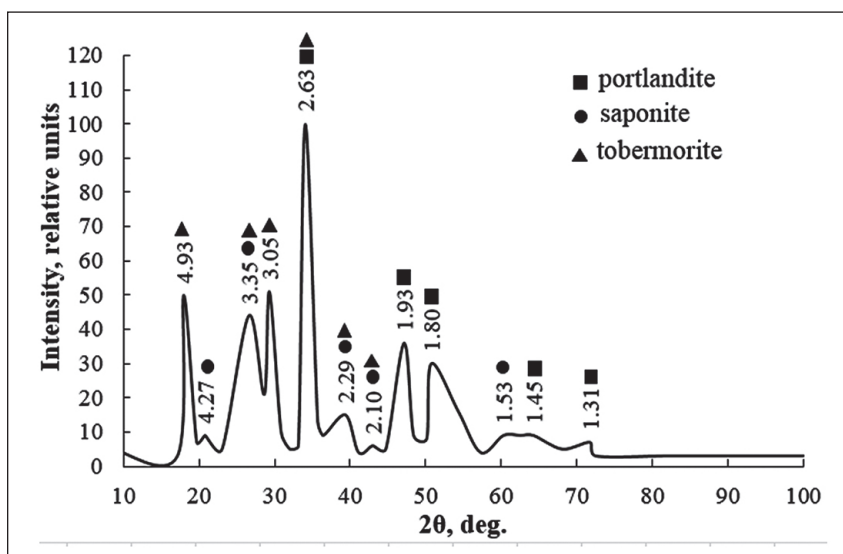


Fig. 3. X-ray photograph of the sample from the group 2 from a mixture of saponite-containing material and carbide sludge (S–CS)<sub>2</sub>

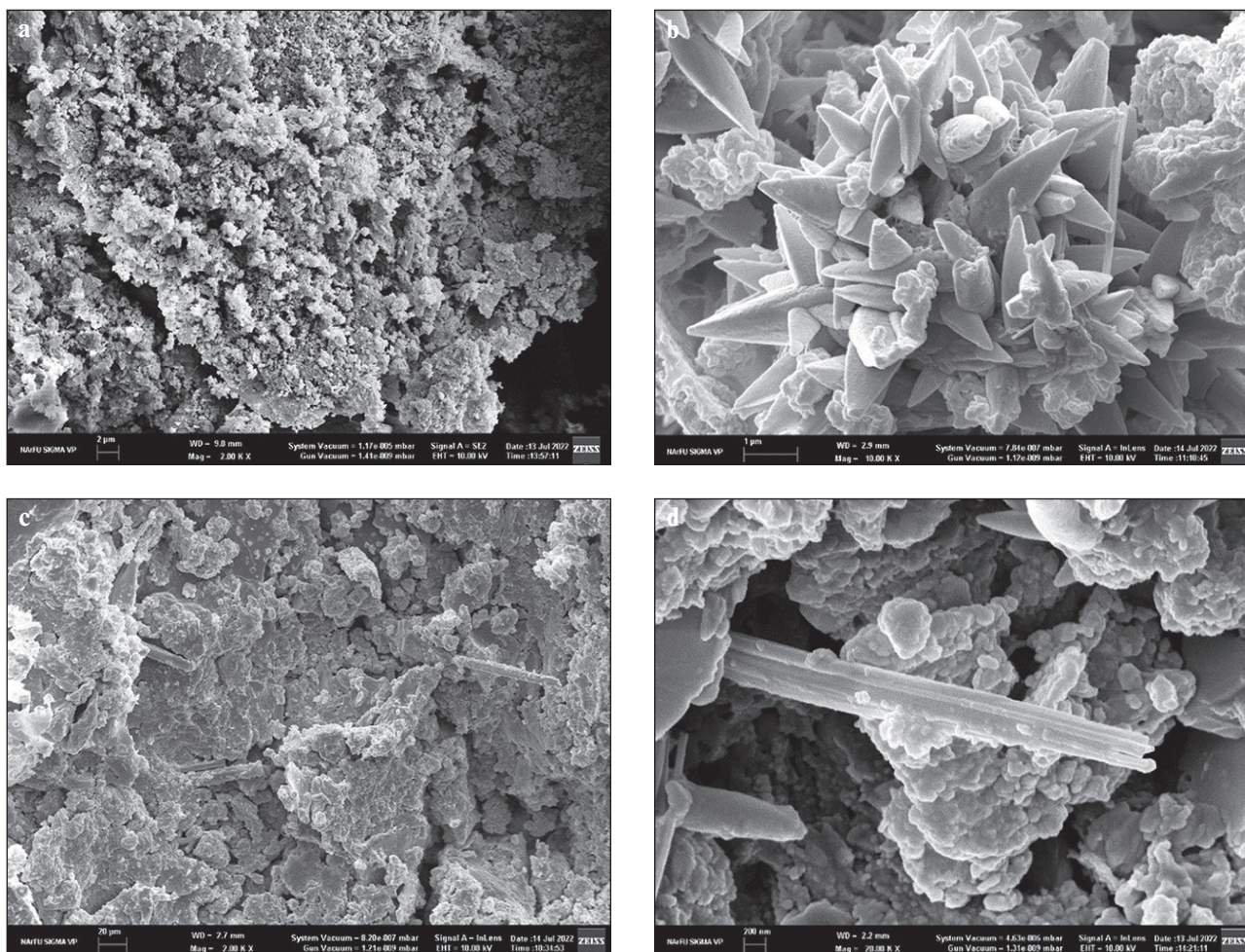


Fig. 4. The microstructure of the sample from the group 2 from a mixture of saponite-containing material and carbide sludge (S–CS)<sub>2</sub>: the images show the sample at different magnifications: a, c – ×2000 magnification; b – ×10,000 magnification; d – ×20,000 magnification, where a, b, c, d are photographs of different areas on the sample’s surface

The DTA results are supported by X-ray phase analysis (Figure 3). X-ray photographs of the (S–CS)<sub>2</sub> sample present reflexes, which are characteristic of the tobermorite group hydrosilicates, and they are absent on the X-ray photographs of the saponite-containing material.

Analysis of microstructural features in the (S–CS)<sub>2</sub> system (Figure 4) showed the formation of the initial phases of calcium silicate hydrate consisting of white microscopic aggregates in the form of randomly oriented lamellar and needle-like crystals 1–60 microns in length.

## CONCLUSION

Thus, using microstructural, differential thermal, and X-ray phase analysis, the mechanism of structure formation in the «clay soil – carbide sludge» dispersed system was established. The mechanism involves the interaction of carbide sludge with active clay components in the soil with the formation of calcium silicate hydrate, contributing to the formation of phase contacts between mineral particles of the soil and, as a result, improving its physico-chemical, and physical and mechanical characteristics.

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CONTRIBUTION OF THE AUTHORS

**Yulia V. Sokolova** – conducting experimental work; collecting and systematizing experimental data

**Maria A. Frolova** – conducting a literature review on the subject of the study

**Arkady M. Ayzenshtadt** – development of the concept and development of research methodology

**Evgeny V. Korolev** – processing and analysis of the experimental data; drawing up final conclusions.

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