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The interaction between the kaolinite or bentonite clay and plasticizing surface-active agents

Взаимодействие каолиновой и бентонитовой глин с пластифицирующими поверхностно-активными веществами

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Abstract. The influence of a number of superplasticizers of various chemical bases on the physical and technical properties of clay soils has been studied. Model soils are considered as clay soils: kaolin clay with kaolinite mineral content up to 95 %, bentonite clay with montmorillonite content up to 70%. The greatest increase in the physical and technical properties of clay soils is achieved when they are modified by a polycarboxylate superplasticizer, which is due to adsorption of polymer molecules on clay minerals even with a negative charge of chips and basal planes. This is explained by the ability of side chains of the polycarboxylate ester, which has a similar composition with polyethylene glycols, to be adsorbed on the aluminosilicate layers of clay minerals by the formation of hydrogen bonds. The interaction of kaolinite and montmorillonite included in kaolin and bentonite clay with a polycarboxylate superplasticizer was studied by infrared spectroscopy. It was found that the additive under study belongs to a type of polycarboxylate superplasticizers modified with organo-silanes. Introduction to kaolin and bentonite clays leads to chemisorption with clay minerals and the formation of organomineral bonds, which leads to an increase in the strength characteristics of clays.

Аннотация. Проведены исследования влияния ряда суперпластификаторов различной химической основы на физико-технические свойства глинистых грунтов. В качестве глинистых грунтов рассмотрены модельные грунты: каолиновая глина с содержанием минерала каолинита до 95 % и бентонитовая глина с содержанием монтмориллонита до 70 %. Наибольшее повышение физико-технических свойств глинистых грунтов достигается при модификации их поликарбоксилатным суперпластификатором, что обусловлено адсорбцией молекул полимера на глинистых минералах даже при отрицательном заряде сколов и базальных плоскостей. Это объясняется способностью боковых цепей эфира поликарбоксилата, имеющий аналогичный состав с полиэтиленгликолями, адсорбироваться на алюмосиликатных слоях глинистых минералов посредством образования водородных связей. Методом ИК-спектроскопии изучено взаимодействие каолинита и монтмориллонита, входящих в каолиновую и бентонитовую глины с поликарбоксилатным суперпластификатором. Установлено, что исследуемая добавка относится к типу поликарбоксилатных суперпластификаторов, модифицированных органо-силанами. Введение в каолиновую и бентонитовую глины, приводит к хемосорбции с глинистыми минералами и образованию органоминеральных связей, что приводит к повышению прочностных характеристик глин.

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Introduction

Clay soils have a wide range of mineral composition and consist of different types of clay and non-clay minerals [1, 2]. They are the product of weathering of feldspathic and other silicate rocks, composed mainly of montmorillonite, kaolinite and hydromica with an admixture of mainly relict minerals – quartz, mica, secondary calcite, opal and others. Kaolinite and montmorillonite are the most common clay minerals including in soils. Kaolinite – is a relatively stable mineral contained relatively in 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 montmorillonite ($2\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 4\text{H}_2\text{O}$) is the second most occurring clay mineral. It is formed under alkaline conditions in marine sediments and in the weathering crust. It belongs to a subclass of phyllosilicates, has the ability to a strong bulking [3]. Usually clay soils are subdivided on mineral composition into monomineral (kaolinite, montmorillonite, illite, etc.) and polymineral consisting of various clay and relict minerals [4, 5].

It is known that intromission of different surface-active agents into clay soils changes its structure and properties [6]. This is because of the presented in the soils finely dispersed and colloidal particles leads to significant increase the total surface of the partition between solid phase and liquid medium and hence to increase the free surface energy [7]. This allows to increase the physical and chemical activity of the particles, to increase the adsorption capacity of the surface, that allows the surface-active agents, concentrated on interfaces, to form the finest adsorbed layers, dramatically altering the molecular nature and properties of the soil surface [8].

Previous studies [9] have shown that the polycarboxylate superplasticizers compared with plasticizing additives, other have the greatest effect on increasing of physical and technical properties of soil-cement in accordance with Russian State Standard GOST 23558-94. It is also determined by a number of positive effects of polycarboxylate superplasticizers on the physical properties of clay soils with different mineral composition [10]. However, the effect of plasticizing surface-active agents with various chemical bases on the physical and strength properties of clay soils has not been sufficiently investigated.

In this way, the aim of the work was to determine the most effective plasticizing surface-active agents for kaolinite and bentonite clay on the bases results of experimental studies.

To achieve this aim, the following tasks are formulated:

- 1) to study the influence the plasticizing surface-active agents with various chemical bases on properties of kaolinite and bentonite clays by methods of laboratory studies of the construction and technical properties of soils;
- 2) to investigate the interactions between considering clays with the polycarboxylate superplasticizer by the infrared spectroscopy.

Materials and methods of research

The following clay soils were used for the research:

- 1) kaolinite clay (KC) with the plasticity index 18.07 and content of sandy particles 6.02 %, pH of aqueous extract 8.3, content of mineral kaolinite up to 95 %;
- 2) bentonite clay (BC) with the plasticity index 22.32 and content of sandy particles 8.00 %, pH of the aqueous extract 8.3, the mineral content of montmorillonite up to 70 %.

The following additives as plasticizing surface-active agents are used.

- 1) Sulfonated melamineformaldehyde compound (SMF) – superplasticizer Melment F10, which is a white powder. It consists of sulfonated powdered polycondensated products based on melamine, obtained by spray drying. Producer BASF Constraction Polymers, Germany.
- 2) Sulfonated naphthaleneformaldehyde compound (SNF) – superplasticizer SP-1 (TU 6-36-0204229-625-90 with changes 1, 2), which is a brown powder. It consists of a mixture of oligomers and polymers of different relative molecular weight obtained by condensation of naphthalene sulfonic acids with formaldehyde and technical lignosulfonates and neutralized with sodium hydroxide. The main component (the active substance) is the unreacted salt of β -naphthalenesulfonic acid and sodium sulfate. Producer company "Polyplast", Russia.

3) Lignosulfonate technical (LST) – plasticizer LST (TU 2455-028-00279580-2004), which is a powdery powder from light yellow to brown color. It is a natural water-soluble sulfonic derivative of lignin, formed during the sulfite process of wood delignification. Producer company "Solikamskbumprom", Russia.

4) Polycarboxylate (PCE) – polycarboxylate superplasticizer Pantarhit PC 160 Plv is a light gray powder obtained by spray drying from a polymer solution. The active substance is polyacrylic acid. Producer is BASF Construction Polymers, Germany. Pantarhit PC 160 Plv was selected in comparison and research as the most effective polycarboxylate superplasticizer based on the results [9].

Dosages of plasticizing additives are 0.05 %, 0.5 %, 1 %, 2 % of the clay soil weight.

Building and technical indicators are accepted following: physical properties – the plasticity index, the optimum moisture content and maximum density, the strength properties – the ultimate compressive strength. The plasticity index (I_p) is calculated in accordance with Russian State Standard GOST 5180-2015 as the difference between the creep stress (W_c) and the rolling-out limit (W_r). Optimum moisture content (W_{opt}) and maximum density (ρ_{max}) are determined in accordance with Russian State Standard GOST 22733-2016 on the standard compaction measuring device of the SoyuzdorNII. To determine the ultimate compressive strength (R_{str}), samples were prepared with the maximum density, which were stored for 7 days under air conditions, and then dried to constant weight in a drying oven at a temperature of 105 °C and tested on a press.

The registration of infra-red spectrum samples was made on IR Fourier-spectrophotometer by Perkin-Elmer firm, the Spectrum 65 model, using a Miracle ATR (ZnSe crystal) attachment in the 4000-650 cm^{-1} area, usually at 20 scans. The background spectrum was recorded and subtracted automatically. The powder, after grinding clay soil and additives, was pressed against the crystal by a special clamp, which is part of the set-top box. After registration, automatic correction and spectrum conservation were automatically performed.

Before registration of the infra-red spectrum, the clay soils were dried at a temperature of 105 °C to a constant mass. Samples for the study were prepared as follows. The surface-active agents were previously diluted in a ceramic bowl with distilled water. Additives were added in amount of 20 % of the clay soil weight. Next, a pre-dried clay soil was added for the paste formation. In this case, the clay soil paste was in the state of creep stress and stored for 7 days in normal-moisture conditions. After that, the modified clay soil was dried, as well as unmodified. The dried clay soil samples were crushed in a vibratory mill for 5 minutes to particles of micron size.

Results and Discussion

Figures 1 and 2 show the research results of the influence of surface-active agents on the construction and technical properties of kaolinite and bentonite clays. It was established that the parameters of standard compaction, creep stress and rolling-out limit, the plasticity index and ultimate compressive strength are changed during intromission of plasticizing surface-active agents into clay composition.

Introduction the SMF from 0.05 % to 2 % of the clay mass weight decreased the optimum moisture content for KC on 0.11–8.22 %, for BC on 0.08–5.47 %. The maximum density increased in KC on 4.73 %, in BC on 3.29 %. The creep stress and the rolling-out limit decreased in KC on 0.26–9.52 % and 0.12–5.43 %, in BC on 2.13–4.33 % and respectively 0.08–4.33 %. The plasticity index decreased for KC on 0.50–16.93 %, for BC on 0.05–6.10 %. The ultimate compressive strength was increased in KC on 2.46–24.18 %, and in BC on 1.38–56.55 %.

Adding the SNF from 0.05 % to 2 % of the clay mass weight, the optimum moisture decreased for KC on 0.11–9.07 %, for BC on 0.11–5.66 %. The maximum density increased in KC on 5.41 %, in BC on 3.95 %. The creep stress and the rolling-out limit decreased in KC on 0.75–10.72 % and 0.31–5.49 %, in BC on 2.17–7.29 % and respectively on 0.11–4.64 %. The plasticity index decreased for KC on 1.55–20.20 %, for BC on 0.09–6.19 %. The compressive strength was increased in KC on 4.10–30.33 %, in BC it is 1.38–58.62 %.

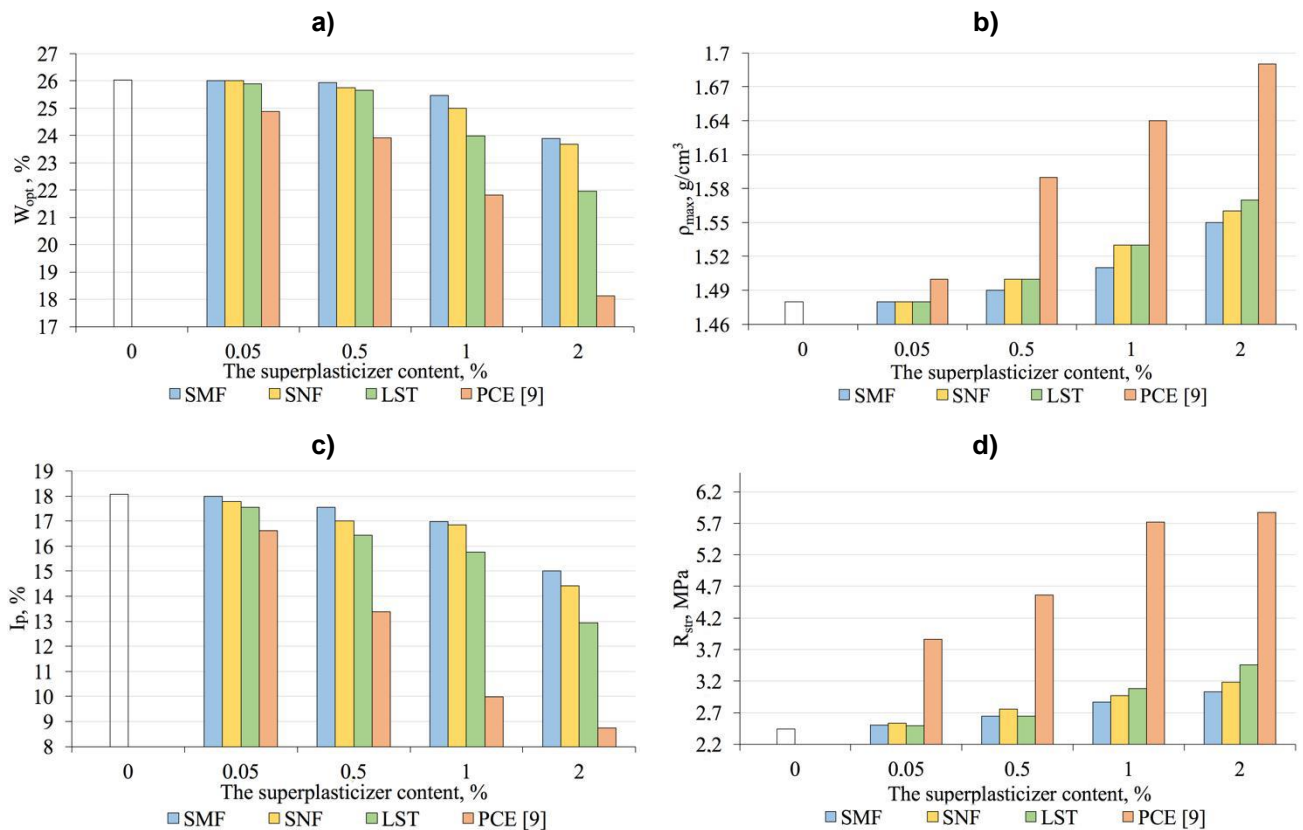


Figure 1. Dependences physical and strength properties of kaolinite clay of the dosage of plasticizing surface-agents:
a) the optimum moisture; b) the maximum density;
c) the plastic index; d) the ultimate compressive strength

Introduction the LST from 0.05 % to 2 % of the clay mass weight led to a decrease of the optimum moisture content of KC on 0.54–15.67 %, BC on 0.23–6.47 %. The maximum density is increased in KC on 6.08%, in BC on 5.26 %. The creep stress and the rolling-out limit decreased in KC on 1.24–13.89 % and 2.82–28.39 %, in BC on 2.21–7.52 % and 0.19–4.67 %, respectively. The plasticity index decreased for KC on 2.82–28.39 %, for BC on 0.09–6.66 %. The compressive strength limit was increased in KC on 2.05–41.80 %, in BC it increased on 1.38–74.48%.

The use of PCE, that was studied in [9], in an amount of 0.05 % to 2 % of the clay mass weight ensured a reduction in the optimum moisture content of the KC on 4.46–30.35 %, BC on 1.07–18.36 %. The maximum density increased in KC on 1.35–14.19 %, in BC on 1.32–11.84 %. The creep stress and the rolling-out limit decreased in the KC on 3.76–23.25 % and 1.40–51.63, in BC on 3.13–21.97 % and 0.46–19.54 %, respectively. The plasticity index decreased for KC on 8.02–51.63 %, for BC on 1.88–35.37 %. The compressive strength limit increased in KC on 58.20–140.57 %, in BC on 2.76–115.86 %.

According to the data presented in Figures 1 and 2, it can be seen that among the studied plasticizers of various chemical bases the most effective plasticizing surface-active agent was polycarboxylate superplasticizer Pantarhit PC 160 Plv (PCE). It should be noted that in [9] other polycarboxylate superplasticizers (Odolit-K, Giperlit, Melflux 2641 F), as well as Pantarhit PC 160 Plv, were more effective than SMF, SNF and LST.

PCE can be adsorbed on clay minerals even with a negative charge of chips and basal planes. This is due to the ability of the side chains of PCE, that have a similar composition with polyethylene glycols, to be adsorbed on the aluminosilicate layers of clay minerals through hydrogen bonds [11–17], which probably explains their high efficiency.

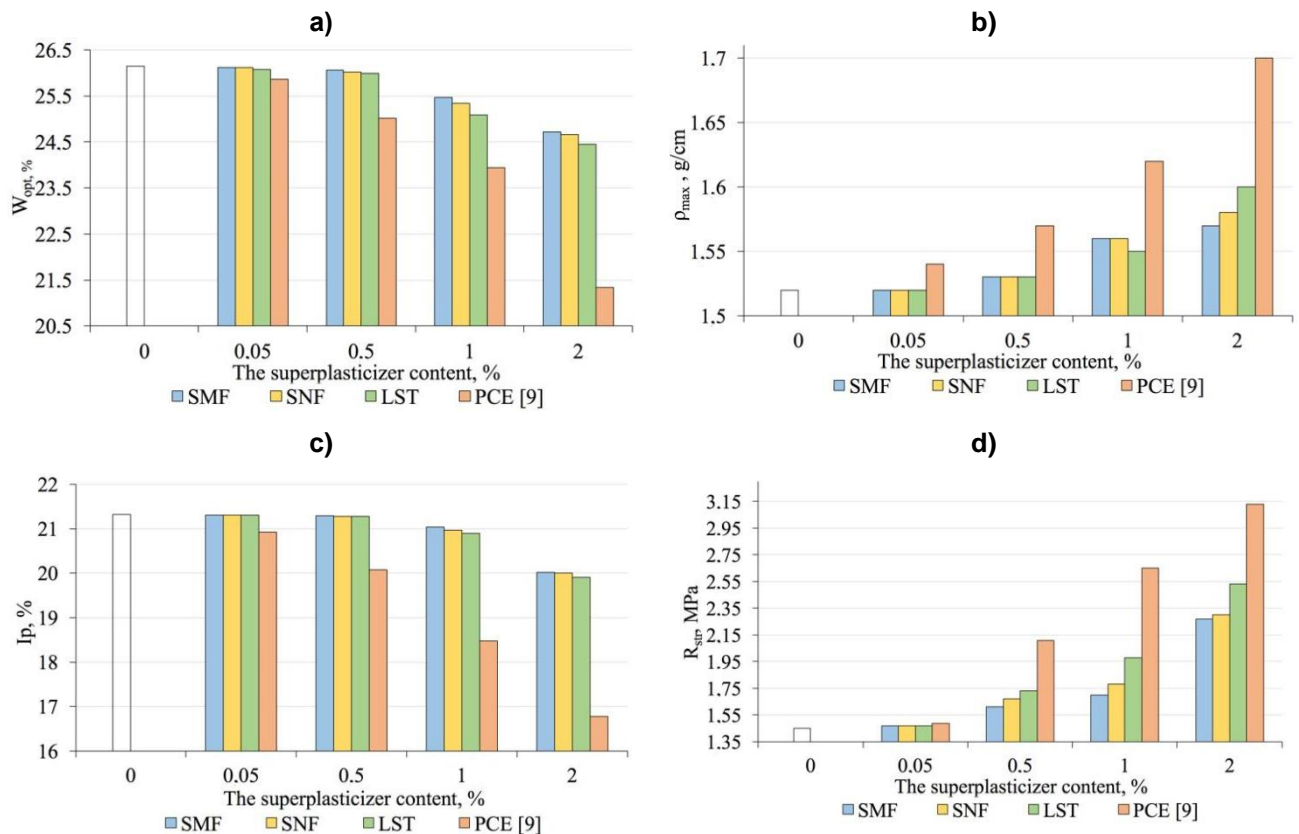


Figure 2. Dependences physical and strength properties of bentonite clay of the dosage of plasticizing surface-agents: a) the optimum moisture; b) the maximum density; c) the plastic index; d) the ultimate compressive strength

According to E. Tombacz, M. Szckres, J.L. Suter, P.V. Coveney, L. Lei, J. Plank [18–20], injecting the PCE in the clay soil increases the basal reflection thickness of montmorillonite from 1.23 to 1.72–1.77 nm, but in kaolinite and muscovite practically does not change. Montmorillonite sorbs about 230 mg/g of PCE, and kaolinite and muscovite sorb about 20 mg/g, which probably explains the more effective effect of PCE on KC, in comparison with BC.

LST also showed greater efficiency in comparison with SMF and SNF. Perhaps this is due to the fact that the molecule of LST is spheroidal and consists of disc-shaped aggregates up to 200 nm in size, where hydrated groups are localized in the internal structure of associates [21–24]. Such molecules configuration limits the water mobility, but at the same time, its lyophobicity increases. Such a structure, in spite of the fact that the mechanism of interaction with particles surface is mainly based on the forces of electrostatic repulsion, can lead to a significant decrease in the interaction between individual clay particles with adsorbed on their surface surface-active agents.

As is known, a continuous spatial grid with a coagulation structure acquiring plastic-viscous properties is formed in the clay-water system. The increase of the thickness of the water shells has a plasticizing effect on the clay, while reducing the strength of the structure and its ability to permanent elastic deformation [8]. Adsorption of surface-active agents on clay minerals in the clay-water system allows to reduce the surface energy at the interface, which contributes to the increase on density due to the decrease the water demand (optimum moisture), to the decrease in creep stress and the rolling-out limit [8, 25], and ultimately to the increase of the strength of the soil.

PCE showed the greatest efficiency among the considered plasticizers, by allowing to reduce the optimum moisture and plastic index, to increase the maximum density and compressive strength limit of kaolinite and bentonite clay. The positive influence of PCE on both the physical and mechanical characteristics of the cement stone [26] and on the physical and strength properties of clay soils explains the strength and frost resistance increase of soil cements modified with such additives.

At the next stage, the interaction of kaolinite and bentonite clays with a polycarboxylate superplasticizer was studied by methods of infrared spectroscopy.

It can be seen from Figure 3 that each of the spectra is characterized by the presence of intense absorption bands in the area of 1000–1200 cm^{-1} . These peaks are due to valence vibrations of Si-O-Si bonds [27]. In our case, they include a doublet 1024-999 cm^{-1} in the spectrum of KC and an intense peak of 1097 cm^{-1} with a shoulder of 1060 cm^{-1} in the PCE spectrum. Probably, the investigated additive PCE (Pantarhit PC 160 Plv) belongs to the group of polycarboxylate superplasticizers modified with organosilanes [28-30], as shown in Figure 4.

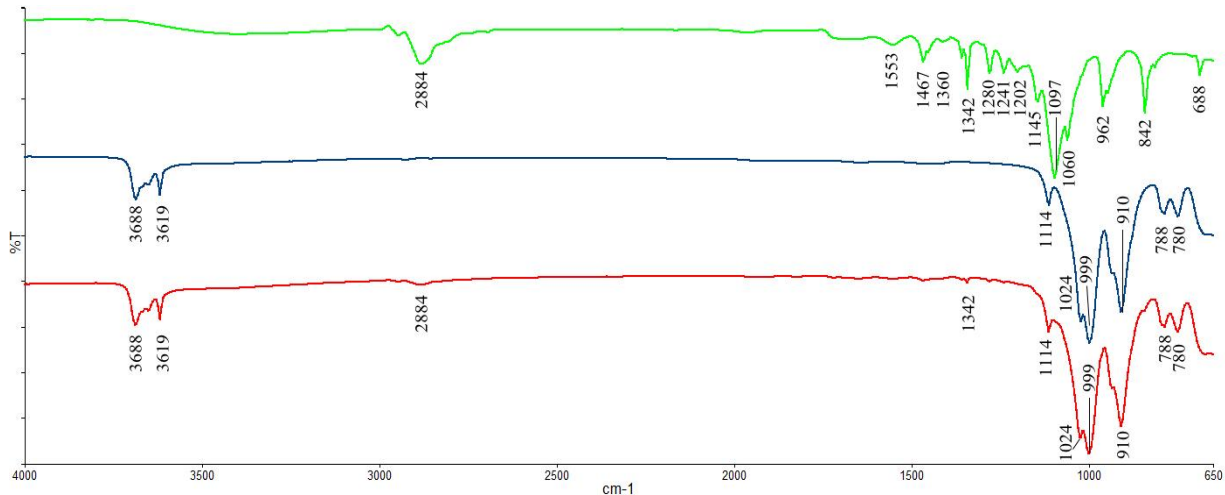


Figure 3. Infrared spectroscopy in the study of kaolinite clay and polycarboxylate:
 — PCE, — KC, — KC + PCE system

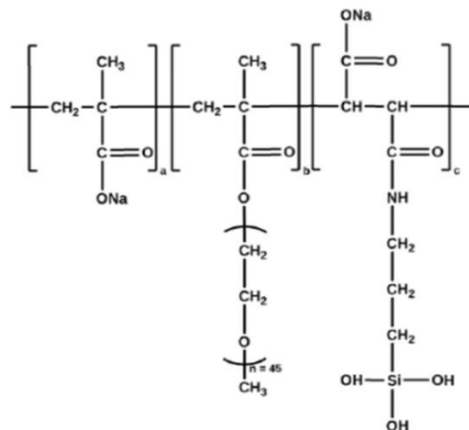


Figure 4. The chemical formula of a polycarboxylate superplasticizer modified by organosilanes, according to J. Plank, E. Sakai, C.W. Miao, C. Yu, J.X. Hong

Peaks 3688, 3673, 3619 cm^{-1} on the KC and KC + PCE curves correspond to the stretching vibrations of the -OH gibbsite layer of the kaolinite mineral in the KC. With the injection of the PCE additive leads to intense band of 1097 cm^{-1} disappears in the spectrum of the KC + PCE system, although the remaining peaks corresponding to the PCE spectrum are conserved. In particular, the bands 2884 cm^{-1} (stretching vibrations of CH_2 and CH_3 groups), 1467 cm^{-1} and 1342 cm^{-1} (deformation vibrations of the same groups) are retained. This indicates that the chemical interaction between kaolinite and PCE proceeds, and this interaction leads to the conversion of the Si-O-Si bonds of the PCE additive to other structures, apparently chemically related to kaolinite. The aliphatic portion of the PCE containing the CH_2 and CH_3 groups does not appear to undergo any changes.

Similar results were obtained with adding PCE into BC.

Oscillations of Si-O-Si bonds in the BC spectrum correspond to peaks of 1002, 912, 873 cm^{-1} . When PCE was injected into BC, results similar to KC have been obtained. The corresponding infrared spectroscopy is shown in Figure 5. Intensive spectra of 1097 and 1060 cm^{-1} , corresponding to stretching vibrations of Si-O-Si bonds in PCE, practically disappear in the spectra of the modified BC. The presence

of aliphatic CH₂ and CH₃ groups will remain. In this case, the modifiers chemically bind to the mineral by Si-O bonds, which leads to their conversion. Interactions on hydroxyl groups as a modifier and a mineral are not observed.

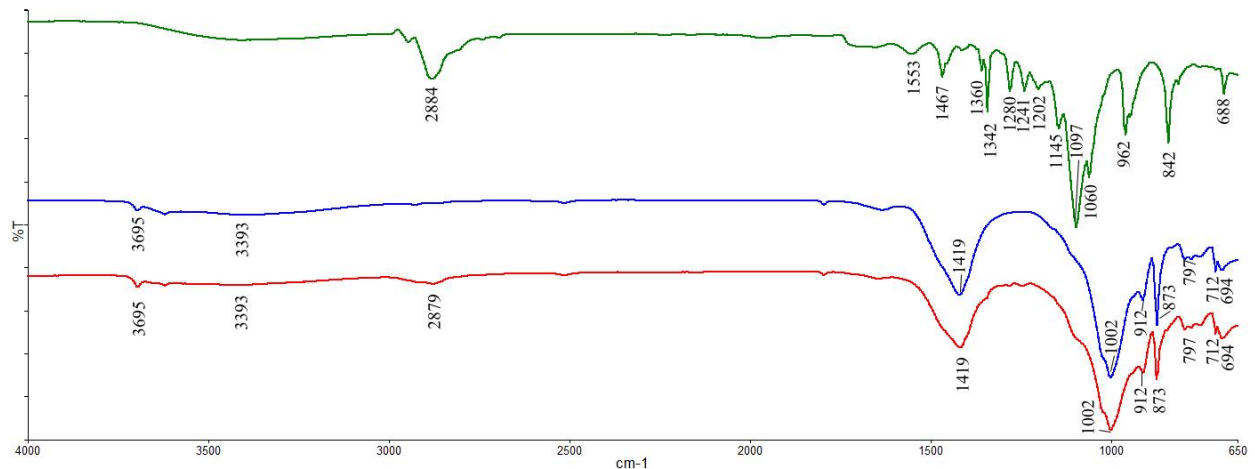


Figure 5. Infrared spectroscopy in the study of bentonite clay BC and polycarboxylate PCE:
 — PCE, — BC, — BC + PCE system

It should be noted that according to W. Fan, F. Stoffelbach, J. Rieger, L. Regnaud, A. Vichot, B. Bresson, N. Lequeux, J. Witt, J. Plank [28, 30] organosilane modified polycarboxylates with -Si-OH bonds adding into cement systems can form chemical bonds with cement neoplasms of the type C-S-H-PCE. That is, these polycarboxylates in the first stage are adsorbed on the surface of the flocculum of cement, and then their chemisorption occurs on the surface of the hydrate particles. Also in the works of E. Sakai, D. Atarashi, M. Daimon, S. Ng, J. Plank [31, 32] noted the possibility of the formation of such bonds in clay minerals.

Conclusions

The influence of plasticizing surface-active agents with various chemical bases on the creep stress and the rolling-out limit, optimum moisture and maximum density, compressive strength of kaolinite and bentonite clay is considered. It was determined that the most effective among the studied surfactants was polycarboxylate superplasticizer. Polycarboxylate showed maximum efficiency in kaolinite clay.

The interaction of kaolinite and montmorillonite included in kaolinite and bentonite clay with a polycarboxylate superplasticizer (Pantarhit PC 160 Plv additive) was studied by infrared spectroscopy.

Characteristic absorption bands corresponding to vibrations of Si-O-Si functional bonds are established.

It has been shown that the introduction of polycarboxylates modified with organosilanes into kaolinite and bentonite clay leads to the disappearance of absorption bands corresponding to Si-O bonds in the surface-active agents. This fact, apparently, indicates the chemical interaction of the modifiers used with clay minerals, leading to the conversion of Si-O groups. There are no interactions (chemical or physical, like hydrogen bonding) relatively to OH groups of clay minerals and modifiers.

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