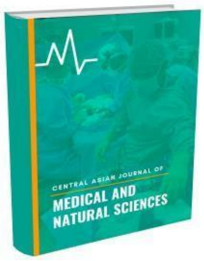

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Article

Mechanisms of Structural Transformation of Soils During Modification by Organic Structuring Agents

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Abstract: This study investigates the mechanisms of structural transformation of loamy (typical sierozem) and sandy saline soils from the Kegeyli and Kungrad districts of the Republic of Karakalpakstan under modification with organic structuring agents of different molecular nature—an anionic surfactant (sulfanol) and a water-soluble polymer (carboxymethyl cellulose, CMC). SEM–EDS analysis revealed that the saline soil is characterized by elevated contents of C (24.78 wt.%), O (46.23 wt.%), Si (12.87 wt.%), Ca (4.41 wt.%), and the presence of Na, indicating high ionic strength and compression of the electrical double layer. The sierozem soil exhibits higher Si (19.09 wt.%) and Ca (7.01 wt.%) contents and lower carbon content (10.68 wt.%), suggesting a predominantly silicate–carbonate matrix and lower salinity. FTIR spectroscopy revealed characteristic absorption bands at 3387–3400 cm^{-1} (O–H stretching), 1435–1452 cm^{-1} (carbonate groups), and 998–1004 cm^{-1} (Si–O–Si vibrations), confirming the presence of active surface sites capable of intermolecular interactions. Benzene vapor adsorption isotherms correspond to type II–IV behavior, indicating a mesoporous structure. The maximum adsorption capacity was observed at a dosage of 6 mL of structuring agent per 50 g of soil. For saline soil modified with sulfanol, adsorption reached 4.09 arbitrary units, while for the sierozem soil it reached 4.52 arbitrary units. Increasing the dosage to 8 mL resulted in a reduced increment of adsorption, indicating partial micropore blocking. It was established that sulfanol induces structural transformation primarily through a surface-energy mechanism involving adsorption and hydrophobization of pore walls, whereas CMC acts via polymer bridging and aggregate formation. The proposed colloid-chemical model explains pore-space redistribution depending on soil granulometric composition and ionic environment.

Keywords: Organic structurants, sulfanol, carboxymethyl cellulose, electron microscopy scanning, IR spectroscopy, benzene vapor adsorption isotherms, porous structure, mesopors, colloidal chemical interactions.

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Introduction

Soils of arid and semi-arid regions are characterized by structural instability caused by salinization, degradation of aggregate organization, and disturbance of pore space architecture. These processes significantly reduce water retention capacity and alter sorption properties, thereby affecting soil productivity and ecological sustainability [1–3]. The soils of the Kegeyli and Xodjeyli districts of the Republic of Karakalpakstan are represented by typical sierozems and saline sandy loam soils, which differ in granulometric composition, mineral structure, and degree of salinity [4].

The pore structure of soils is a hierarchically organized system of micro-, meso-, and macropores formed by mineral composition, particle size distribution, and interparticle interactions [5]. Colloid-chemical processes occurring at mineral surfaces—including adsorption, coagulation, and aggregation—play a decisive role in pore-space transformation [6]. Under saline conditions, these processes are strongly influenced by electrolyte concentration, which affects the thickness of the electrical double layer and interparticle forces [7].

Organic structuring agents are widely used to improve soil structure. Surfactants are capable of modifying surface energy and the hydrophilic–hydrophobic balance of mineral matrices [8], while water-soluble polymers promote aggregate stabilization through polymer-bridging mechanisms [9]. However, most existing studies examine surfactants and polymers separately and rarely provide a comparative analysis of their mechanisms in soils with different granulometric compositions. Moreover, structural changes are often assessed indirectly without integrating morphological, spectroscopic, and adsorption analyses.

Benzene vapor adsorption provides an effective tool for evaluating pore-structure evolution. As a non-polar adsorbate, benzene is sensitive to hydrophobic surface modifications and allows assessment of accessible internal surface area and mesopore redistribution [10, 11]. Despite numerous studies on soil modification with organic compounds, the mechanisms of structural transformation in saline and non-saline soils of arid regions remain insufficiently understood. In particular, the relationship between mineral composition, surface functional groups, and adsorption behavior under the action of surfactants and polymers requires further clarification.

The present study aims to establish the mechanisms of structural transformation of loamy (typical sierozem) and sandy loam saline soils from the Kegeyli and Kungrad districts of Karakalpakstan under modification with organic structuring agents—sulfanol and carboxymethyl cellulose—based on SEM–EDS, FTIR spectroscopy, and benzene vapor adsorption analysis.

Materials and Methods

Two soil types collected from the Republic of Karakalpakstan were used as research objects: a loamy soil (typical sierozem) from the Kegeyli district and a sandy loam saline soil from the Kungrad district.

Soil samples were collected from the plow layer (0–20 cm). The samples were air-dried at room temperature to a constant mass, cleared of plant residues, and sieved through a 1 mm mesh. Prior to analysis, the soils were additionally ground to obtain homogeneous material. The following organic structuring agents were used: an anionic surfactant (sulfanol) and a water-soluble polymer (carboxymethyl cellulose, CMC). Soil modification was performed using 1 wt.% aqueous solutions of the additives. To each 50 g portion of air-dried soil, 4, 6, and 8 ml of the respective solution were added, followed by thorough mixing to ensure uniform distribution of the additive. The treated samples were kept at room temperature for 24 h to stabilize the structure and then re-dried to the air-dried state [12]. Untreated soils were used as control samples. Surface morphology and elemental composition were examined using scanning electron microscopy equipped with an energy-dispersive X-ray spectrometer (SEM–EDS). Samples were mounted on conductive holders and coated with a thin gold layer to prevent surface charging. Measurements were carried out at an accelerating voltage of 15–20 kV. Elemental composition was determined in wt.% based on EDS spectra in the detectable range from C to Fe. For each sample, at least three different surface areas were analyzed, and the results were averaged. FTIR spectra were recorded in the range of 4000–400 cm^{-1} using the KBr pellet method at room temperature. Particular attention was paid to absorption bands corresponding to hydroxyl groups (3200–3600 cm^{-1}), bending vibrations of water (~1630 cm^{-1}), Si–O–Si stretching vibrations (1000–1100 cm^{-1}), and Al–OH vibrations (500–800 cm^{-1}). Benzene vapor adsorption isotherms were determined by the desiccator method at a constant temperature of 25 ± 1 °C. Analytical-grade benzene was used as the adsorbate.

A 1.0 g soil sample was placed in a sealed desiccator with a predetermined relative vapor pressure (P/P_0) established using saturated solution systems. Equilibrium was assumed when the mass change was less than 0.0002 g over 24 h. Adsorption capacity was expressed in arbitrary units per gram of soil. Adsorption isotherms were constructed as a function of relative benzene vapor pressure. The obtained isotherms were analyzed to determine the pore-structure characteristics and to evaluate the redistribution of micro- and mesopores after modification. Comparative analysis was performed for control and modified samples at different structuring-agent dosages.

Results and discussion

Surface morphology and elemental composition of the initial saline and sierozem soil samples were investigated using scanning electron microscopy coupled with energy-dispersive X-ray spectroscopy (SEM–EDS). The obtained spectra and quantitative data are presented in Figures 1, 2 and Tables 1, 2.

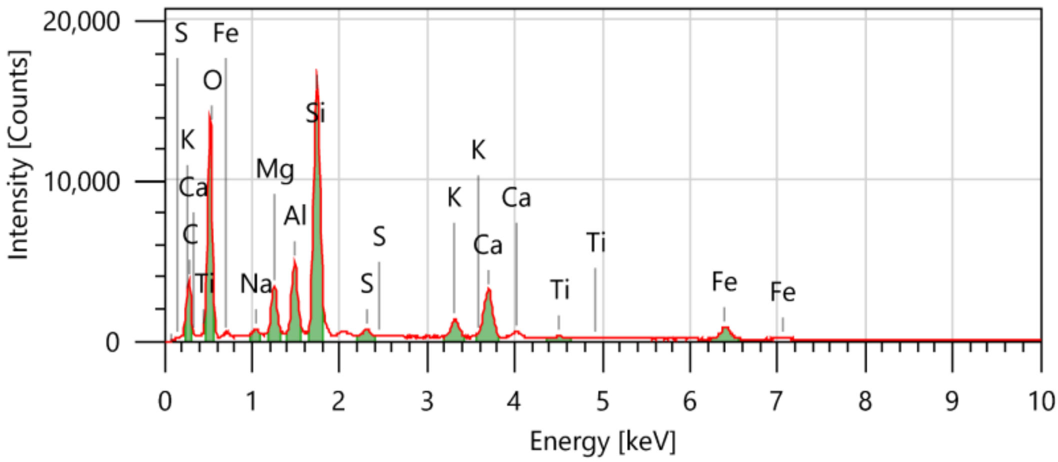


Figure 1. Scanning electron micrograph (SEM) of the initial saline soil.

Table 1. Energy-dispersive X-ray (EDS) analysis of the initial saline soil.

Element	Line	Mass%	Atom%
C	K	24.78±0.15	35.04±0.22
O	K	46.23±0.26	49.08±0.27
Na	K	0.47±0.02	0.35±0.02
Mg	K	2.61±0.04	1.82±0.03
Al	K	3.44±0.04	2.17±0.02
Si	K	12.87±0.07	7.78±0.04
S	K	0.48±0.01	0.26±0.01
K	K	1.42±0.03	0.61±0.01
Ca	K	4.41±0.05	1.87±0.02
Ti	K	0.22±0.01	0.08±0.01
Fe	K	3.07±0.06	0.93±0.02
Total		100.00	100.00
Spc_002			Fitting ratio 0.0198

The salted sample is characterized by high oxygen (46.23 max%) and carbon (24.78 max%), which is related to the presence of carbonate compounds and organic matter.

The significant proportion of silicon (12.87% max) indicates the predominance of silicate matrix, also noted are: Na (0.47% max), Ca (4.41% max), Mg (2.61% max), K (1.42% max), which confirms the salted nature of the soil and the presence of easily soluble salts. Iron content (3.07% max) and aluminium content (3.44% max) indicate the presence of clay minerals and Fe oxides.

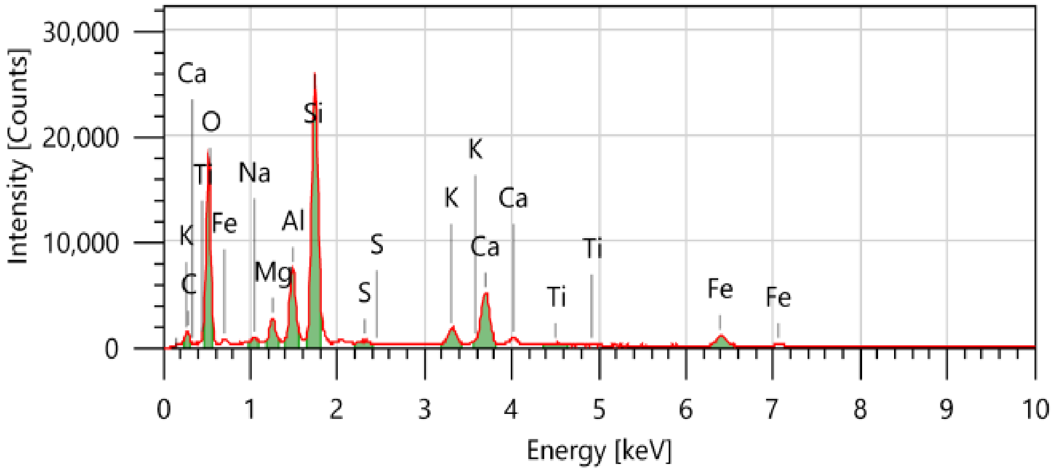


Figure 2. Electron Scanning Microscopy (EMS) of the original hydrogen soil.

Table 2. Energy Stress Analysis (EDS) of the original saline soil.

Element	Line	Mass%	Atom%
C	K	10.68±0.11	16.92±0.18
O	K	49.42±0.24	58.80±0.29
Na	K	0.62±0.02	0.51±0.02
Mg	K	1.78±0.03	1.39±0.02
Al	K	5.09±0.05	3.59±0.03
Si	K	19.09±0.09	12.94±0.06
S	K	0.29±0.01	0.17±0.01
K	K	1.88±0.03	0.92±0.01
Ca	K	7.01±0.06	3.33±0.03
Ti	K	0.33±0.02	0.13±0.01
Fe	K	3.80±0.07	1.29±0.02
Total		100.00	100.00
Spc_001			Fitting ratio 0.0207

The high electrolyte concentration explains the compression of the electrical double layer, reduction of interparticle repulsion, and increased tendency toward coagulation of the dispersed phase. This creates favorable conditions for active adsorption of the anionic surfactant (sulfanol) on the mineral surface.

The sierozem soil contains a higher fraction of Si (~19.1 wt.%) and Ca (~7.0 wt.%) and a lower carbon content (~10.7 wt.%). The predominance of the quartz–carbonate phase indicates a coarser-dispersed structure and a lower proportion of the active colloidal fraction. Morphologically (according to SEM observations), the saline soil exhibits denser aggregates with well-defined inter-aggregate voids, whereas the sierozem soil is characterized by a more dispersed granular structure.

The FTIR spectra of the initial saline and sierozem soil samples are presented in Figures 3 and 4. Spectral analysis allows identification of the mineral matrix features and surface functional groups that determine adsorption properties. In the FTIR spectrum of the saline soil, the following characteristic bands are observed 3387 cm⁻¹ — a broad O–H stretching band associated with hydroxyl groups and adsorbed water, ~1630–1452 cm⁻¹ (notably 1452 cm⁻¹) — bending vibrations of H–O–H and possible carbonate groups, 998.87 cm⁻¹ — an intense Si–O–Si stretching vibration characteristic of the silicate matrix, 873.22 cm⁻¹ and 778.23 cm⁻¹ — bands corresponding to carbonate compounds and quartz phase, 649.60, 529.69, and 465.88 cm⁻¹ — deformation vibrations of Si–O and Al–O bonds.

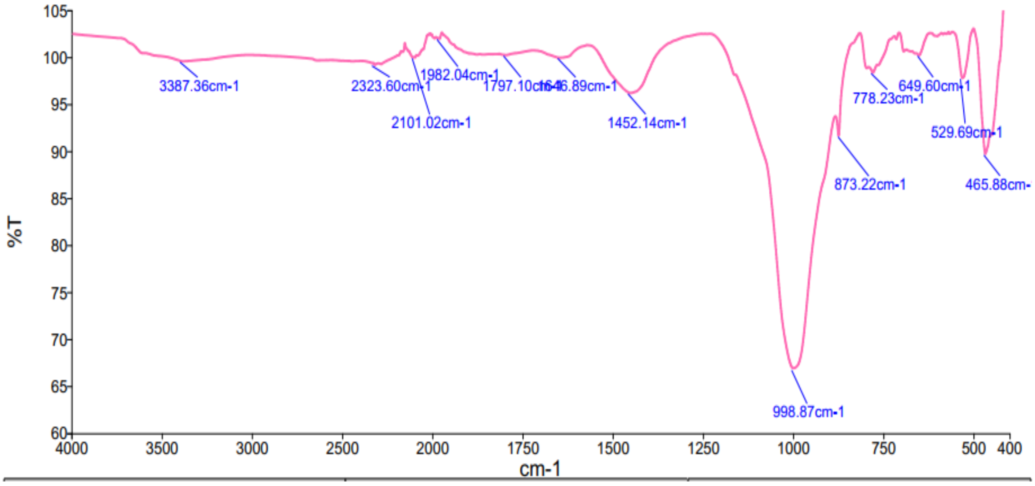


Figure 3. FTIR spectrum of the initial saline sandy loam soil.

The pronounced intensity of the band around 1000 cm⁻¹ indicates the dominance of aluminosilicate structures in the mineral matrix. The presence of bands at 873 and 778 cm⁻¹ confirms the occurrence of calcium carbonates, which are characteristic of saline soils in arid regions.

The broad absorption band in the region of 3400 cm⁻¹ confirms the presence of a significant amount of bound water, which correlates with the elevated electrolyte content identified by SEM–EDS analysis.

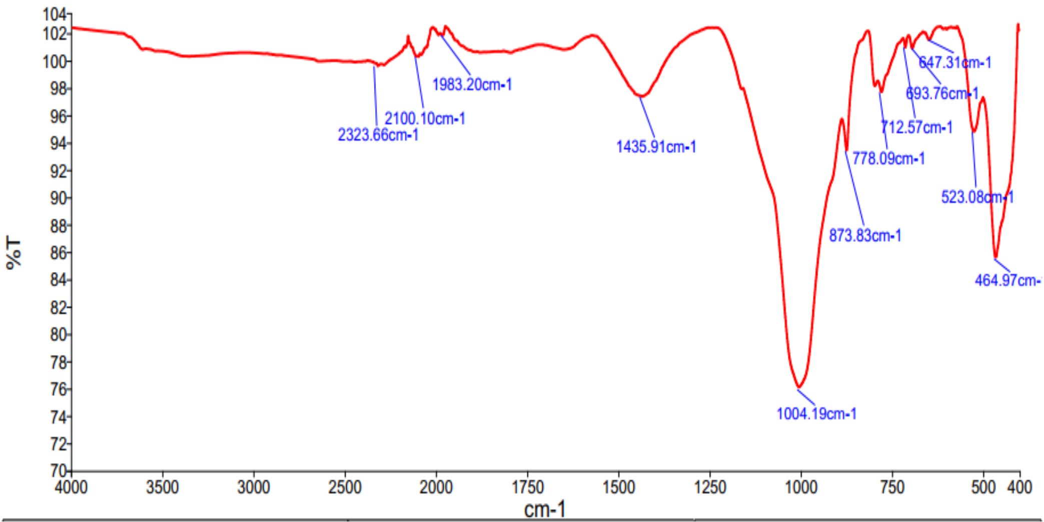


Figure 4. FTIR spectrum of the initial loamy soil (typical sierozem).

The FTIR spectrum of the sierozem soil is characterized by the following bands: ~3400 cm⁻¹ corresponding to O–H stretching vibrations; 1435.91 cm⁻¹ attributed to carbonate groups (CO_3^{2-}); 1004.19 cm⁻¹ representing an intense Si–O–Si stretching band, more pronounced than in the saline soil; 873.83 cm^{-1} and 778.09 cm^{-1} assigned to quartz and carbonate phases; 712.57 cm^{-1} and 693.76 cm^{-1} corresponding to carbonate-phase vibrations; and 523.08 and 464.97 cm^{-1} associated with deformation vibrations of the silicate lattice.

The intensity of the 1004 cm^{-1} band and the distinct carbonate signals indicate a more pronounced silicate–carbonate nature of the sierozem soil and a lower proportion of hydrated ions compared to the saline soil.

Comparative spectral analysis shows that both soils possess a well-developed aluminosilicate matrix. The saline soil exhibits a broader O–H band, indicating a higher degree of hydration. The sierozem soil demonstrates more distinct carbonate bands (1435–712 cm^{-1}). The intensity of the Si–O–Si bands is higher in the sierozem soil, which is consistent with its higher silicon content according to SEM–EDS data.

Figures 5 and 6 present benzene vapor adsorption isotherms for saline (sal.s.) and sierozem (g.s.) soils modified with sulfanol and CMC at dosages of 4, 6, and 8 ml. Based on the shape of the curves, the isotherms can be classified as type II–IV according to the BET classification, indicating a well-developed mesoporous structure. A distinct region of linear adsorption growth is observed at intermediate relative pressures, followed by redistribution of the adsorption volume.

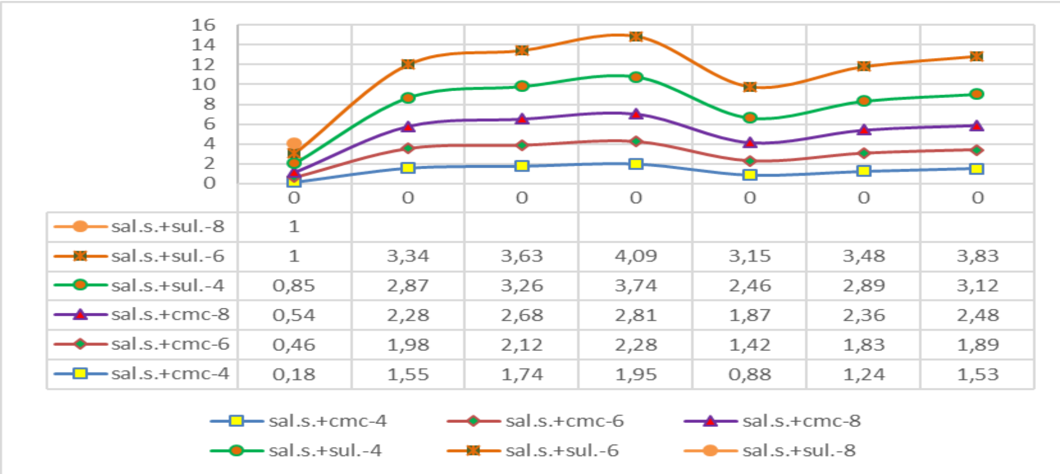
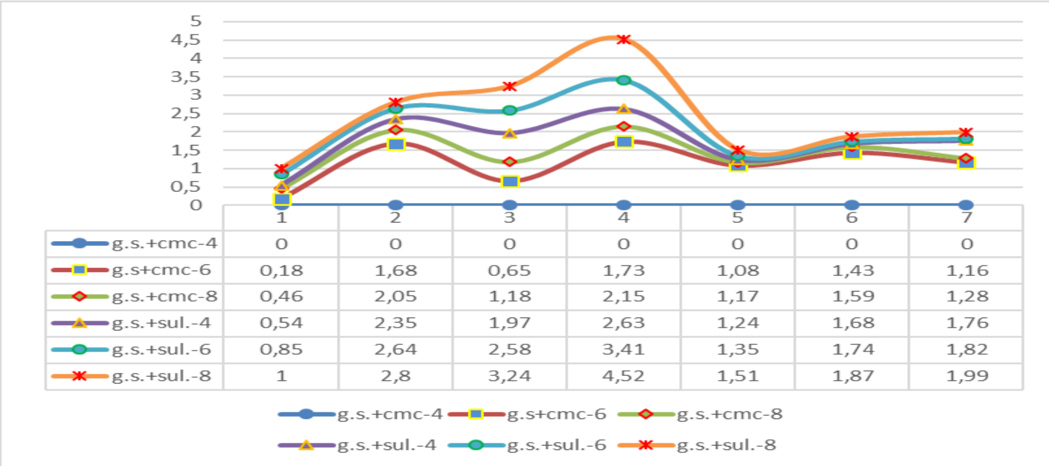


Figure 5. Benzene vapor adsorption isotherms of saline soil modified with organic structuring agents.

For the saline soil, significantly higher adsorption values are observed upon sulfanol addition, with a maximum at a dosage of 6 ml and a pronounced increase in adsorption in the intermediate P/P_0 region. Notably, the sample sal.s.+sul-6 exhibits the highest sorption capacity. At a dosage of 8 ml, a tendency toward stabilization or partial pore blocking is observed.

In saline soil, the presence of electrolytes reduces the thickness of the electrical double layer, facilitating adsorption of the anionic surfactant onto the mineral surface. Sulfanol forms an adsorption layer, partially hydrophobizes the surface, promotes redistribution of mesopores, and increases the accessibility of the internal surface to the non-polar adsorbate (benzene). When the optimal concentration (6 ml) is exceeded, partial micropore blocking, structural densification, and a reduced increment in adsorption volume may occur. In contrast, the increase in adsorption for CMC-modified samples is less pronounced. This behavior is associated with polymer-bridging between particles, enlargement of inter-aggregate voids, and partial shielding of the internal surface.

The sierozem soil demonstrates more moderate adsorption values. Its characteristic features include smoother isotherms, the absence of a sharp increase in sorption capacity, and a maximum also observed at 6 ml of sulfanol. These results indicate a lower proportion of fine-dispersed fractions, less developed microporosity, and the predominance of macropores in the sierozem soil.



	1	2	3	4	5	6	7
g.s.+cmc-4	0	0	0	0	0	0	0
g.s+cmc-6	0,18	1,68	0,65	1,73	1,08	1,43	1,16
g.s.+cmc-8	0,46	2,05	1,18	2,15	1,17	1,59	1,28
g.s.+sul.-4	0,54	2,35	1,97	2,63	1,24	1,68	1,76
g.s.+sul.-6	0,85	2,64	2,58	3,41	1,35	1,74	1,82
g.s.+sul.-8	1	2,8	3,24	4,52	1,51	1,87	1,99

Figure 6. Benzene vapor adsorption isotherms of sierozem soil modified with organic structuring agents.

A comparative analysis of the transformation mechanisms indicates that sulfanol enhances benzene adsorption, increases the effective internal surface area, and promotes mesopore redistribution, with a more pronounced effect observed in the saline soil. In contrast, CMC acts primarily through aggregate formation, increasing interparticle distances and exerting a weaker influence on the internal surface; its effect is more evident in the sierozem soil.

The integrated analysis of SEM–EDS, FTIR spectroscopy, and benzene vapor adsorption data allows formulation of a generalized model of structural transformation of soils modified with organic structuring agents of different molecular nature (Figure 7). According to SEM–EDS and FTIR results, both soils possess an aluminosilicate matrix with a hydroxylated surface containing active sites (–OH, Si–O–Si, Al–OH).

In the saline soil, elevated concentrations of Na^+, Ca^{2+}, and Mg^{2+} create a high ionic-strength environment, leading to compression of the electrical double layer, reduced electrostatic repulsion between particles, and enhanced coagulation of the dispersed phase. In contrast, in the sierozem soil, where the silicate fraction predominates, the structure is governed mainly by mechanical packing of particles with less pronounced electrolyte effects.

The pore space of both soils exhibits a predominantly mesoporous character (type II–IV isotherms); however, pore-size distribution is largely determined by granulometric composition.

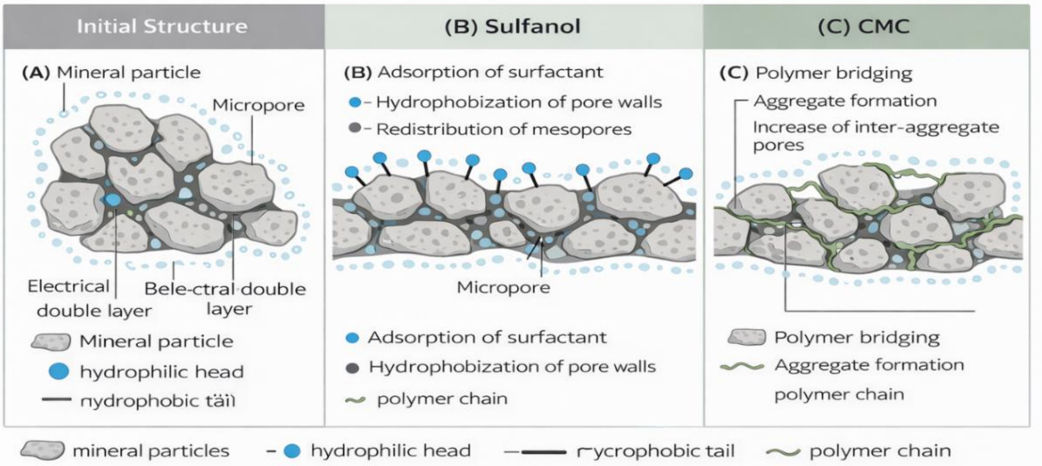


Figure 7. Colloid-chemical model of structural transformation of soils under the action of organic structuring agents.

Sulfanol, as an anionic surfactant, induces structural transformation through several sequential stages. Surfactant molecules adsorb onto positively charged sites and cationic centers (Ca^{2+}, Mg^{2+}), a process particularly pronounced in saline soil. The hydrophilic sulfonate group anchors to the mineral surface, while the hydrophobic hydrocarbon tail is oriented toward the pore space. This results in partial hydrophobization of pore walls, leading to modifications in interparticle interactions, reduction of interfacial surface energy, and redistribution of mesopores. At the optimal dosage (6 ml), the maximum increase in benzene adsorption is observed (up to 4.09 arbitrary units for the saline soil), indicating an increase in accessible internal surface area. When the dosage is increased to 8 mL, an excessive adsorption layer forms, partially blocking micropores and reducing the increment in sorption capacity. Thus, sulfanol primarily acts through a surface-energy-driven mechanism of structural transformation.

Carboxymethyl cellulose operates through a different mechanism. The carboxyl and hydroxyl groups of CMC form hydrogen bonds with surface –OH groups of mineral particles. Polymer macromolecules connect individual particles, forming a spatial network. As a result, inter-aggregate voids increase, macro- and mesoporosity are modified, while internal microporosity changes only slightly. The effect on benzene adsorption is moderate, since CMC has a weaker influence on the hydrophobicity of the internal surface. Therefore, CMC induces transformation predominantly through a structural–aggregation mechanism.

Overall, structural transformation of soils under modification with organic structuring agents proceeds along two principal pathways: surface-energy-driven pore redistribution (surfactant mechanism) and polymer-induced aggregate formation (CMC mechanism). The optimal structuring-agent concentration represents a balance between surface modification and preservation of pore-space permeability.

Conclusion

As a result of the comprehensive investigation, the mechanisms of structural transformation of loamy (typical sierozem) and sandy loam saline soils from the Kegeyli and Kungrad districts of Karakalpakstan under modification with organic structuring agents—sulfanol and carboxymethyl cellulose—were established. SEM–EDS analysis demonstrated that the saline soil is characterized by elevated contents of carbon (24.78 wt.%), oxygen (46.23 wt.%), and calcium (4.41 wt.%), as well as the presence of sodium, indicating high ionic saturation and compression of the electrical double layer. In contrast, the sierozem soil exhibits higher silicon (19.09 wt.%) and calcium (7.01 wt.%) contents and a lower carbon fraction (10.68 wt.%), reflecting the predominance of a silicate–carbonate matrix and a lower degree of salinity. FTIR spectroscopy revealed intense absorption bands at 3387–3400 cm^{-1} (–OH stretching), ~1452–1435 cm^{-1} (carbonate groups), and 998–1004 cm^{-1} (Si–O–Si vibrations), confirming the presence of active adsorption sites and a hydroxylated surface capable of intermolecular interactions with organic additives. Benzene vapor adsorption isotherms correspond to type II–IV behavior, indicating a mesoporous structure of the investigated soils. The maximum adsorption capacity was achieved at a dosage of 6 ml of structuring agent per 50 g of soil. For saline soil modified with sulfanol, adsorption reached 4.09 arbitrary units, while for sierozem soil it reached 4.52 arbitrary units. Increasing the dosage to 8 ml resulted in a reduced increment in adsorption, indicating partial micropore blocking and structural densification. It was established that sulfanol induces transformation primarily through a surface-energy mechanism involving adsorption on mineral surfaces, hydrophobization of pore walls, and redistribution of mesoporosity. In contrast, CMC operates through a structural–aggregation mechanism, forming polymer bridges between particles and increasing inter-aggregate voids.

Thus, it has been demonstrated that structural transformation of soils under modification with organic structuring agents is governed by different mechanisms depending on mineral composition and degree of salinity. The proposed colloid-chemical model explains the observed redistribution of pore space and may serve as a theoretical basis for developing technologies aimed at regulating the sorption and water-physical properties of soils in arid regions.

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