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Glyoxal and its derivatives for stabilization of aluminium silicate colloid-dispersed systems

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Abstract. Preliminary studies have shown the possibility of using organic-mineral additive based on glyoxal and saponite containing waste for injection strengthening of soils. Modification of the sandy soil with the organic-mineral additive increases its specific cohesion up to 50 times. However, the resulting periodic colloidal structure (PCS) is not resistant to external temperature and humidity effects. The research aim was to evaluate the possibility of obtaining glyoxal derivatives by its chemical interaction with bark lignin substances without their preliminary extraction and test these structures for stabilization of the aluminium silicate colloid-dispersed systems. To activate surface centers of lignin, the bark was ground to the average particle size of 3.2 μm. Identification of IR spectra showed that the characteristic bands for lignin-glyoxal and bark-glyoxal compositions coincide practically and differ significantly from the spectrum of lignin in the range of 500-1500 cm^{-1}. It can confirm the formation of interaction products of bark lignin substances with glyoxal. Before the effect of moisture compressive strength of sandy soil samples with an organic-mineral additive based on glyoxal (reference sample) and its derivatives (test sample) is almost the same and at an average of 6 MPa. However, after moisture saturation the reference sample strength is reduced by 50 times, and the test sample strength is decreased by only 4 times. It confirms the formation of a stable PCS.

1. Introduction

Aluminium silicate colloid-dispersed systems are sand-clay soils, which are a mechanical mixture of abundant minerals of the earth's crust – aluminium silicates and characterized by high variability of properties under the influence of natural and climatic and technogenic factors [1]. Therefore, one of crucial tasks is the stabilization of sand-clay soils by creating a structure with predetermined physical and mechanical characteristics.

Preliminary studies presented in the papers [2, 3] have shown the possibility of using organic-mineral additive based on glyoxal and saponite containing waste for injection strengthening of soils in groundwork base and utility systems. Glyoxal is a dialdehyde of ethane diacid exhibiting surface-active properties and capable of spontaneous polymerization. To ensure a uniform polycondensation of glyoxal throughout the volume of strengthened sandy soil, it is required to inject a sorbent – a saponite containing waste extracted from the circulating water suspension of the industrial ore dressing of the diamond deposit named after M.V. Lomonosov. Modification of the sandy soil by the tailored organic-mineral additive content increases its specific cohesion up to 50 times. This effect is connected with increasing dispersion interaction between the soil particles by synthesis of a sealing layer from the organic polymer. However, the resulting periodic colloidal structure (PCS) is not resistant to external temperature and humidity effects.

In addition, it is known that when organic compounds containing phenolic hydroxyl groups in their structure interact with glyoxal, a waterproof tetraphenol (1,1,2,2-tetra-hydroxyphenol ethane) structure forms [4-6]. Structural formula of tetraphenol is given in the Figure 1.

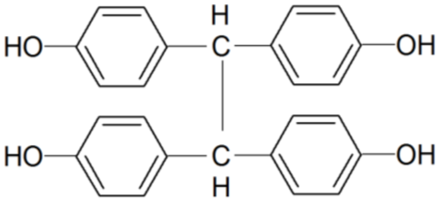


Figure 1. Structural formula of tetraphenol [6].

The most common vegetable polymer, characterized by a high content of phenolic hydroxyl groups (2-4.5 %), is lignin [7]. Lignin is incorporated firmly physically and chemically in the plant tissue structure, and its effective extraction by industrial methods is a complex engineering problem. However, in laboratory and research practice, the extraction methods of native lignin are based on the activation stage of the plant raw material by its mechanical grinding. It should be noted that more than 35 million m^3 of wood waste is being produced annually in the woodworking industry in Russia. One such waste is the bark. The content of lignin reaches 44 % in the bark [8]. The share of bark is at an average of 6-25 % of the volume of the tree trunk [9].

Therefore, the aim of the research presented in this paper was to evaluate the possibility of obtaining glyoxal derivatives by its chemical interaction with bark lignin substances without their preliminary extraction and test these structures for stabilization of the aluminium silicate colloid-dispersed systems.

2. Method

Pinus sylvestris bark was preliminarily made to its constant weight under the temperature of 40 °C. To activate surface centers of lignin, the bark was ground in the planetary ball mill Retsch PM100 using the dry mechanical dispersion method. Optimal dispersion parameters were selected experimentally to achieve finely dispersed state (grinding jar with volume 500 ml; 18 grinding balls from stainless steel alloy with diameter of 20 mm; sun wheel speed – 420 rpm; grinding time – 10 minutes).

The particle size of bark was determined by photon correlation spectroscopy using the Delsa Nano Series Zeta Potential and Submicron Particle Size Analyzers. The specific surface area was measured by adsorption method in the AUTOSORB-iQ-MP automatic analyzer.

To identify polymerization products in the bark-glyoxal system, infrared spectroscopy was used. The amount of glyoxal was taken on the basis of the hypothesis: one molecule of glyoxal binds two phenolic hydroxyl groups of lignin. IR spectra of bark, lignin (coniferous Bjorkman lignin) and bark-glyoxal and lignin-glyoxal compositions were recorded using Vertex 70 FTIR spectrometer (Bruker, Germany) and the attenuated total reflectance accessory with the GladiATR diamond crystal (Pike Tech., USA) (Arktika CUC at the NArFU named after M.V. Lomonosov). Conditions for recording spectra: range 4000-400 cm^{-1}, resolution 4 cm^{-1}, rapid scan – 128 spectra/sec. The background was atmospheric air. To identify IR spectra, special tables (atlases) compiled from the spectra of model samples with known structure were used [10].

To study the resistance to external effects of the aluminium silicate structure treated with a bark-glyoxal modifier, the compressive strength of sandy soil samples with an organic-mineral additive based on glyoxal (reference sample) and its derivatives (test sample) before and after moisture saturation was determined. The introduction of the organic-mineral additive into the sandy soil was carried out in two stages. Initially, the sand was mixed with ground bark and saponite containing material (SCM). Then an aqueous solution of glyoxal was added to this mixture. The quantitative ratio of components of the organic-mineral additive was taken on the basis of preliminary studies: 17 % of SCM, based on its specific water absorption, and 0.52 % of glyoxal of the sand fraction mass. The amount of bark was taken based on the content of phenolic hydroxyl groups. The components having been mixed thoroughly and compacted on a PLG-20 hydraulic press at a pressure of 1 MPa, samples were kept in natural conditions at a temperature of 25 ± 2 °C for a day until the glyoxal polycondensation completion. The parameter of the polymerization completion was to achieve constant weight of the soil sample. The diameter of the samples was 30 mm and the height was 18 mm. Part of the polymerized samples of sandy soil with organic-mineral additive based on glyoxal and its derivatives were kept in natural conditions (temperature of 25 ± 2 °C, humidity of 60 ± 2 %), part – in wet conditions in a desiccator. The dimensions, mass and moisture content of the samples were determined at different time intervals (1, 2, 5, 6, 7, 8, 9, 12 days) selected experimentally. The compressive strength was determined on AGS-5kNX SHIMADZU universal table-top tester after the samples had reached a constant weight. The set of experiments consisting of three parallel measurements having been completed, the average value of the maximum compressive stress was calculated.

3. Results

Bark particle size distribution is shown in Figure 2. The average particle size of the bark was 3.2 μm, the specific surface area was 1629 m^2/kg.

IR spectra of bark, lignin and bark-glyoxal and lignin-glyoxal compositions are given in the Figure 3 (a-d).

Table 1 contains strength characteristics of the sandy soil with organic-mineral additive based on glyoxal (reference sample) and its derivatives (test sample) before and after moisture saturation.

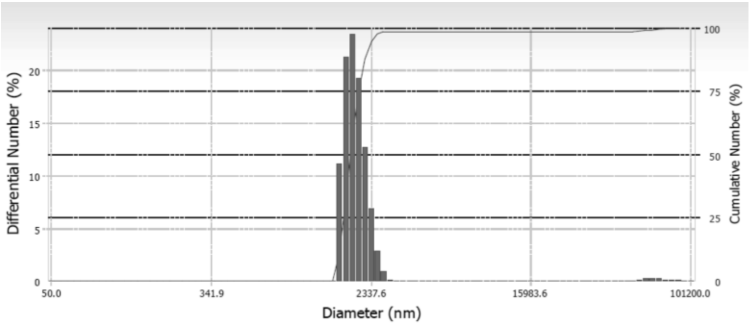
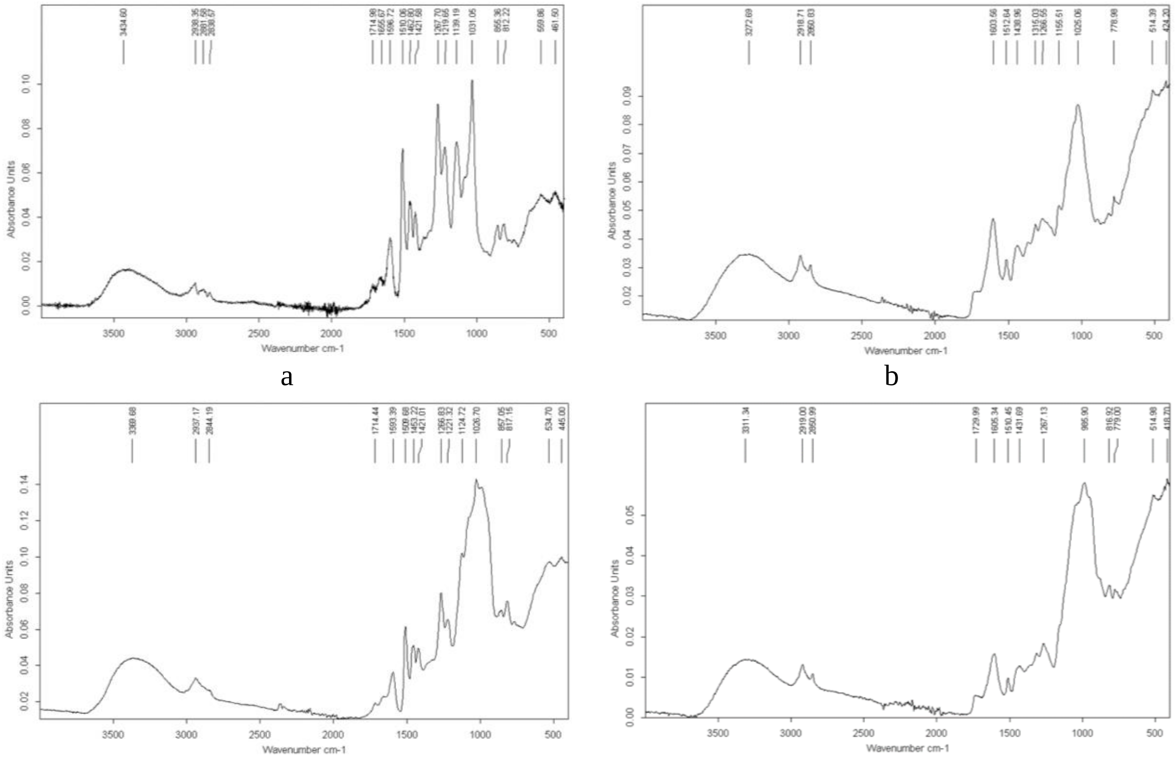


Figure 2. Bark particle size distribution.



a b

c d

Figure 3. IR spectra: a – lignin, b – bark, c – lignin with glyoxal, d – bark with glyoxal.

Table 1. Strength characteristics of the sandy soil.

Sample	Initial compressive strength, MPa	Compressive strength after moisture saturation, MPa
sand-bark	0.016	-[a]
sand-bark-glyoxal	0.026	-[b]
sand-SCM	2.21	1.04
sand-SCM-bark	2.47	0.91
sand-SCM-glyoxal	5.51	0.10
sand-SCM-bark-glyoxal	6.64	1.54

[a, b] The samples are destroyed spontaneously when exposed to moisture.

4. Discussion

Grain-size analysis of Pinus sylvestris bark using the Delsa Nano Series Zeta Potential and Submicron Particle Size Analyzers demonstrated that the investigated bark is a finely dispersed system (10^{-5}-10^{-7} m), on the particle surface of which lignin with active reaction groups is contained.

Identification of IR spectra showed that the characteristic bands for lignin-glyoxal and bark-glyoxal compositions coincide practically and differ significantly from the spectrum of lignin in the range of “fingerprints” (500-1500 cm^{-1}). It can confirm the formation of interaction products of bark lignin substances with glyoxal.

Before the effect of moisture compressive strength of sandy soil with an organic-mineral additive based on glyoxal (reference sample) and its derivatives (test sample) is almost the same and at an average of 6 MPa. However, after moisture saturation the strength of the reference sample is reduced by

50 times, and the strength of the test sample is decreased by only 4 times. It confirms the formation of a stable periodic colloidal structure.

Thus, it has been established that as a result of the chemical interaction of glyoxal with mechanically activated lignin substances of the bark, derivatives are formed that make it possible to create a moisture-resistant periodic colloidal structure based on aluminium silicate colloid-dispersed systems.

Acknowledgments

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