Laboratory study of properties of sand grouted by dual-component resins

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Abstract. Nowadays, technologies of chemical action on a rock mass, including the injection of polymer resins, the filling of voids by plugging with foaming compounds etc., are widely used to stabilize, to strength fractured rocks and to waterproof underground mines during development of solid minerals. In the paper we consider the issues of enhancing of the physical and mechanical properties of loose sedimentary rocks by chemical grouting with dual-component polymer resins. We perform laboratory experiments to strength fine-grained siliceous sand by the dual-component silicate-isocyanate and the foamed polyurethane resins. The grouting is performed through 10 cm diameter columns, which are filled with fine-grained siliceous sand. The main particle size fraction of the used sand is 0,150 – 0,210mm. The resin pumping into the test samples is carried out by two methods. The first method consists in sequential pumping of separate components of the polymer compounds. In this case, the solidification of the polymer compositions begins inside the rock sample. The second method consists in pumping of the fully prepared polymer compounds (two components are mixed immediately before injection in the rock sample). In this case, the solidification of the polymer compositions begins before the pumping in the rock, which leads to a rapid increase of the viscosity of the injected compositions. For both methods, the pumping is performed both with a pulse pressure increase and with a gradual (stepwise) pressure increase in the range from 0 to 15 bar. Based on the experimental results, the volume of the strengthened sand is determined. It was obtained that the volume of the strengthened (consolidated) sand is on average 2-3.5 times greater with separate-component injection, than with the injection of fully prepared compositions. For both pumping methods, a larger volume of consolidated rock is provided with a gradual (stepwise) pressure increase. Cylindrical specimens are cut out of the consolidated sand, and deformation-strength and filtration tests of the obtained material are carried out. The results show that the sequential pumping of separate components of the dual-component polymer compounds provides the greatest rock strengthening. It is obtained that the hardened sand uniaxial compressive strength is on average 1,5 times more for the sequential pumping of separate components compared to the injection of the fully prepared compositions. Similarly, the deformation modulus of hardened sand is 2,5 - 6 times more for the sequential pumping of separate components compared to the injection of the fully prepared compositions. The use of the dual-component silicate-isocyanate compositions provides more effective strengthening of loose sedimentary rocks compared to the foaming polyurethane compositions. The uniaxial compressive strength and the deformation modulus of the consolidated fine-grained sand are on average 10 - 12 times more for the dual-component silicate-isocyanate compositions compared to the foaming polyurethane compositions. The obtained data can be used to select and optimize the method of pumping of dual-component polymer compositions to stabilize disturbed mines.
1. Introduction
Currently, technologies that are used to stabilize rocks include strengthening of the rock mass by injection of polymer compositions. Polymer foams and resins are also used for waterproofing of mines, strengthening of fractured rocks in watered conditions, etc. The technology of chemical impact on the rock uses polymer and polymer-cement compositions, including resins, foams, gels, materials to apply coating layers on the mine surface, etc. [1-5]. The main advantages and disadvantages of the typical compositions are considered in works [6-8]. Typically, the chemical strengthening methods are based on the injection of either one-component or dual-component compositions. In the first case, the composition is usually cured, when it interacts with the formation water. In the second case, two components are mixed immediately before injection in the rock mass, where they cure, interacting with each other and with the formation water. The penetration of the composition depends on the curing time, on the viscosity and adhesion during the polymerization process. These properties limit the impact zone (rock-composition), especially in low-permeability rocks and in rocks with a significant surface microstructural defects, etc. In this study, to increase the time of impregnation and the penetration of the composition into the rock, it is proposed to use dual-component compositions with separate-component injection. The separate-component injection consists of the sequential injection of different components of polymer compositions. Initially the one component is pumped into the rock, then the second component is pumped. And their interaction with each other occurs in the rock [9-11]. The paper provides the results of the experimental study of strengthening of fine-grained sand with dual-component original silicate-isocyanate and polyurethane compositions under the conditions of the injection of fully prepared compositions and sequential pumping of separate components. The results of a study of permeability of sand, consolidated with a foaming polyurethane and silicate-isocyanate composition under all-round compression are presented.

2. Materials and Methods

2.1. Materials
Experiments were carried out with fine-grained siliceous sand, which was pretreated. Pretreatment included washing, drying in a laboratory drying oven at 105-110 °C to constant weight, and determining the particle size distribution. After drying sand samples were weighed thrice, while an error of no more than 0.01 g was considered acceptable. Then, the particle size distribution was determined. The main fraction (particle size 0.150-0.210 mm) was more than 60 wt. %.

For the chemical strengthening of the sand, two dual-component polymer compositions were used. The first one is a silicate-isocyanate composition intended for filling of voids in a rock mass. It is prepared by mixing components A and B in a volumetric ratio of 1:1 immediately before pumping in the rock. The second one is a foaming polyurethane composition designed to strengthen unstable and fractured rocks in watered conditions. It is also prepared by mixing components A and B in a volumetric ratio of 1:1 immediately before pumping in the rock. Both polymer compositions were developed and tested at the N.A. Chinakal Institute of Mining of the Siberian Branch of the Russian Academy of Sciences. The technical properties of the compositions are shown in table 1.

Table 1. Properties of dual-component silicate-isocyanate and polyurethane compositions.

| Viscosity at 25°C (mPa) | Density at 25°C (g/cm³) | Foaming factor | Time of the start of the reaction/complete curing (s) | Temperature (max) of reaction (°C) | Uniaxial compressive strength of the cured composition (MPa) |
|------------------------|------------------------|----------------|-----------------------------------------------|----------------------------------|----------------------------------------------------------|
| Two-component silicate-isocyanate composition | | | | | |
2.2. Experimental procedure

A test sample of fine-grained sand was placed in a disposable removable cylindrical sealed chamber, a volume of which was 1000 cm$^3$. The volume of sand test sample was 400 cm$^3$. The chamber has an outlet to connect injection pump, and withstands pressures up to 10 MPa. Then, a dual-component polyurethane composition was pumped into the test sample. The injection was carried out in two different methods. The first one is sequential injection of components A and B into the rock (separate-component injection). In this case, the composition curing starts inside the rock sample. The second one consists in the composition pumping immediately after mixing components A and B (the injection of fully prepared compositions). In this case, the composition curing starts outside the rock sample. The volume of each pumped component in the experiments was 100 cm$^3$. The injection was carried out with step and impulse pressure supply. In the first case, the polymer composition pumping in the rock sample was carried out with a step pressure increase from 0 to 0.5 MPa with a step of 0.02 MPa. The holding time at each step was 60 seconds. Impulse injection was carried out with a sharp pressure increase from 0 to 1.5 MPa. After the polymer composition was injected, a time interval of 24 hours was maintained. Then the sample was removed, the volume of the loose rock part was estimated, cylindrical cores were made from the consolidated sand. Strength and filtration tests of the consolidated sand were carried out. The volume of the consolidated part was determined as the difference between the initial sample volume and the volume of loose material remaining after the pumping of polymer compositions. Deformation and strength tests of the consolidated fine-grained sand were carried out according to regulatory documents for laboratory deformation and strength tests of rocks (uniaxial compression strength, volumetric compression and elastic modulus, etc.).

The permeability of the consolidated sand was determined by results of the filtration tests carried out under the hydrostatic stress state. To determine the coal permeability, experiments were carried out using a laboratory apparatus developed at the Chinakal Institute of Mining SB RAS [12]. The apparatus is designed to measure the rock gas permeability for stationary linear flow of gas. It consists of a test cell, gas preparation unit and air-hydraulic system for axial and lateral compression of cylindrical rock samples. The installation provides automated maintenance of the present pressure gradient and measurement of the permeation time of a fixed gas volume. The experimental procedure, description and main characteristics of the laboratory apparatus are given in [12]. The cores with a diameter and

| Component | Value (±) | Component | Value (±) | Prepared composition |
|-----------|----------|-----------|----------|----------------------|
| A         | 137±2    | B         | 131±5    |                      |
|           |          |           |          | 1210-240             |
|           |          |           |          | 20-23                |

Two-component polyurethane composition

| Component | Value (±) | Component | Value (±) | Prepared composition |
|-----------|----------|-----------|----------|----------------------|
| A         | 180±5    | B         | 230±20   |                      |
|           |          |           |          | 150/480-600          |
|           |          |           |          | 10-11                |
length of 3 cm were made and used for filtration tests. The coal permeability experiments were carried out using nitrogen, filtered along the axial direction, with a constant pressure difference $\Delta P$ and hydrostatic pressure $P$ in the specimen. The value $P$ was varied from 1 to 3 MPa with a step of 1 MPa. For each value $P$, a series of laboratory tests was carried out with $\Delta P$ values from 0.01 to 0.05 MPa with a step of 0.01 MPa. The coal gas permeability factor was calculated for stationary linear flow [13]. A similar experimental procedure was performed for sand strengthened with the silicate-isocyanate composition.

3. Results and Discussion

3.1. Experimental results

As a result of the separate-component injection of polyurethane and silicate-isocyanate compositions with a step pressure increase, the volume of the consolidated sand was 35-45% of the initial rock sample volume. With a separate-component and impulse pressure injection, the volume was 20-35%. As a result of the injection of fully-prepared silicate-isocyanate composition with a step pressure increase the volume of the consolidated sand was 10-15% of the initial rock sample volume. It was found that sequential separate-component injection of the dual-component compositions with a smooth step pressure increase and maintaining a time interval at each step provides better results. It was obtained, that that sequential separate-component injection of the dual-component compositions provides the more rock impregnation, which is on average 2-3.5 times greater than with the injection of fully prepared compositions.

Laboratory strength tests showed that the separate-component injection of the two-components compositions provide better strength properties of the consolidated fine-grained sand. A comparison of the experimental results shows that the uniaxial compression strength determined for sand, strengthened by polymer compositions with separate-component injection is on average by 1.5 times more than those determined for sand, strengthened with fully-prepared compositions. A comparison of the experimental results shows that the Young’s modulus determined for sand, strengthened by polymer compositions with separate-component injection is on by 2.5 – 6 times more than those determined for sand, strengthened with fully-prepared compositions (table 2). The physical and mechanical properties of the consolidated sand samples obtained by impulse and step pressure injection of the compositions are close. The use of dual-component silicate-isocyanate compositions provides a more effective strengthening of loose rock, compared to foaming polyurethane composition. A comparison of the experimental results shows that the uniaxial compression strength and Young’s modulus determined for sand, strengthened by silicate-isocyanate composition are in average 10-12 times more than those determined for sand, strengthened by the foaming polyurethane composition.

The dependence of the strengthened sand permeability on the confining pressure is determined by the results of the laboratory filtration tests. The filtration tests showed that the permeability of fine-grained sand consolidated by a polyurethane composition with separate-component injection decreases from 650 mD to 440 mD, or about 1.5 times as the confining pressure increases by 3 times (from 1 to 3 MPa). The permeability of fine-grained sand, consolidated by the silicate-isocyanate composition with separate-component injection decreases from 586 mD to 287 mD, or about 2 times as the confining pressure increases by 3 times (from 1 to 3 MPa) (Figure 1). Significant differences in the strength and filtration properties of consolidated sand are associated with the difference in the physical and mechanical properties, structure of the cured polymers, a variation of rheological properties of the compositions during curing, the foaming factor et al. These factors determine the distribution of compositions in the volume of loose rock during injection.
Table 2. Properties of fine-grained sand strengthened by dual-component polymer compositions.

| Sample | Method of injection of two-component composition into rock sample | Injection pressure (MPa) | Uniaxial compressive strength (MPa) | Elastic modulus (MPa) |
|--------|---------------------------------------------------------------|--------------------------|------------------------------------|-----------------------|
| 1.1    | Separate-component injection                                 | 0 – 0,5                  | 12                                 | 883,2                 |
| 1.2    | Separated-component injection                                 | 0 – 0,5                  | 7,6                                | 953,9                 |
| 1.3    | Injection of fully-prepared composition                      | 0 – 0,5                  | 16,9                               | 1450,5                |
| 2.1    | Injection of fully-prepared composition                      | 0 – 0,5                  | 9,8                                | 214,7                 |
| 2.2    | Injection of fully-prepared composition                      | 0 – 0,5                  | 7,7                                | 157,8                 |
| 2.3    | Injection of fully-prepared composition                      | 0 – 0,5                  | 7,5                                | 153                   |

Figure 1. Dependence of gas permeability coefficient ($k$) of the fine-grained sand strengthened by two-component polymer compositions on the hydrostatic confining pressure ($P$): black circles correspond to the experimental data, obtained for rock samples, strengthened by silicate-isocyanate composition; circles correspond to the experimental data, obtained for rock samples, strengthened by...
polyurethane composition; black and gray curves are exponential approximations for experimental data respectively.

3.2. Application and practical significance
Separate-component injection of dual-component silicate-isocyanate and the foamed polyurethane resins into the soil is carried out through a cased hole with an open bottom or its perforated interval, isolated by a mechanical seal (packer). The packer is installed on the pipe through which the composition is supplied to the isolated zone. Wellhead fittings contain two inlets equipped with valves. Through one of the inlets, the injected composition is supplied, on the other - water or compressed air. Each component is pumped into the soil in several parts with intermediate blowing with compressed air or nitrogen between them. After the injection of the silicate component of the silicate-isocyanate composition, the equipment is flushed with water. The volume of the injected composition is taken equal to 0.3-0.5 to the volume of the voids of the soil to be strengthened in the impact zone. Blowing with compressed air is used to distribute the composition in the form of a layer over the surface of the voids without filling them completely. This provides a reduction in polymer consumption per unit volume of the grouted soil compared to the conventional mixing technology of the components before feeding into the soil without compromising the loading capacity of the grouted soil.

4. Conclusions
The strengthening of loose sedimentary rocks with dual-component polymer compositions, including the separate-component injection and the injection of fully-prepared compositions, was experimentally studied. The methods were tested on samples of fine-grained sand (the main fraction is a particle size of 0.150-0.210 mm over 60 wt.%) and dual-component silicate-isocyanate and foaming polyurethane resins. It was found that sequential separate-component injection of the dual-component compositions with a smooth step pressure increase and maintaining a time interval at each step provides better results. It provides a more extensive impregnation zone and high deformation and strength characteristics of strengthened rocks. The volume of the consolidated sand is on average 2-3.5 times more than with the volume of the consolidated sand, obtained by the injection of fully-prepared compositions. Laboratory strength tests showed that the separate-component injection of the two-components compositions provide better strength properties of the consolidated fine-grained sand. A comparison of the experimental results shows that the uniaxial compression strength determined and the Young’s modulus for sand, strengthened by polymer compositions with separate-component injection are on average by 1.5 times and by 2.5 – 6 times more than those determined for sand, strengthened with fully-prepared compositions. The use of dual-component silicate-isocyanate compositions provides a more effective strengthening of loose rock, compared to foaming polyurethane composition. The permeability of fine-grained sand consolidated by a polyurethane composition with separate-component injection decreases from 650 mD to 440 mD, or about 1.5 times as the confining pressure increases by 3 times. The permeability of fine-grained sand consolidated by the silicate-isocyanate composition with separate-component injection decreases from 586 mD to 287 mD, or about 2 times as the confining pressure increases by 3 times. The obtained results can be used to select and optimize the injection method of dual-component polymer compositions to strengthen loose and stabilize fractured rocks, including the rocks in watered conditions.

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