Development of chemical compositions for impervious screens in rocks

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Abstract. The paper presents the method to create anti-seepage screens by hydraulic fracturing with three-component polyurethane mixture. The proposed working fluids and their pumping circuits allow creation of a fracture and an adjacent insulation layer. Gas permeability of porous medium is determined at limit consumption of reagents per insulating screen unit area.

Impervious screen employment as protection of underground working increases mining safety. Impervious screens can be used to seal embedded degassing boreholes [1, 2], to prevent accidental steam entry in mine oil production [3] and underground leaching of non-ferrous metals [4, 5]. Formed in rock mass, the barrier contains a hydraulic fracture and adjoining area with the artificially decreased permeability. It is possible under rock masses treatment with insulating composition which has low initial viscosity and easily permeates into existing interstices and cracks [6].

One-component polymeric compositions and sealants cannot solve most of the mining engineering problems due to high viscosity, weak ability to penetrate and high hydraulic resistance during injection. At the same time two-component fluids possess the necessary properties. They are based on polyurethane resins and acrylate jells with independent supply of low-viscosity components into an impervious screen by separate channels. The mixture of components and edge water causes chemical formation with flexible polymer [7].

Compositions existing on the market used for insulating works are usually based on two-component acrylate jells. The beneficial property of the jells is low viscosity commensurable to water viscosity and lack of organic solvent.

The polyurethane resin components ability to penetrate is weaker than the ability of acrylate jells. However, the weaker ability to penetrate is an advantage for the formation of impervious screens because it decreases leakage to rock masses without injection problems. Due to cheaper reagents polyurethane compositions are more economically beneficial than the acrylate ones.

Most of the polyurethane compositions used for water shutoff have a form of high-viscosity (up to 5–6 Pa·s) prepolymerms. They are obtained as a result of isocyanate and polyl component mixture before the work start. Catalyst composition is fixed and baking time of resins is not regulated. These compositions are not suitable for the formation of impervious screens due to viscous prepolymer is insufficiently capable to penetrate and completely fill hydraulic fracture.

There have been developed an insulating composition containing three working fluids, one of which is hydraulic fracturing fluid, two others are polyurethane resin components. The components
are low-viscosity prepolymer with isocyanate redundancy (further, prepolymer) and hydroxyl plasticizer with catalyst (further, plasticizer) [8].

During the works performed in an isolated area of rock masses near a hydraulic fracture, it is necessary to follow the described order of injection:

1) At the first step the working fluid is supplied into fracture interval until a crack of a required size is formed in rock. Then, it takes some time till the pressure in the closed fracture interval declines to the formation pressure. The crack surfaces are closed, and the injected fluid permeates the rock mass;

2) At the second step a part of plasticizer (20–30% from the planned volume of prepolymer) is injected into the hydraulic fracturing crack, and it forms a layer saturated with this composition;

3) At the third step the prepolymer and the rest of the plasticizer (volume ratio 1:1) are supplied into the crack by two separate high-pressure hoses. They mix in the fracture interval and penetrate in enclosing rocks through the layer previously saturated with plasticizer. Contained in the layer, diol is reactive with free isocyanate, which leads to the increase of the prepolymer viscosity.

To estimate capabilities of the developed insulating composition and its influence on porous medium permeability, laboratory tests have been conducted.

The following compositions of samples have been used to model the porous medium: abrasive material—white corundum (classification group 24A), medium-soft (classification group SM1 by GOST 19202–80), medium structure (classification group 7 by volume content of the abrasive material), 25–315 micron grain (classification group 25 by GOST 3647–80) with ceramic bond (classification group $K$ by GOST 2424–83). From the abrasive wheel, we have drilled and cut cylindrical samples 30 mm in diameter and height. The porous medium model is made of two samples end-conjoined and placed into rubber cover. The insulating composition fluids have been put on the contact surface between the samples with specific charge 0.14–0.84 ml/cm².

![Figure 1. Scheme of bench to study insulating composition influence on porous medium permeability: GT—compressed gas tank; PG1 and PG2—pressure gauges; V1–V3—valves; PR1 and PR2—pressure reducers; GM—gasometer; 1–4—inputs and outputs for the connecting hoses to plumb in; 5—porous samples; 6—rubber cover; 7—test cell body; 8—loading rod; 9—basement.](image)

The study of insulating composition influence on porous medium permeability has been carried out in accordance with the following technique:
1) Setting the prepared model of porous medium in the test cell;
2) Compression the model along its axis on the press with power 2 kN;
3) Opening of the valves V1–2 (Figure 1) and reducer PR1, using pressure gauge PG1 2.5 MPa, lateral compression of the model;
4) The required nitrogen drop of pressure $\Delta P$ between the test cell outputs 1 and 3 is set by reducer PR2 using pressure gauge PG2;
5) Water in amount of 3 dm$^3$ is poured into gasometer GM through funnel;
6) Opening of the valve V3 and observation of water being displaced with gas from gas meter into the funnel;
7) When water reaches “2.5 l” gasometer GM scale mark timer is turned on;
8) When water reaches “1.0 l” scale mark turn off the timer, close the valve V3 and stop the experiment;
9) According to the timer readings filtration time is determined, and in accordance with thermometer and barometer temperature $T$ and air pressure in the laboratory are determined.

Figure 2 shows schemes of porous medium treatment with different working fluids employed in the experiments. We have studied the models saturated with water (Figure 2a), hydraulic oil HLP–68 with 175 mPa∙s viscosity in 24.8°C (Figure 2b), composition of prepolymer and plasticizer with 2.4 Pa∙s (Figure 2c) and insulating composition with catalyst concentration in plasticizer is equal to 0.6% (Figure 2d).

![Figure 2. Scheme of studied sample treatment with the following compositions: a—water, b—oil HLP-68, c—composition of prepolymer and plasticizer, d—fracture fluid with insulating composition.](image)

The value of gas permeability coefficient $K_g$ is calculated from the formula for gas linear flow and static filtration [9]

$$K_g = \frac{2 \times 10^4 \cdot V \cdot \mu_a \cdot P_3 \cdot L}{t_{av} \cdot S \cdot \left( \frac{1}{P_1^2} - \frac{1}{P_3^2} \right)},$$

where $K_g$—gas permeability coefficient, $10^{-3}$ mcm$^2$ (mD); $P_1 = P_3 + \Delta P$—pressure at the test cell input 1 (Figure 1), $10^{-1}$ MPa (bar); $P_3$—pressure at the test cell output 3 (atmospheric), $10^{-1}$ MPa (bar); $V$—gas volume with pressure $P_3$, gone through the model cm$^3$; $\mu_a$—nitrogen viscosity, mPa∙s (sP); $S$—sectional area of the model, cm$^2$; $L$—model length, cm; $t_{av}$—filtration time, averaged by several dimensions, s.
The experiments are carried out at $T = 24.8^\circ$C and nitrogen viscosity of $0.01778$ mPa·s [9]. In the course of the experiments it is found that insulating properties of the composition with 0.6% catalyst concentration in plasticizer stabilize in 20–25 min after polymerization is started. It conforms with the cure time of the same composition under room temperature.

Figure 3a shows a graph of porous medium model gas permeability coefficient (treatment scheme in Figure 2d) depending on nitrogen pressure drop $\Delta P$ after the insulating composition cure in porous sample. The time after the composition is made is $t_{pm} \geq 20$ min. Figure 3b shows $K_g$ as function of drop of averaged gas pressure in the model $P_{av} = P_3 + \Delta P/2$ implemented for extrapolation of measurement results on gas permeability in range of small values $\Delta P$ [10].

According to the graph in Figure 3a gas permeability $K_g$ has non-linear dependence on the pressure drop $\Delta P$. When the pressure drop is low gas permeability increases. In actual practice of degassing borehole shielding, gradient of pressure between the mine working and methane extraction zone is about 100 Pa/cm, which corresponds to $6 \times 10^{-4}$ MPa pressure drop for 6 cm-long model.

The linear trend of $K_g(1/P_{av})$ dependence shown in Figure 3b is described by the expression $K_g = a/P_{av} - b$, where $a = 362.963$ mD·bar, $b = 89.934$ mD. Determination coefficient is $r^2 = 0.98$. Using the formulated expression we obtain the following value $K_g = 237$ mD ($10^{-3}$ micron²) for $\Delta P = 0.25 \times 10^{-1}$ MPa (bar) and $t_{pm} \geq 20$ min. The obtained value is significantly lower than the measured one in case of incomplete cure of insulating composition ($t_{pm} = 5–15$ min. For $\Delta P = 0.006 \times 10^{-1}$ MPa we have $K_g = 278$ mD ($10^{-3}$ micron²).

![Figure 3. Dependence of gas permeability coefficient $K_g$ on: (a) nitrogen pressure drop $\Delta P = P_1 - P_3$; (b) reciprocal to averaged nitrogen pressure in model when $t_{pm} \geq 20$ min.](image)

Comparing gas permeability coefficient for different treatment schemes (Figures 2a–2d) when $\Delta P = 0.25 \times 10^{-1}$ MPa and $\Delta P = 0.5 \times 10^{-1}$ MPa we have determined that insulating composition with the specific charge of 0.28 ml/cm² (without considering fracture fluid based on water) decreases porous medium permeability more than water by 7–9 times. It decreases the permeability of liquid with viscosity higher than 175 mPa·s and the same charge by 6–8 times, liquid of the same viscosity with 3 times bigger charge—by 5–6 times, and liquid with viscosity of 2.4 Pa·s—by 4 times.

To conclude with, the main role of insulating composition in an impervious screen is to decrease leakage of viscous fill fluid from the crack into enclosing rocks. The developed composition provides the decrease in porous medium permeability as well as in fill fluid leakage by several times in case of low charge per unit screen area.

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