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Special Cement Slurries for Strengthening Salt Rock Mass

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Abstract: Every year, the number of exploited mine workings necessary to seal the exploited mines increases in the world. As a result of experiments, technologies are developed that allow slurry to be pumped to fill free rock spaces or to liquidate rock mass discontinuities. The slurry preparation technologies can be divided into: subsurface and surface preparation and injection. Due to the pressure that forces the sealing slurry to move, the following can be distinguished: pressure technologies and technologies of gravity injection. The effectiveness of the work is determined by the correct selection of the technique and technology of the treatment and the selection of the optimal cement slurry recipe. The type of sealing liquid is especially important during works related to filling the exploited mine workings in salt mines. Therefore, this article presents the criteria for the selection of slurry recipes and their technological parameters, used for sealing and strengthening the salt rock mass. For this purpose, laboratory tests are carried out on various formulas of sealing slurries, prepared on the basis of full saturated brine and CEM I 32.5R Portland cement, ground granulated blast furnace slag, fly ash, and silt. The proposed concept for the selection of sealing slurry formulas has been positively verified during the performed works on sealing and strengthening the salt rock mass.

Keywords: cement; ground granulated blast furnace slag; fly ash; brine; cement slurry; salt rock mass; salt mine

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1. Introduction

As a result of the exploitation activities carried out in mines, and especially in salt mines, empty post-mining spaces are created, which pose direct or indirect threats such as:

- Specific physico-chemical and mechanical properties of the extracted mineral;
- Variety of rocks found in the salt rock mass (salt, shales, clayey, mudstones);
- The manner of arrears.

They often face various types of threats, the most common of which include:

- Water hazards;
- Heart attack risk;
- Uncontrolled movement of the rock massif.

Most publications [1–5] analyzing the possibilities of prevention as well as methods and technologies of repair related to hazards in salt mines emphasize that the most effective solutions to these problems include:

- Sealing and/or strengthening of the rock mass by borehole injection methods with the use of appropriately selected sealing slurries;
- Permanent insulation of the entire endangered area (e.g., by damming a leak) and filling free voids in the rock mass or mining excavation with a cement slurry using the pipeline injection method [6,7].

Depending on the chemical composition (sodium, calcium, magnesium, and potassium chlorides) and the mineralogical salt rock mass, waterproofing works should be performed with specially selected sealing slurries based on brine with full salt saturation, in which strengthening and sealing works are performed [8–10]. When developing various types of recipes and complex technological systems of cement slurries, it is necessary to select the individual ingredients of the cement slurries recipes in such a way as to eliminate the undesirable effect of brine as a batching liquid on the technological parameters of both fresh and hardened cement slurry [11–13].

Cement Slurries for Sealing the Salt Rock Mass

The salt rock mass significantly influences the physical and chemical properties of fresh and hardened cement slurry. Depending on the chemical composition of the salt rock mass (sodium, calcium, magnesium chlorides), sealing works are made of slurries prepared on the basis of brine with full salt saturation, in which the rock mass is sealed and strengthened.

The hardened cement slurry during operation as a sealing and strengthening medium of the salt rock mass is subjected to the action of a number of destructive factors (mainly through mineralized reservoir waters and brines with various degrees of saturation). If brine or solutions containing a mixture of different salts are used as the batching liquid, then the risk of the durability of the hardened cement slurry increases additionally. We can talk about extreme operating conditions of such sealing slurries. In such cases, we deal with all types of chemical corrosion (i.e., extractive corrosion, the formation of soluble salts through the exchange reaction, and the formation of secondary hydration products of increased volume).

Cement slurries made on the basis of brines are often called saline cement slurries in the literature [14–17]. For the first time, brine cement slurries were used in the United States (1940) when cementing casing pipes in a salt dome in the Gulf Coast region. Currently, they are routinely used for sealing and strengthening the salt rock mass. Long-term applications of brine sealing slurries are possible thanks to the constant technological progress with regard to new types of binders with increased durability [18–20]. Scientific and research works and engineering experiments focus on the modification of the physical and chemical properties of the hydraulic binders themselves as well as new generation additives and admixtures used to regulate the technological parameters of both fresh and hardened cement slurries [21–23]. Ensuring the effectiveness of sealing works in the salt rock is possible after taking into account the following criteria [24–26].

The first is the condition of compliance with the environment. The chemical composition of the brine should correspond to the rock mass to be sealed, both in terms of its chemical nature and the degree of solubility. The mixing liquid should therefore be fully saturated brine, at a temperature corresponding to the temperature of the environment of the sealed rock mass [27,28].

The second condition should be defined as the pumpability criterion. It is carried out through the appropriate selection of the rheological model and rheological parameters of the cement slurry. Correctly determined rheological parameters make it possible to calculate the flow resistance of the cement slurry in the circulation system from the injection aggregates to the place of its location. The knowledge of hydraulic resistance allows for a rational selection of techniques and technologies for filling voids in the rock mass [29–31].

Brine cement slurries can have different rheological properties. The flow curves may be reversible or may show hysteresis. This may be caused, among others, by the fact that, with short measurement times, the destruction of the cement slurry structure is dominant, while at longer times, its reconstruction. Thus, in fresh cement slurries there is an overlapping of the processes of structure destruction under the influence of shear in the viscometer and its reconstruction by the products of cement grain hydration. The actual rheological properties of fresh cement slurries are of interest to many researchers. It is related to the complex nature of the interaction of various physico-chemical factors, such as [4,14,16,27,32]:

- Complex binding processes of cement slurries;
- Chemical composition and mineralization of water;

- Variable cement slurry consistency as a function of time;
- The stability of the cement slurry;
- Technology of injection and extruding the slurry into a sealed rock mass;
- Slurry flow resistance in the circulation system of the cement slurry from the place of its preparation to the place of its placement in the rock mass.

Due to the grain size of the cement, fresh cement slurries can be regarded as dispersion systems as already indicated. The structure of such a system depends mainly on the mass quotient of water and dry cement as well as on the cement grain size and, consequently, on its specific surface. The physicochemical properties of the resulting cement slurry structure are also influenced by the forces acting between the cement grains and water molecules, which in turn are influenced by [33–35]:

- Surface charge;
- Concentration of ions in the slurry;
- Adsorption phenomena.

In cement slurry, due to the high reactivity of cement phases to water, the rheological properties of the sealing slurry, apart from the water-binder coefficient and hydraulic binder dispersion, depend mainly on the type and amount of hydration products (cement), while the nature of the surface of the clinker phases is of less importance. Another factor influencing the structure of fresh cement slurry is the mineral composition of the cement, which in turn influences the course of the hydration reaction. The third requirement is the need to ensure adequate mechanical strength of the hardened cement slurries in mining conditions. The formulas of the slurries should be selected so that the resulting hardened slurry has the same mechanical properties or comparable to those of a natural rock mass. By ensuring the stability and consolidation of the rock mass, the causes of additional displacements and deformations in the rock mass are eliminated. Another criterion that must be met is the need to ensure adequate durability of the hardened cement slurries. The hardened cement slurry during operation as a medium for sealing the salt rock mass is subjected to the action of a number of destructive factors, mainly highly mineralized reservoir waters and brine of varying degrees of saturation [36,37]. If, in addition, brine or solutions which are a mixture of different salts are used as the batching liquid, the risk of the stability of the hardened cement slurry increases additionally.

In this situation, various types of chemical corrosion may occur (i.e., extraction corrosion, formation of soluble salts through the exchange reaction and formation of secondary hydration products of increased volume) [38].

The durability of the hardened cement slurry is determined by a number of parameters, in particular its phase composition, texture, and the morphology of the hydration products. So what is necessary is:

- High durability and low solubility of hydration products;
- Low overall porosity and high tightness of the hardened cement slurry.

Another condition for the selection of cement slurries recipes is the economic and ecological factor. In order to minimize the costs related to the unit price of brine cement slurries, these types of slurries are sometimes cheap waste hydraulic-pozzolanic materials. The use of these types of additives and salty post-exploitation deposit waters significantly reduce costs and reduce the risk of degradation of the natural environment [35,38,39].

At the Faculties of Drilling, Oil and Gas, as well as at the AGH University of Science and Technology, as well as at the Oil and Gas Institute—National Research Institute in Krakow, for many years joint research has been carried out on obtaining and using new unconventional cement slurry for sealing and strengthening the salt rock mass. Research, both basic and applied, results in a number of solutions with patent and implementation values. Therefore, they are widely used in works related to the liquidation of physical discontinuities of the rock mass, especially in salt mines. The most important advantages of these include:

- Good adhesion to rocks with different lithological education (both to evaporative rocks as well as to swelling clays and clay shales);
- Possibility to adjust the technological parameters of both fresh and hardened cement slurries;
- Eliminating the dissolution of the salt rock mass by the cement slurry and its filtrate;
- Increased resistance to corrosive factors, especially to brines (reservoir waters) with strong mineralization;
- Adequate rheological parameters to the conditions of the sealed rock mass;
- Relatively low unit costs of their preparation.

2. Materials and Methods

2.1. Materials

For the preparation of brine cement slurries, CEM I 32.5R Portland cement and a ground granulated blast furnace slag are used as the hydraulic binder and as the basic binder of the brine slag-alkali slags, respectively. The working liquid in each case is mine brine with full saturation. In order to modify selected technological parameters of fresh and hardened cement slurries, optional hydraulic-pozzolanic additives such as

- Fly ash from hard coal combustion in conventional furnaces;
- Bentonite, calcium, sodium;
- Ground granulated blast furnace slag [40–42].

2.2. Methods

Laboratory tests of technological parameters of fresh and hardened cement slurries are carried out on the basis of the following standards:

- API Recommended Practice for Testing Oil-Well Cements and Cement Additives. API RP 10 B. April 1997,
- PN—EN 197—1: 2002, Cement. Part 1. Composition, requirements and compliance criteria for common cements,
- PN—EN ISO 10426—2. Oil and gas industry. Cements and materials for cementing holes. Lot 2: Tests for drilling cements. 2012.

The following parameters are tested:

- for fresh sealing slurries:
  - ✓ Density—with the use of a Baroid shoulder scale, flow—by means of a truncated cone (AzNII);
  - ✓ Conventional (relative) viscosity using a Ford cup No. 4;
  - ✓ Sedimantation (standstill)—using a measuring cylinder;
  - ✓ Filtration—using a baroid filter press;
  - ✓ Setting time—using the Vicat apparatus;
  - ✓ Rheological properties (plastic viscosity, apparent viscosity, flow limit)—using a rotary viscometer with coaxial cylinders, Ofite 900 type, with twelve rotational speeds (600, 300, 200, 100, 60, 30, 20, 10, 6, 3, 2.1 rpm, corresponding to shear rates: 1022.04; 511.02; 340.7; 170.4; 102.2; 51.1; 34.08; 17.04; 10.22; 5, 11; 3.41; 1.70 s-1);
- For hardened cement slurries:
  - ✓ Bending strength;
  - ✓ Compressive strength.

The selection of the optimal rheological model of sealing slurries consists in determining the rheological curve that allows the best description of the measurement results in the coordinate system: shear stress (τ)—shear rate (γ).

Using the regression analysis method, rheological parameters for individual models are determined. Then, with the help of performed statistical tests, the optimal rheological model is determined for a given cement slurry recipe. The best rheological model for each of the analyzed sealing slurries is the one with the highest correlation coefficient.

The following rheological models are analyzed:

- model Newton

$$\tau = \eta \cdot \left(-\frac{d\nu}{d\gamma}\right) \quad (1)$$

- model Bingham

$$\tau = \tau_y + \eta \cdot \left(-\frac{d\nu}{d\gamma}\right) \quad (2)$$

- model Ostwalda de Wael

$$\tau = k \cdot \left(-\frac{d\nu}{d\gamma}\right)^n \quad (3)$$

- model Casson

$$\sqrt{\tau} = \sqrt{\tau_y} + \sqrt{\eta} \cdot \sqrt{\left(-\frac{d\nu}{d\gamma}\right)} \quad (4)$$

- model Herschel—Bulkley

$$\tau = \tau_y + k \cdot \left(-\frac{d\nu}{d\gamma}\right)^n \quad (5)$$

where:
n—exponent,
k—coefficient of consistency $Pa \cdot s^n$,
τ_y—flow limit, Pa,
η—dynamic viscosity coefficient for Newton's model; plastic viscosity for the Bingham model, Casson plastic viscosity for the Casson model Pa·s,
$\frac{d\nu}{d\gamma}$—shear rate gradient–γ—s^{-1}.

In order to facilitate the calculations related to the determination of the optimal rheological models for the tested slurries, the "Rheo Solution" computer program was used. This program is owned by the Department of Drilling and Geoengineering at the Faculty of Drilling, Oil and Gas at AGH.

3. Results and Discussions

The observations presented below were formulated on the basis of many prepared formulas of cement slurries and many years of experience. It's found that the pozzolanic activity of the additives used allows to increase the strength of the hardened cement slurry as a result of changes in the microstructure, such as [11,33,43,44]:

- Change in the pore structure;
- Creation of a homogeneous, tight contact zone: slurry-rock (soil);
- Modification of the morphology of the basic product of hydration which is the C-S-H phase.

Changes in the phase composition and microstructure of the hardened cement slurry are observed after the introduction of pozzolans. They very effectively reduce the permeability and improve the resistance to corrosive agents and other factors determining the durability of such materials. The beneficial effects of pozzolans are as follows:

- There is a significant reduction in the rate of diffusion and liquid gases in the slurry (this applies to, for example, chloride ions);
- As a result of the decrease in $Ca(OH)_2$ content, the reactions of the formation of soluble calcium salts in contact with many corrosive environments are limited or eliminated;
- Due to the tight microstructure, carbonization is slower;

- As a result of the chemical imbalance of ettringite formation, the resistance to sulphate corrosion increases (this is the main, but not the only cause);
- Inhibition of liquid penetration, as well as the specific microstructure and chemical composition of the C-S-H product based on the additive eliminate the risk of destruction due to the alkali-rock (soil) reaction [12,33].

The addition of clay is intended to:

- Reduction of filtration;
- Elimination of downtime and increasing the stability of the cement slurry;
- Adjusting the strength parameters of the hardened cement slurries to the parameters of the sealed rock mass;
- Reduction of the permeability of hardened cement slurries.

The addition of sodium carbonate in brine cement slurries is to [15,26,40]:

- Accelerating the beginning of binding of the cement slurry and shortening its end of binding,
- Improvement of rheological properties (slurry liquefaction).

The direct action of Na_2CO_3 as a fluidizer consists in adsorbing on the surfaces of cement particles or the addition of a hydraulic-pozzolanic surface-active compound previously dissolved in the mixing water. Cement and pozzolana are thus surrounded by a double ionic layer. The top hydrophilic layer keeps a water film on the surface, bound directly to the surface by molecular forces. The main effect of the hydrophilic compound is the additional peptization of cement particle clusters and facilitating the dispersion of the particles themselves in the aqueous environment and their stabilization (i.e., increasing their resistance to coagulation processes in the first period of cement-water interaction). Air bubbles that get between the cement grains during the mixing of the cement slurry, due to the repulsion of these grains, are hydrated as a result of their adsorption of a surface-active substance, forming a double ionized layer.

In summary, it is stated that the effect of Na_2CO_3 as a fluidizer is to better disperse the grains of hydraulic material particles in the mixing water. This also affects the properties of the fresh and hardened cement slurry. Increasing the dispersion of cement grains reduces the sedimentation of the slurry, which in turn reduces the separation of water during the setting of the cement slurry. As a result, the hardened cement slurry reduces the number of pores in the film water [33,35].

Laboratory tests carried out for many years, and then their applications, allowed for the development of a number of saline recipes for sealing slurries. From among the many practically used recipes for sealing the salt rock, Table 1 presents only some that take into account various additives, especially the hydraulic-pozzolanic type. Selected technological parameters of fresh brine sealing slurries are presented in Table 2.

Table 1. Exemplary recipes of saline slurries.

Composition		No. 1	No. 2	No. 3	No. 4	No. 5	No. 6	No. 7	No. 8	No. 9	No. 10
Full saturated brine, m^3	Składniki receptury potrzebne do sporządzenia 1 m^3 solankowego zaczynu	0.636	0.713	0.786	0.834	0.558	0.639	0.713	0.495	0.507	0.531
Portland cement class CEM I 32,5 R, kg		1050	819.6	589.4	416.9	136.2	116.9	260.9	197.8	304.4	531.3
Ground granular blast furnace slag, kg		-	-	-	-	1192	974.4	608.7	-	-	-
Fly ash, kg		-	-	-	-	-	-	-	791.2	690.0	510.1
Bentonite clay, kg		57.26	42.76	31.44	50.03	-	-	-	-	20.3	21.3
Sodium carbonate, Na_2CO_3, kg		9.54	17.82	31.44	33.35	34.06	23.39	17.39	-	-	-
Water-binder coefficient, w/b		0.684	0.972	1.446	2.000	0.500	0.700	1.000	0.600	0.600	0.600
The density of the cement slurry, kg/m^3		1880	1735	1595	1501	2044	1894	1757	1582	1624	1700
Mass of dry ingredients needed to prepare 1 m^3 of cement slurry, kg		1117	880.18	652.31	500.28	1362.0	1115.0	887.0	989.0	1000.0	1100.0

Table 2. Technological parameters of saline sealing slurries established in lab conditions at temperature 20 (±2 °C) [293 K].

Parameter	Composition									
	No. 1	**No. 2**	**No. 3**	**No. 4**	**No. 5**	**No. 6**	**No. 7**	**No. 8**	**No. 9**	**No. 10**
Flow by cone AzNII, nm	90	260	260	>260	170	260	>260	>260	>260	220
Relative viscosity according to Ford cup No. 4, s	-	19.4	13.5	11.4	42.2	13.3	11.3	17.1	22.3	27.5
Free water, %	0.5	0.8	1.5	4.5	0.5	3.0	8.2	7.0	2.5	1.2
Setting time h:min	1:45	1:50	10:15	27:15	4:10	7:20	22:10	12:15	13:05	13:40
	11:40	12:25	33:25	78:00	8:20	27:30	35:20	28:30	25:15	21:05

The selection of the optimal rheological model of the tested cement slurries is based on the determination of the flow curve that allows the best description of the measurement results in the coordinate system: shear stress (τ)—shear rate (γ) [11,44–46].

Using the regression analysis method, rheological parameters are determined for individual models (Newton, Bingham, Casson, Ostwald de Waele, Herschel—Bulkley). Then, with the help of performed statistical tests, the optimal rheological model is determined for a given cement slurry recipe. The best rheological model for each of the analyzed cement slurries was the one with the highest correlation coefficient. In order to facilitate the calculations related to the determination of the optimal rheological models for the tested slurries, the "Rheo Solution" computer program was used. This program is owned by the Faculty of Drilling, Oil and Gas of the AGH University of Science and Technology St. Staszica in Kraków [27,47,48].

Table 3 shows the calculated rheological parameters of the tested cement slurries for all analyzed rheological models.

Table 3. Rheological parameters of brine cement slurries determined at 20 °C for various rheological models.

Rheological Model	Rheological Parameters	Composition									
		No. 1	**No. 2**	**No. 3**	**No. 4**	**No. 5**	**No. 6**	**No. 7**	**No. 8**	**No. 9**	**No. 10**
Newtonian model	Newton's dynamic viscosity, Pa·s	0.046	0.014	0.093	0.084	0.226	0.038	0.027	0.391	0.109	0.056
	Correlation coefficient, -	0.994	0.992	0.986	0.981	0.987	0.991	0.962	0.957	0.860	0.937
Bingham's model	Plastic viscosity, Pa·s	0.043	0.016	0.009	0.008	0.234	0.035	0.025	0.340	0.089	0.049
	Flow limit, Pa	1.508	0.217	0.071	0.065	7.153	1.545	2.066	11.325	12.924	5.555
	Correlation coefficient, -	0.996	0.988	0.985	0.987	0.998	0.998	0.988	0.989	0.947	0.993
Ostwald-de Waele's model	Coefficient of consistency, Pa·s^s	0.440	0.082	0.041	0.035	2.531	0.376	0.437	3.683	2.521	2.094
	Exponent, -	0.619	0.691	0.716	0.655	0.586	0.623	0.575	0.575	0.532	0.421
	Correlation coefficient, -	0.951	0.947	0.926	0.945	0.969	0.974	0.987	0.993	0.997	0.946
Casson's model	Casson's viscosity, Pa·s	0.036	0.012	0.008	0.007	0.176	0.028	0.018	0.255	0.065	0,032
	Flow limit, Pa	0.449	0.058	0.016	0.014	2.636	0.467	0.753	4.033	4.942	2.987
	Correlation coefficient, -	0.998	0.998	0.998	0.994	0.999	0.998	0.994	0.995	0.965	0.995
Herschel-Bulkley's model	Flow limit, Pa	1.40	0.215	0.087	0.077	5.280	0.827	0.932	3.122	3.379	4.309
	Coefficient of consistency, Pa·s^n	1.021	0.014	0.008	0.007	0.428	0.089	0.143	1.933	4.261	0.152
	Exponent, -	1.026	0.987	1.015	1.02	0.903	0.865	0.753	0.709	0.452	0.844
	Correlation coefficient, -	0.999	0.999	0.999	0.999	0.999	0.999	0.997	0.999	0.999	0.997
Apparent viscosity at 1022.04, s^{-1}, Pa·s		0.008	0.044	0.014	0.012	0.047	0.037	0.026	0.099	0.089	0.051

Table 4 presents the results of strength tests (bending and compression) of hardened cement slurries seasoned for 7 and 28 days.

Table 4. Mechanical compressive and bending strength of the hardened saline cement slurry.

Composition			**No. 1**	**No. 2**	**No. 3**	**No. 4**	**No. 5**	**No. 6**	**No. 7**	**No. 8**	**No. 9**	**No. 10**
Flexural strength, MPa	After the curing time [day]	7	3.15	2.85	0.15	0.00	2.21	1.12	2.34	1.03	1.87	2.05
		28	14.62	12.32	5.34	1.84	10.43	8.32	4.01	3.06	3.89	4.07
Compressive strength, MPa		7	3.25	3.84	1.53	0.000	2.94	1.45	0.94	0.89	0.28	0.23
		28	18.56	21.43	6.46	2.87	6.42	7.32	5.26	3.42	3.32	4.27

The developed recipes were used with great success in work related to the elimination of hazards in salt mines. In these technologies, methods of both borehole and pipeline injection were used.

Research on the structure and microstructure of hardened saline cement slurries in terms of phase composition, microporosity, and microstructure allow us to formulate the thesis that the type of hydraulic binder used and pozzolanic additives and their mineral composition, especially the content of calcium aluminates and other mineral additives, are of significant importance for shaping technological parameters of both fresh and hardened cement slurries [2,10,14,16,25,49,50]. Further research should concern the possibility of applying new additives (e.g., ash from lignite combustion, ash from combustion in fluidized bed boilers) and admixtures (e.g., new generation superplasticizers).

4. Conclusions

1. In order to strengthen and seal the salt rock, it is recommended to use slurries based on hydraulic binders (cement, ground granulated blast furnace slag), which are modified with the addition of clay and sodium carbonate. The working liquid should be a fully saturated brine.
2. The addition of clay to brine slurries significantly extends the setting time and reduces the strength parameters of the hardened cement slurry.
3. The addition of Na_2CO_3 causes:
    - Improving the rheological parameters of the cement slurry, increases the fluidity of the cement slurry and favors better filling of voids and caverns in salt mine workings;
    - Shortening the setting time of the cement slurry.
4. Among the rheological models used to describe the relationship between shear stress and shear rate, the Herschel-Bulkley models have the highest values of correlation coefficients.
5. Rectilinear models (Newton and Bingham) of the tested cement slurries should not be used for accurate calculations of the flow resistance that may occur in the process of sealing casing columns in boreholes.
6. The developed recipes for brine sealing slurries were applied with great effectiveness in works related to:
    - Strengthening and sealing of the salt rock mass;
    - Tight filling of liquidated workings in salt mines.

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