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Forecasting Strength Parameters of Hardened Geopolymer Slurries Applied to Seal Casing Columns in Boreholes

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Abstract: Ensuring effective sealing of casing columns in boreholes requires the use of the appropriate technology of cement slurry injection into the annular space and the use of a properly designed cement slurry recipe. Very often, when selecting the technological parameters of the cement slurry, special attention is paid to the technological parameters of the fresh cement slurry, but little attention is paid to the mechanical parameters of the cement sheath that is being formed (the cement slurry after setting). In order to improve the parameters of the hardened cement slurry in the annular space, the cement slurry of a new generation with increased durability (so-called geopolymers) is used. Slurries based on geopolymers are obtained by modifying slurries based on common-use cements with mineral additives with pozzolanic or hydraulic properties. Most often, these additives are fly ashes from the combustion of hard coal or ground granulated blast furnace slags. The article presents the results of testing the mechanical parameters of hardened cement slurries prepared on the basis of CEM V multi-component cement. It was found that the increase in the amount of silica fly ash in the slurry causes a delay in the strength growth rate; such slurries have lower values of early strength. The water–cement coefficient has the strongest influence on the mechanical parameters. The test results are also statistically developed, thanks to which it is possible to select the appropriate mathematical model, and this enables the prediction of mechanical parameters for slurries as a function of their hardening time. Such a mathematical solution can save some labor-intensive research, which, however, cannot be omitted in the final stage of slurry design.

Keywords: well cementing; borehole sealing; increasing the efficiency of oil recovery; improved borehole sealing; technical and technological challenges; cement slurry technological parameters; compressive strength; geopolymer

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1. Introduction

The procedure of sealing casing columns in boreholes is one of the most important stages in the drilling process, regardless of the depth, geological and hydrogeological conditions, and technical and technological conditions prevailing in the borehole. First of all, the slurry must effectively fill the annular space between the borehole wall and the casing column, and be resistant to various types of corrosive agents [1–4]. The durability of the cement shell is strongly affected by the conditions in which the process of binding and hardening the sealing slurries in the borehole environment takes place. The cement slurry located outside the casing pipe column must be set within a certain time and acquire appropriate mechanical parameters in order to obtain adequate hydraulic insulation throughout the life of the well. The most important factors affecting fresh and then hardened cement slurry include increased temperature, increased pressure, the aggressiveness of formation waters, as well as the presence of drilling mud. The formation waters that occur in the profile of the drilled borehole are highly mineralized. Depending on their chemical composition and the type and degree of concentration of individual ions, their impact on the technological parameters of fresh and hardened slurries is different. This applies in particular to the setting time and corrosion resistance [4–8]. Many field studies and experimental works, as well as theoretical considerations, are carried out on the problem of effective removal of the drilling mud from the annular space of the borehole during the cementation of the casing column. Despite this, it is stated that the problem of mud disposal is still present and not fully solved. This is mainly due to the complexity of the problem of cementing casing columns (deep holes, directional holes, horizontal holes, the use of smaller clearances, etc.) [9–12].

In addition, based on the analysis of the causes of unsuccessful cementing procedures, which were technically carried out correctly, it was found that the type and composition of the washing liquid and cement slurry affect the degree of sealing of the hole. The parameters of these liquids play a very important role in their interaction during flushing and displacing the mud from the hole during its cementation [13–15].

An improvement in the cleaning of the annular space can be achieved through the appropriate selection of the composition and properties of the washing and buffer liquids, as well as the use of appropriate pumping regimes and the time of contact of these liquids with the space based on specialized research. However, these issues cover a different thematic scope than the aspects covered in this work [16–18].

It is stated that the quality of the cement slurry used plays an essential role in the process of proper sealing of the borehole. The composition and parameters of the slurry depend primarily on the geological and hydrogeological conditions of the borehole in which the process of binding and formation of the hardened slurry takes place, the type of drilled rocks, the depth, the temperature, and the pressure in the borehole. Knowledge of the technological properties of both binders, admixtures, and additives is necessary for the correct selection of the type of cement slurry and the concentration of individual components included in the slurry recipe [19,20].

A novelty in this work is to determine whether or to what extent the change in the concentration of both fly ash and ground granulated blast furnace slag shapes the mechanical parameters of hardened geopolymer slurries.

1.1. Additives Modifying the Technological Properties of Fresh and Hardened Cement Slurries

In order to increase the effectiveness of sealing procedures of casing pipe columns in boreholes, intensive research has been carried out in recent years on the development of a new generation of special binders called geopolymers [3,5,21–26].

Cement slurries based on geopolymers are based solely on waste components of inorganic origin. They are obtained by modifying the composition of slurries prepared on the basis of common-use cements with additives with pozzolanic and/or hydraulic properties.

The most commonly used mineral additives in common cements are [27–32]:

- Fly ash from hard coal combustion;
- Ground granulated blast furnace slag.

Fly ash is one of the additives with pozzolanic properties. On the other hand, ground granulated blast furnace slag is characterized by pozzolanic-hydraulic activity.

According to the PN-EN 197-1 standard, cements containing:

- Portland clinker (K) in the range of 20–64%;
- Ground granulated blast furnace slag (S) 18–49%;
- Silica fly ash (V) 18–49%;

Are called CEM V composite cements.

The purpose of developing this type of cement was to use the synergy effect, i.e., the beneficial shaping of the properties of cement composites through the interaction of individual main components. The environmental aspect is also important. From the point of view of CO_2 emissions and saving energy and natural resources, the use of hydraulic-pozzolanic additives (granulated blast furnace slag and fly ash) gives measurable benefits [30–32].

The influence of fly ashes on the hydration process taking place in the cement slurry, according to many authors, is caused by the fact that the ash components tend to react with Ca^{2+} ions derived from the hydrolysis of calcium silicates in the aqueous environment, creating the so-called C-S-H phases. These are products very similar to the hydrates formed by the hydration of alite and belite. The C-S-H phases show a lower average CaO content in the presence of ash. The content of $Ca(OH)_2$ in hydration products is generally reduced, not only as a result of the pozzolanic reaction of binding Ca^{2+} ions by ash silica, but also as a result of lowering the share of clinker in cement. Active alumina from ash forms hydrated aluminates, ettringite, calcium monosulfate aluminate, and hydrogelenite [27,32].

The effect of the ash depends on its fragmentation and the content of the active substance. The surfaces of ash grains, especially with a high content of fine fractions, facilitate the nucleation of hydration products from the liquid phase, as well as any, even inactive filler with a micron particle size. It should be remembered, however, that the addition of ash is always associated with a reduction in the clinker content in the cement and, thus, with an increase in the water–cement ratio. The introduction of ash to the hydrating system is also always associated with the disruption of the existing balances; the alkalis and sulfates present in small amounts in the ash and passing into the solution exert multiple effects, not only accelerating but also delaying certain processes. Hence, a smaller or greater delay in setting and strength growth rate in the initial period of hardening is generally observed, as well as a decrease in the hydration heat of cements with the addition of ashes [5,7,22,29,33–35].

Significant properties of fly ash-containing cements also include high resistance to the corrosive effects of chemical environments, high watertightness, and limited shrinkage [4,5,33].

The increased resistance to chemical attack of cement with the addition of fly ash is mainly determined by the following [21,22]:

- Reduction in the content of clinker phases susceptible to corrosion, i.e., tricalcium aluminate C_3A in the cement composition, which is related to the reduction in the share of clinker in the cement composition in favor of ash;
- Reduction in $Ca(OH)_2$ content in the hardened cement slurry matrix;
- Change in the microstructure of the hardened cement slurry as a result of the fly ash pozzolanic reaction;
- Tightening of the structure by pozzolanic reaction products and non-hydrated fly ash particles.

Ground granulated blast furnace slag is a material with hydraulic properties. This means that in the presence of water or an activator (e.g., calcium hydroxide, sodium carbonate, alkali, and calcium sulfate), it binds to the formation of the C-S-H phase as the main product. It is also possible to bind without the use of an activator, but then it is a very slow process. Thanks to its binding capacity, blast furnace slag acts as an active hydraulic binder. The C-S-H phase formed during the hydration of blast furnace slags is characterized by a lower CaO/SiO_2 quotient compared to Portland cement. A lower C/S ratio is advantageous in terms of chemical stability. In addition, such a C-S-H phase has an increased ability to incorporate aluminum ions, as well as alkaline cations and chlorine, into its structure. These are the factors that increase the durability of the C-S-H phase derived from slag hydration. Another factor affecting the durability of cement slurries with slag is the content of portlandite, i.e., calcium hydroxide. As the most soluble and least durable phase, portlandite is the weakest link from the point of view of the durability of the cement slurry, and it is the first to corrode. Another advantageous phenomenon from the point of view of the durability of slurries made of binders containing ground granulated blast furnace slag is a different porosity structure compared to Portland cement. The capillary porosity in slurries containing ground granulated blast furnace slag is reduced compared to slurries made of Portland cements. This effect is particularly visible in longer periods, because the hydration of slag is slower compared to the hydration of Portland cement.

This is related to the problem of permeability of slurries containing ground granulated blast furnace slag. Significantly lower permeability of slag pastes results in reduced ion diffusion coefficients. This is a key issue from the point of view of the durability of hardened slurries. This is a valuable property, especially when it comes to slurries used in aggressive waters containing significant amounts of dissolved salts, such as sulfates, carbonates, or magnesium salts [6,27,30,33,36–41].

In addition to modifying the functional properties of hardened slurries, cements with the addition of ground granulated blast furnace slag also affect the technological properties of slurries in the fresh state. Cements containing ground granulated blast furnace slag inevitably have a reduced content of tricalcium aluminate, i.e., the most active phase in cement, which has the strongest influence on the behavior of fresh slurries. This results in extended setting times, which is beneficial, especially in the case of deeper holes. Cements based on blast furnace slag have a lower dynamics of strength increase in the initial period of hardening [23,33,36]. Up to 28 days, slag slurries have lower strength than slurries made of pure Portland cement. After this time, this strength evens out, and over time it begins to significantly exceed this strength. The process of increasing strength can take up to several years.

Cements containing ground granulated blast furnace slag, due to the lower content of active clinker phases, liquefy better with commonly used admixtures. This is despite the fact that the water demand associated with a larger milling surface is usually greater. Slag grains, however, are less active in relation to admixture particles, which results in better liquefaction effects.

Special binders called geopolymers and fine-grained additives often increase the mechanical strength and reduce the permeability of the cement sheath. It is important that the cements used in drilling should allow extrusion during injection and extrusion into the borehole. However, after binding, they must show high mechanical strength [42–45]. For this purpose, tests of compressive strength and bending strength are carried out. Determination of these mechanical parameters allows for the correct design of the cement slurry recipe, which will form an impermeable structure of the cement sheath, preventing the flow of gas after cementing. In addition, the knowledge of the bending strength allows for the design of, among others, a cement slurry with a higher coefficient of elasticity, which contributes to the increase in the durability of the cement coat under the influence of emerging stresses.

1.2. Study Objectives

The laboratory tests carried out are aimed at determining the influence of the chemical and mineralogical composition of the tested hydraulic geopolymer binders. The influence of fresh and hardened cement slurries applied during the cementation of lining pipe columns in boreholes is determined. Table 1 shows the chemical composition of the cement and the additives used, while Table 2 shows the percentage of these additives in the tested geopolymer cement. The tests are carried out on the basis of applicable standards. The main purpose of the research is to determine the concentration of ground granulated blast furnace slag and silica fume on the mechanical parameters of hardened cement slurries for various water–cement coefficients.

Table 1. Chemical composition of cement and additives used.

Component	Portland Cement CEM I 32.5R	Silica Ash *	Ground Granulated Blast Furnace Slag **
SiO_2	21.7	47.52	39.70
Al_2O_3	5.00	30.64	8.15
Fe_2O_3	2.27	7.32	0.82
CaO	64.7	3.35	42.90
MgO	2.20	2.15	5.97
SO_3	3.00	1.44	1.97

* silica fly ash from hard coal combustion in a swirl-pulverized coal boiler was used in the tests. Ignition losses are 2.1%. ** Blast furnace slag comes from Huta im. T. Sendzimir in Nowa Huta.

Table 2. Percentage share of main ingredients included in Formulas A and B.

The Main Ingredients of the Recipes	Cement CEM V/A According to the PN-EN Standard 197–1	CEM V/A Cement Prepared According to Formula A	CEM V/A Cement Prepared According to Recipe B
Clinker content, % by weight	40–60	53.0	45.0
Slag content (S), % by weight	18–30	25.0	21.0
Silica ash content (V), % by weight	18–30	18.0	30.0
Set time regulator, % by weight	0–5	5.0	5.0

2. Materials and Methods

2.1. Materials

The material for laboratory tests is CEM V multi-component cement. Two recipes of CEM V multi-component cement, marked as recipe A and recipe B, are prepared for laboratory tests.

Table 1 shows the chemical composition of the cement and the additives used, and Table 2 shows the percentage share of the main components of the CEM V/A 32.5R multi-component cement.

Silica ash used as a mineral additive in Portland cements extends the setting time and affects the strength, which is characterized by quite slow dynamics in the initial phase. In the longer maturation period, the strength of ash cements exceeds the compressive strength of, for example, Portland cement of the same strength class. Ground granulated blast furnace slag is a material with hydraulic properties. This means that in the presence of water or an activator (e.g., calcium hydroxide, sodium carbonate, alkali, and calcium sulfate), it acts as an active hydraulic binder, causing the binding process with the formation of the C-S-H phase as the main product. As a result, there is a significant improvement in technological parameters of both fresh and hardened cement slurry.

2.2. Methods

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

1. PN–EN 197–1. Cement. Part 1. Composition, requirements, and compliance criteria for common cements, 2012 (after amendment) [46];
2. PN–EN ISO 10426–1. Oil and gas industry. Cements and materials for cementing holes. Part 1. Specification, 2010 [47];
3. PN–EN ISO 10426–2. Oil and gas industry. Cements and materials for cementing holes. Part 2: Testing of drilling cements, 2006 [48].

The laboratory tests carried out were aimed at determining the technological parameters of fresh and, above all, the mechanical properties of hardened cement slurries prepared on the basis of multi-component cement CEM V in terms of application possibilities when cementing columns of casing pipes in deep boreholes.

Tables 3–6 present the results of laboratory tests on the impact of the water–cement coefficient on the technological parameters of fresh cement slurries.

Table 3. Technological parameters of fresh cement slurries' water–cement coefficients: 0.4; 0.5; 0.6; 0.8; 1.0; and 1.2.

Density, kg/m^3		Sedimentation, %		Fluidity, mm		Relative Viscosity, s		Filtration, mL/s	
Cement A	Cement B	Cement A	Cement B	Cement A	Cement B	Cement A	Cement B	Cement A	Cement B
1870	1840	0.0	0.0	115	95	-	-	60/15	33/10
1810	1730	0.0	0.0	130	125	-	-	86/32	71/22

Table 3. *Cont.*

Density, kg/m^3		Sedimentation, %		Fluidity, mm		Relative Viscosity, s		Filtration, mL/s	
Cement A	Cement B	Cement A	Cement B	Cement A	Cement B	Cement A	Cement B	Cement A	Cement B
1700	1650	0.0	0.64	205	165	28.96	36.37	104/23	100/26
1530	1540	2.78	5.0	245	115	21.67	17.83	138.24	150/35
1480	1440	12.12	14.6	>260	>260	12.97	13.64	164/26	174/33
1390	1360	16.89	19.4	>260	>260	9.99	11.56	178/19	198/33

Table 4. Setting times of the tested cement slurries.

Start of Cement Setting, Hour/Minute		Start of Cement Setting, Hour/Minute		Setting Time, Hour/Minute	
Cement A	Cement B	Cement A	Cement B	Cement A	Cement B
4:44	4:58	7:14	7:58	2:30	3:00
5:12	6:40	8:12	10:50	3:00	4:10
6:18	7:19	10:28	12:49	4:10	5:30
12:16	13:34	20:16	22:54	8:00	9:20
19:59	21:38	32:39	35:28	12:40	14:50
25:13	27:47	43:23	47:47	18:10	20:00

Table 5. Rheological parameters for various rheological models of cement slurry prepared on the basis of CEM V/A cement.

Rheological Model Type								
Bingham			Oswald de Waele			Casson		
Plastic Viscosity. Pa·s	Yield Limit. Pa	Correlation Coefficient. -	Consistency Factor. Pa·s^n	Exponent. -	Correlation Coefficient. -	Plastic Viscosity. Pa·s	Yield Limit. Pa	Correlation Coefficient. -
0.1878	30.1511	0.9252	5.4882	0.5299	0.9863	0.1443	11.4248	0.9468
0.0951	10.3443	0.9849	2.4033	0.5257	0.9913	0.0680	4.3147	0.9941
0.0802	8.1247	0.9852	1.9619	0.5237	0.9904	0.0588	3.2458	0.9949
0.0165	3.1323	0.9884	1.2755	0.3514	0.9420	0.0092	1.8420	0.9754
0.0132	1.4979	0.9880	0.4881	0.4512	0.9378	0.0080	0.8107	0.9849
0.0072	1.0656	0.9539	0.2484	0.4858	0.9924	0.0052	0.4398	0.9722

Table 6. Rheological parameters for different rheological models of cement grout based on CEM V/B cement.

Rheological Model Type								
Bingham			Oswald de Waele			Casson		
Plastic Viscosity. Pa·s	Yield limit. Pa	Correlation Coefficient. -	Consistency Factor. Pa·s^n	Exponent. -	Correlation Coefficient. -	Plastic Viscosity. Pa·s	Yield Limit. Pa	Correlation Coefficient. -
0.2065	32.1488	0.9235	6.2071	0.5213	0.9884	0.1596	12.0115	0.9454
0.1143	16.0715	0.9630	3.9503	0.4864	0.9966	0.0814	6.8714	0.9811
0.0660	9.0184	0.9855	2.5733	0.4610	0.9836	0.0423	4.5109	0.9957
0.0205	2.1667	0.9934	0.7932	0.4343	0.9408	0.0129	1.1281	0.9959
0.0102	1.3864	0.9914	0.5764	0.3810	0.9303	0.0059	0.8000	0.9938
0.0079	0.7660	0.9878	0.2555	0.4590	0.9450	0.0053	0.3552	0.9931

Laboratory tests related to the determination of mechanical parameters of hardened cement slurries include the measurement of bending and compressive strength using a testing machine—model E183 PN 100 by Matest (Figure 1).

This machine is used to test samples of hardened cement slurry with dimensions of 40 × 40 × 160 mm (Figure 2). It has two test chambers, one for testing the breaking of beams and the other for compressing samples (Figure 3). It has automatic control of the increase in force (kN/s) depending on the expected compressive strength. The test range is 15 kN for breaking and 250 kN for compression.

Figure 1. Testing machine model E183 PN 100 by Matest.

Figure 2. Samples of hardened cement slurry for compressive and bending strength tests.

2.3. Experimental Procedures

The strength test is carried out on beams with dimensions of 40 mm × 40 mm × 160 mm. The slurries are prepared at the temperature of 20 °C (293 K). After 24 h of curing, the beams made of slurries, for which the end of setting is less than 15 h, are demolded. In the case of longer setting times, demolding is carried out only after 48 h. (this eliminates the destruction of samples during demolding). Then, the beams are stored in a bath with water at 20 ± 2 °C until they reach the age of maturation, at which the assessment of strength characteristics was assumed.

Figure 3. Testing using a testing machine model E183 PN 100 by Matest. Compressive strength test of hardened cement slurry.

Beams intended for tests after a longer period of maturation (except those tested after 24 h or 48 h in the case of delayed demolding) are taken out of the water 15 min before the date of the tests. The maturing age of the beams is counted from the time the grout is prepared, and the mold is poured with it.

Strength tests are carried out with slurries with a w/c coefficient of 0.4, 0.5, and 0.6 after 1, 2, 7, 14, 21, and 28 days after earning. Samples with a w/c ratio of 0.8 are tested after 2, 7, 14, 21, and 28 days. On the other hand, slurries with w/c coefficients of 1.0 and 1.2 are tested after 7, 14, 21, and 28 days.

3. Results and Discussion

3.1. Flexural Strength

The results of bending tests of hardened cement slurries after various times from the moment of making the samples are presented in Table 7. Based on these data, differences in the formation of bending strength of hardened slurries prepared with cements of recipes A and B can be seen in the maturation of the samples. The water–cement coefficient has the greatest impact on strength. Increasing the concentration of water in the cement slurry reduces the strength. Slurries based on the cement of recipe A have greater strength in relation to slurries prepared with the cement of recipe B. Changes in strength occur already after 2 days from the moment of their preparation. The highest flexural strength is characterized by slurries with a water–cement coefficient of 0.4, prepared with the cement of recipe A. The lowest strength, on the other hand, is characterized by slurries based on the cement of recipe B and had a w/c equal to 1.2.

Table 7. Summary of bending strength of cement slurries of recipes A and B.

w/c	Flexural Strength. MPa											
	1 day		2 days		7 days		14 days		21 days		28 days	
	Cement A	Cement B	Cement A	Cement B	Cement A	Cement B	Cement A	Cement B	Cement A	Cement B	Cement A	Cement B
0.4	<1.29	<1.29	3.68	3.29	6.77	5.65	7.94	6.39	9.37	8.79	9.94	9.18
0.5	<1.29	<1.29	2.12	1.98	4.15	4.10	5.87	5.21	7.19	6.64	8.03	7.01
0.6	<1.29	<1.29	<1.29	<1.29	3.17	2.43	3.93	3.75	5.12	4.41	5.38	4.67
0.8	-	-	<1.29	<1.29	1.41	<1.29	1.87	1.83	2.46	2.29	2.89	2.47
1.0	-	-	-	-	<1.29	<1.29	<1.29	<1.29	<1.29	<1.29	1.57	<1.29
1.2	-	-	-	-	<1.29	<1.29	<1.29	<1.29	<1.29	<1.29	<1.29	<1.29

3.2. Compressive Strength

The results of compressive strength tests of hardened cement slurries after various times from the moment of making the samples are presented in Table 8. Based on the analysis of the obtained results, it is concluded that the seasoning time of the hardened cement slurry samples, as well as the w/c coefficients and the mineralogical composition of the cement, affect the formation of strength for compression. The water–cement coefficient has the most significant impact on strength. With its increase, the mechanical compressive strength decreases. The value of silica ash concentration in cement significantly affects strength parameters. An increase in the concentration of ash in cement causes a slower increase in the strength of the samples during its hardening. The highest compressive strength is characterized by the hardened slurry with a water–cement coefficient equal to 0.4, mixed with the cement of recipe A (from 3.51 to 42.36 MPa), while the lowest strength has slurries with a w/c equal to 1.2, mixed with the cement of formula B (from 0.85 to 2.48 MPa).

Table 8. Compressive strength of cement slurries of recipes A and B.

w/c	Compressive Strength. MPa											
	1 day		2 days		7 days		14 days		21 days		28 days	
	Cement A	Cement B	Cement A	Cement B	Cement A	Cement B	Cement A	Cement B	Cement A	Cement B	Cement A	Cement B
0.4	3.51	2.92	9.79	9.17	22.01	20.07	32.78	27.89	39.17	33.99	42.36	35.67
0.5	1.87	1.25	4.86	4.83	11.11	10.42	17.01	15.34	22.99	19.65	25.12	21.18
0.6	0.94	0.83	2.92	2.78	6.81	6.24	10.01	9.65	14.24	12.92	15.55	13.55
0.8		-	1.34	0.86	3.39	2.72	4.53	3.97	6.46	5.23	7.89	6.90
1.0		-			1.71	1.41	2.97	2.74	2.78	2.51	4.14	3.23
1.2		-			1.39	0.85	1.94	1.41	2.38	1.71	2.86	2.48

Figure 4 shows the mechanical compressive strength of samples of hardened grout after 2 days of hydration, while Figure 5 shows the increase in compressive strength after 28 days of hydration.

3.3. Statistical Elaboration of Strength Parameters of Hardened Slurries

The purpose of statistical research is to determine the dependence of the bending and compressive strength of hardened slurries as a function of time. To obtain the correlation, three mathematical models are considered (linear, logarithmic, and exponential), which are presented below:

linear model

$$y = a \times x + b \tag{1}$$

logarithmic model

$$y = a + b \times \ln(x) \tag{2}$$

exponential model

$$y = a \times \exp(b \times x) \tag{3}$$

where

x—sample maturation time, [days];
y—mechanical strength, [MPa].

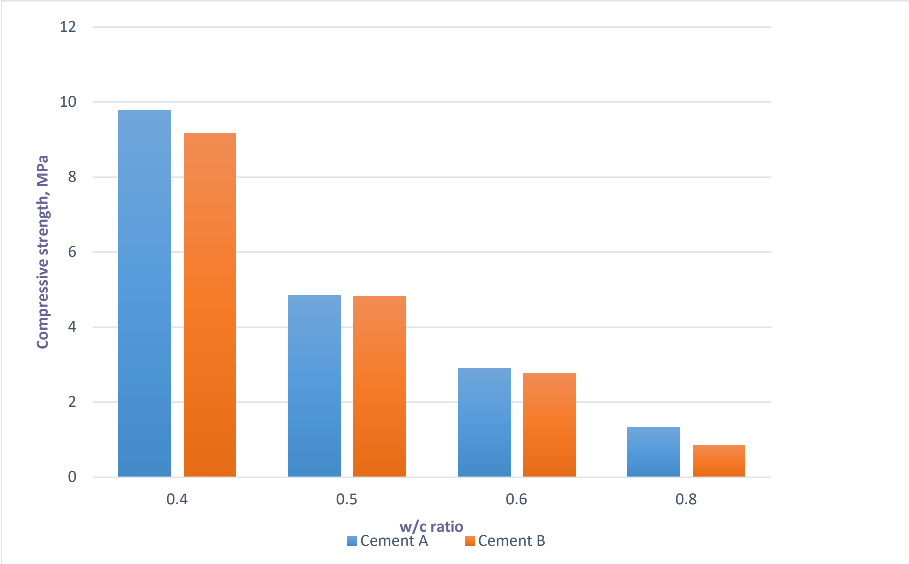

Figure 4. Mechanical compressive strength of samples of hardened grout after 2 days of hydration.

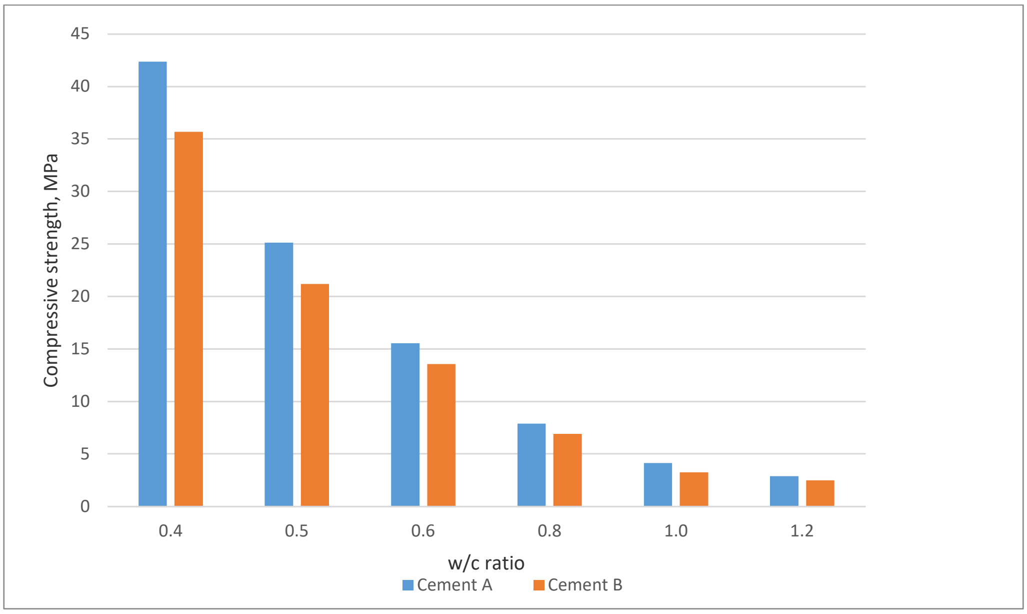

Figure 5. Mechanical compressive strength of samples of hardened cement slurry of recipes A and B after 28 days of hydration.

Statistical calculations aimed at determining the model that best describes the course of changes in bending strength as a function of time are made on the basis of the measurement of hardened samples of cement slurry for w/c coefficients of 0.4, 0.5, 0.6, and 0.8. Water–cement coefficients 1.0 and 1.2 were omitted in these calculations, because changes in bending strength within 21 days of their preparation do not exceed the minimum standard value of 1.29 MPa. On the other hand, statistical calculations aimed at determining the model that best describes the course of changes in compressive strength as a function of their hardening time are performed for all water–cement coefficients. Statistical calculations of the strength parameters of samples prepared with the cement of recipes A and B were made for all three mathematical models (Tables 9–14).

Table 9. Parameters of linear regression for the course of changes in flexural strength of cement slurries of recipes A and B.

LINEAR MODEL $y = a \times x + b$						
Type of Cement Slurry	***w/c***	**Regression Equation Coefficient**		**Correlation Coefficient r, [-]**	**Factor R^2, [-]**	**Fisher–Snedecor Coefficient F, [-]**
		a	**b**			
Cement slurry recipe A	0.4	0.2244	4.3082	0.9430	0.8893	24.1060
	0.5	0.2536	2.1122	0.9797	0.9599	71.7953
	0.6	0.1250	2.1350	0.9725	0.9459	34.9361
	0.8	0.0718	0.9000	0.9983	0.9965	571.1264
Cement slurry recipe B	0.4	0.2234	3.4421	0.9672	0.9355	43.5372
	0.5	0.1879	2.2811	0.9617	0.9249	36.9384
	0.6	0.1054	1.9700	0.9516	0.9055	19.1587
	0.8	0.0457	1.2367	0.9695	0.9400	15.6735

Table 10. Logarithmic regression parameters for the course of changes in flexural strength of cement slurries of recipes A and B.

LOGARITHMIC MODEL $y = a + b \cdot \ln(x)$						
Type of Cement Slurry	***w/c***	**Regression Equation Coefficient**		**Correlation Coefficient r, [-]**	**Factor R^2, [-]**	**Fisher–Snedecor Coefficient F, [-]**
		a	**b**			
Cement slurry recipe A	0.4	2.0502	5.4229	0.9970	0.9940	3112.4804
	0.5	−0.0731	5.7660	0.9746	0.9499	198.3691
	0.6	−0.9098	4.3964	0.9882	0.9776	829.7789
	0.8	−0.7455	2.4391	0.9790	0.9584	375.3474
Cement slurry recipe B	0.4	1.4761	5.1208	0.9697	0.9404	262.7923
	0.5	0.5017	4.4317	0.9920	0.9840	792.2141
	0.6	−0.7192	3.8098	0.9934	0.9869	1554.5960
	0.8	−0.6198	2.1579	0.9888	0.9778	1517.1351

Table 11. Parameters of exponential regression for the course of changes in flexural strength of cement slurries of recipes A and B.

EXPONENTIAL MODEL $y = a \cdot \exp(b \cdot x)$						
Type of Cement Slurry	***w/c***	**Regression Equation Coefficient**		**Correlation Coefficient r, [-]**	**Factor R^2, [-]**	**Fisher–Snedecor Coefficient F, [-]**
		a	**b**			
Cement slurry recipe A	0.4	4.9360	0.0273	0.9091	0.8266	106.4780
	0.5	3.0499	0.0399	0.9416	0.8866	86.7829
	0.6	2.6045	0.0277	0.9508	0.9041	202.0713
	0.8	1.1736	0.0330	0.9913	0.9827	903.6949
Cement slurry recipe B	0.4	4.0752	0.0313	0.9411	0.8857	136.3506
	0.5	2.9149	0.0341	0.9223	0.8506	83.3380
	0.6	2.3718	0.0261	0.9232	0.8524	136.9600
	0.8	1.4226	0.0204	0.9586	0.9189	415.3929

Table 12. Parameters of linear regression for the course of changes in compressive strength of cement slurries of recipes A and B.

LINEAR MODEL $y = a \cdot x + b$						
Type of Cement Slurry	***w/c***	**Regression Equation Coefficient**		**Correlation Coefficient r, [-]**	**Factor R^2, [-]**	**Fisher–Snedecor Coefficient F, [-]**
		a	**b**			
Cement slurry recipe A	0.4	1.4047	7.8456	0.9569	0.9156	43.3882
	0.5	0.8594	3.3711	0.9788	0.9580	91.2240
	0.6	0.5367	1.8813	0.9800	0.9604	97.0610
	0.8	0.2434	1.2167	0.9924	0.9848	194.7949
	1.0	0.1069	1.0300	0.9700	0.9410	31.8842
	1.2	0.0693	0.9300	0.9990	0.9980	1000.9574

Table 12. *Cont.*

LINEAR MODEL y = a·x + b						
Type of Cement Slurry	*w/c*	**Regression Equation Coefficient**		**Correlation Coefficient r, [-]**	**Factor R^2, [-]**	**Fisher–Snedecor Coefficient F, [-]**
		a	**b**			
Cement slurry recipe B	0.4	1.1700	7.3828	0.9465	0.8958	34.3880
	0.5	0.7160	3.4008	0.9645	0.9303	53.3948
	0.6	0.4713	1.9272	0.9691	0.9392	61.8059
	0.8	0.2195	0.7748	0.9922	0.9845	190.7455
	1.0	0.0813	1.0500	0.9548	0.9117	20.6388
	1.2	0.0741	0.3150	0.9863	0.9728	71.5054

Table 13. Logarithmic regression parameters for the course of changes in compressive strength of cement slurries of recipes A and B.

LOGARITHMIC MODEL y = a + b·ln(x)						
Type of Cement Slurry	*w/c*	**Regression Equation Coefficient**		**Correlation Coefficient r, [-]**	**Factor R^2, [-]**	**Fisher–Snedecor Coefficient F, [-]**
		a	**b**			
Cement slurry recipe A	0.4	1.9919	27.1983	0.9945	0.9890	716.6913
	0.5	0.3002	16.0342	0.9803	0.9610	179.1859
	0.6	−0.0007	9.9719	0.9773	0.9552	150.3351
	0.8	−0.7439	5.3993	0.9631	0.9275	106.8569
	1.0	−1.4291	3.6374	0.9540	0.9101	135.9283
	1.2	−0.6763	2.3684	0.9866	0.9734	622.3449
Cement slurry recipe B	0.4	2.2483	22.9610	0.9970	0.9940	1379.236
	0.5	0.5526	13.7020	0.9909	0.9818	407.3334
	0.6	0.1373	8.9193	0.9845	0.9692	227.5012
	0.8	−1.0060	4.8818	0.9654	0.9320	100.3627
	1.0	−0.9903	2.9095	0.9874	0.9749	587.4224
	1.2	−1.3432	2.4834	0.9544	0.9110	94.60491

Table 14. Exponential regression parameters for the course of changes in compressive strength of cement slurries of recipes A and B.

EXPONENTIAL MODEL y = a·exp(b·x)						
Type of Cement Slurry	*w/c*	**Regression Equation Coefficient**		**Correlation Coefficient r, [-]**	**Factor R^2, [-]**	**Fisher–Snedecor Coefficient F, [-]**
		a	**b**			
Cement slurry recipe A	0.4	1.9919	27.1984	0.8954	0.8017	374.3244
	0.5	0.3001	16.0342	0.9235	0.8529	749.2554
	0.6	−0.0007	9.9719	0.9261	0.8577	589.4292
	0.8	2.1589	0.0480	0.9662	0.9335	116.6963
	1.0	1.4572	0.0370	0.9686	0.9381	197.9869
	1.2	1.1887	0.0319	0.9910	0.9820	922.2521
Cement slurry recipe B	0.4	11.6613	0.0441	0.8842	0.7818	35.82672
	0.5	6.1084	0.0483	0.9049	0.8188	39.09004
	0.6	3.7669	0.0499	0.9079	0.8243	38.25687
	0.8	1.6797	0.0518	0.9648	0.9308	98.66027
	1.0	1.3932	0.0312	0.9286	0.8624	106.3110
	1.2	0.6658	0.0468	0.9903	0.9807	439.9509

Tables 15 and 16, for comparison purposes, show the values of correlation coefficients for the tested samples of hardened cement slurries made of cements of recipes A and B.

Table 15. List of correlation coefficients of regression equations for the course of changes in flexural strength of cement slurries of recipes A and B.

Type of Cement Slurry	*w/c*	Mathematical Model Type					
		Linear		Logarithmic		Exponential	
		Correlation Coefficient r, [-]	Factor R^2, [-]	Correlation Coefficient r, [-]	Factor R^2, [-]	Correlation Coefficient r, [-]	Factor R^2, [-]
Cement slurry recipe A	0.4	0.9430	0.8893	0.9970	0.9940	0.9091	0.8266
	0.5	0.9797	0.9599	0.9746	0.9499	0.9416	0.8866
	0.6	0.9725	0.9459	0.9882	0.9776	0.9508	0.9041
	0.8	0.9983	0.9965	0.9790	0.9584	0.9913	0.9827
Cement slurry recipe B	0.4	0.9672	0.9355	0.9697	0.9404	0.9411	0.8857
	0.5	0.9617	0.9249	0.9920	0.9840	0.9223	0.8506
	0.6	0.9516	0.9055	0.9934	0.9869	0.9232	0.8524
	0.8	0.9695	0.9400	0.9888	0.9778	0.9586	0.9189

Table 16. List of correlation coefficients of regression equations for the course of changes in compressive strength of cement slurries of recipes A and B.

Type of Cement Slurry	*w/c*	Mathematical Model Type					
		Linear		Logarithmic		Exponential	
		Correlation Coefficient r, [-]	Factor R^2, [-]	Correlation Coefficient r, [-]	Factor R^2, [-]	Correlation Coefficient r, [-]	Factor R^2, [-]
Cement slurry recipe A	0.4	0.9569	0.9156	0.9945	0.9890	0.8954	0.8017
	0.5	0.9788	0.9580	0.9803	0.9610	0.9235	0.8529
	0.6	0.9800	0.9604	0.9773	0.9552	0.9261	0.8577
	0.8	0.9924	0.9848	0.9631	0.9275	0.9662	0.9335
	1.0	0.9700	0.9410	0.9540	0.9101	0.9686	0.9381
	1.2	0.9990	0.9980	0.9866	0.9734	0.9910	0.9820
Cement slurry recipe B	0.4	0.9465	0.8958	0.9970	0.9940	0.8842	0.7818
	0.5	0.9645	0.9303	0.9909	0.9818	0.9049	0.8188
	0.6	0.9691	0.9392	0.9845	0.9692	0.9079	0.8243
	0.8	0.9922	0.9845	0.9654	0.9320	0.9648	0.9308
	1.0	0.9548	0.9117	0.9874	0.9749	0.9286	0.8624
	1.2	0.9863	0.9728	0.9544	0.9110	0.9903	0.9807

A total of 10 samples for bending strength and 20 samples for compression are analyzed for each water–cement ratio. It was assumed that the most important factor influencing the strength parameters of hardened cement slurries is its w/c ratio and slurry hardening time, assuming that the grout recipe and setting and hardening temperature are constants.

Bearing this fact in mind, statistical elaboration of the strength results was carried out, taking into account the hardening time of the samples for each of the tested w/c coefficients.

All tested slurries are characterized by high correlation coefficients. The power model can be adopted as the best description of changes in bending and compressive strength as a function of hardening time. For bending strength, the highest correlation coefficient is characterized by a cement slurry with a w/c coefficient of 0.8 with the described linear model (0.9983) mixed with the cement of recipe A. In addition, a very high coefficient is characterized by a slurry with a w/c coefficient of 0.6 mixed with the cement of recipe A, which is described by the power model (0.9934). On the other hand, for compressive strength, the highest correlation coefficient has a slurry with w/c equal to 1.2, prepared with the cement of recipe A (0.9990), described by a linear model. For the cement of recipe B, the highest correlation has a slurry with w/c 0.4 (0.9970) described by the power model.

Based on the values of the correlation coefficients of the analyzed recipes, it is concluded that the best fit of the rheological model to the measurement data occurs for the Casson model. An increase in the w/c ratio, i.e., a decrease in the concentration of cement in slurries, improves rheological parameters in the tested cement slurries. The value of these parameters is also affected by the percentage of ash in the cement. The calculations presented in the tables show that slurries made of cement A are characterized by lower values of plastic viscosity. On the other hand, the highest viscosity is characterized by cement slurry with a w/c coefficient of 0.4, made on the basis of cement B, which has a viscosity of 0.2065 Pa·s, while the slurry with the same w/c but made on cement A has a viscosity of 0.1878 Pa·s. All tested slurries are characterized by high correlation coefficients. For slurries prepared on cement A, the highest correlation of 0.9941 has a w/c coefficient of 0.6. However, for cement B, the slurry with the highest correlation coefficient is the one with a w/c coefficient of 0.5, and the correlation value is 0.9966.

4. Conclusions

Detailed remarks concerning the determination of the influence of additives in the form of ground granulated blast furnace slag and silica fly ash on the formation of technological parameters of fresh and hardened cement slurries are formulated in the previous chapters of this publication. The above observations are determined on the basis of literature research and analysis of the developed results of laboratory tests presented in tabular and graphic form, and are statistically developed. The results of statistical modeling indicate the accuracy of the analyzed models with the results obtained from laboratory tests. Their summary leads to specifying the following general conclusions:

1. The hydration time of the hardened cement slurry and the w/c ratios, as well as the mineralogical compositions of cements of recipes A and B, influence the development of strength parameters;

2. An increase in the concentration of silica fly ash in the cement slurry causes a delay in the rate of increase in strength in the initial period of its hardening;

3. Ground granulated blast furnace slag reduces the dynamics of strength growth in the initial period of hardening. Up to 28 days of hardening, slurries with the addition of ground granulated blast furnace slag have lower strength than cement slurries made of pure Portland cement;

4. The most important influence on the strength parameters of hardened cement slurries is primarily the w/c ratio. The higher its w/c values, the lower the strength. The cement slurries prepared on the basis of cement A have higher flexural and compressive strengths than the cement slurries made with the cement of recipe B;

5. Throughout the analyzed period of time up to 28 days, the tested hardened cement slurries showed an increase in both bending and compressive strength. The obtained results from laboratory tests allow forecasting their compressive strength for w/c from 0.4 to 1.2;

6. The strength parameters of hardened cement slurries are mainly influenced by the w/c coefficient. The higher its value, the lower the strength;

7. Cement slurries based on cement A have higher flexural and compressive strengths than slurries based on cement B; this is due to the fact that cement A contains more Portland clinker;

8. Throughout the analyzed time period (up to 28 days), the tested hardened cement slurries show an increase in both bending and compressive strength;

9. Forecasting mechanical parameters depending on the concentration of the additives used is useful in drilling practice, as it allows the optimization of the so-called "setting time" after cementing casing columns in boreholes.

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