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## **THE INFLUENCE OF ANTI-SEDIMENTATION AGENTS ON THE TECHNOLOGICAL PARAMETERS OF CEMENT SLURRY\*\*\***

### **1. INTRODUCTION**

The efficiency of insulating permeable zones between pipes and beyond them mainly depends on properly designed slurry. Significantly, the requirements for cement slurries are mainly conditioned by the number of tripped casing columns, geological-technical conditions, design of the well and its designation.

Cement slurries to be used for sealing boreholes in definite geological-technical conditions have specific physical properties, e.g. density, filtration, lack of shrinking during hydration. The cement sheath made of such slurry should have appropriate mechanical parameters and be impervious to gas. However, when sealing directional and horizontal boreholes attention should be paid to considerably stricter regimes for slurry parameters, i.e. sedimentation stability, water settling (free water) and the filtration of slurry. These are the most important parameters determining the usability of slurry for sealing directional boreholes [2, 10, 13, 18].

The number of additional factors influences this situation by directly improving the sedimentation stability of cement slurry, i.e. one of the most important factors contributing to efficient sealing of directional and horizontal boreholes.

### **2. PARAMETERS DETERMINING THE SEDIMENTATION STABILITY OF CEMENT SLURRY DISPERSION**

When sealing casing columns, solid particles settle and the water settling effect takes place (Fig. 1). The values of these parameters increase with the increasing drift of the

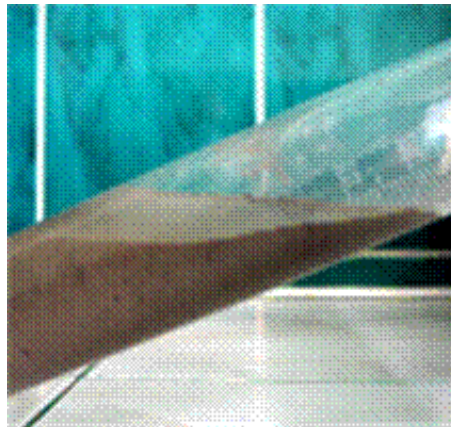
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borehole's axis from vertical. Those fractions of the slurry which are heavier than working water gravitationally drop down much faster than lighter ones. This phenomenon is observed mainly in the case of drilling muds containing weighting material. The fractioning of cement slurry in a vertical borehole takes place on much of its length, therefore this effect can hardly be detected. In directional or horizontal boreholes, the fractioning can be observed at a section of ten or so centimeters long, i.e. which sometimes equals to the diameter of the borehole. For this reason, the homogeneity of fresh slurry in directional and horizontal boreholes is very important [5, 11, 13, 17]. Moreover, the sediment sealing slurry may have a lower density in the upper part of the borehole, which results in anisotropy of the hardened slurry. The cement sheath made of nonhomogeneous slurry will considerably vary (from one measuring point to another) in such parameters as mechanical strength, adhesiveness to steel tubes, porosity and permeability [8, 11, 12, 19]. Also bearing in mind the higher values of water settlement in cemented annular space under the top part of wall of horizontal or strongly drifted borehole, we may assume that when the cement binds, this space will be a canal space (Fig. 2) for potential gas migration along the casing column on the cement/casing interface [3, 6, 9, 14].



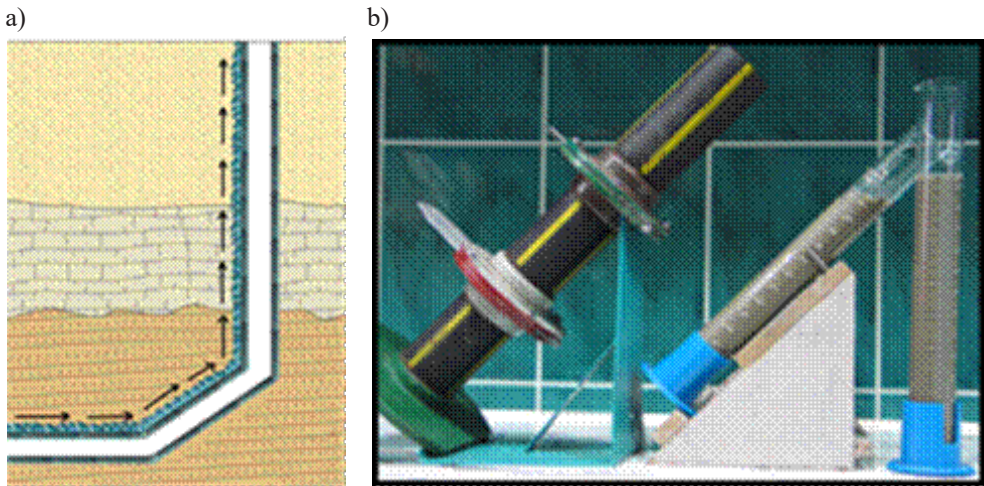
**Fig. 1.** Water settling in slurry having low sedimentation stability

The sedimentation stability of cement slurry is closely connected with the amount of working water in cement slurry, i.e. water/cement ratio. The amount and type of dispersing agent (liquefier) also plays an important role. For the sake of obtaining a uniform structure of liquid cement slurry, various fine-grain additives, admixtures or polymers are used to increase the viscosity of the dispersion [11, 12, 16, 20]. Attention should be paid to the problems when designing the composition of a sedimentation-stable slurry, which will be so liquefied as to enable its pumping at low pressure, where the turbulent flow is possible. This inconvenience is connected with the fact that lowering of intermolecular attraction forces, thanks to which the liquidity of the slurry is improved, is connected with the free movement of fine fractions due to external forces [1, 6]. Accordingly, the liquefied slurry tends to destabilize under the influence of the settling particles and precipitation of free water. When sealing directional and horizontal boreholes, attention is mainly paid to properties such as sedimentation stability, water settling (free water) and filtration of slurry. It should be emphasized that they

are the most important parameters deciding about the efficiency of sealing of directional boreholes [2, 7, 10]. The sedimentation stability of cement slurry can be established with the use of different techniques. The most accurate ones lie in laser measurement of quantitative distribution of particles in a volume of fluid in the analyzed column at a given measuring point. In industrial conditions it is most frequently the amount of water precipitated over the cement slurry (water settling) which is measured. Owing to the fact that cement slurry may settle, and does not show water settlement, the measurement of the density of slurry in the sedimentation column is most efficient (photo 2). Based on the measurement of the density of the slurry in particular parts of the borehole (top, middle, bottom) we can determine the fractioning of the cement slurry dispersion.

The analysis of sedimentation allows for the determination of the homogeneity of the liquid slurry, though it is important to know that also other factors affect the fractioning of the slurry, i.e. :

- water settling (free water),
- density of cement slurry,
- rheological parameters of cement slurry,
- filtration of cement slurry,
- time of thickening and bounding of cement slurry [8, 12].



**Fig. 2.** Stand for determining sedimentation stability of cement slurry and water settling:  
a) sedimentation column; b) cylinder for water settling measurement

During the general interpretation of the above parameters, some kind of dependence between mentioned slurry properties can be observed. This is associated with the presence of cement slurry in the dispersion which contains a number of additives and admixtures playing different functions. Accordingly, each cement slurry should be treated individually, depending on its designation for specific geological-technical conditions. Moreover, applied additives and admixtures should be analyzed in view of their influence on the other technological parameters (except sedimentation stability), which is the subject of this publication.

### 3. RESEARCH WORK

Research works on determining the influence of anti-sedimentation agents on the technological parameters of cement slurries were based on standards: PN-EN 10426-2 *Oil and gas industry. Cements and materials for cementing boreholes. Part 2: Analysis of drilling cements* and API SPEC 10 *Specification for materials and testing for well cements*.

The main purpose of laboratory experiments was to determine the quantitative and qualitative influence of anti-sedimentation admixtures on the technological parameters of cement slurry. The slurry recipes were analyzed for two borehole conditions: temperature 30°C and pressure 5 MPa, and 50°C and 18°C MPa, respectively. All recipes were based on drilling cement G HSR.

Base slurry was used for analyses (low sedimentation stability). Then the recipe was modified, i.e. various anti sedimentation admixtures were used in definite quantities (based on preliminary analyses). The type of applied agents, recipes of slurries, as well as quantities of particular additives and admixtures are listed in Tables 1 and 2, whereas the obtained technological parameters for the analyzed group of recipes are presented in Tables 3 and 4.

**Table 1**

Recipes of cement slurries for borehole conditions: temp. 30°C, pressure 5 MPa

Constant components: cement G HSR = 100%, microcement 20.0%, salinity of working water KCl = 3.0% (bwow), defoamer = 0.3% (bwoc), swelling agent = 0.3% (bwoc)											
No.	Composition (No.) ratio (w/c)	Anti filtration agent	Liquefier	Latex	Latex stabilizer	2G Anti migration polymer	Polymer increasing viscosity3X	4B Bentonite	5P Fine grain agent	6C Water dispersion of sodium silicate	Calcium choride
1.	No. 1 w/c = 0.45	0.20	0.20	10.0	1.0	-	-	-	-	-	4.0
2.	No. 2 w/c = 0.45	0.20	0.30	10.0	1.0	4.0	-	-	-	-	4.0
3.	No. 3 w/c = 0.45	0.20	0.35	10.0	1.0	-	0.15	-	-	-	2.0
4.	No. 4 w/c = 0.50	0.20	0.20	10.0	1.0	-	-	0.50	-	-	2.0
5.	No. 5 w/c = 0.52	0.20	0.20	10.0	1.0	-	-	-	0.50	-	2.0
6.	No. 6 w/c = 0.50	0.20	0.20	10.0	1.0	-	-	-	-	3.0	2.0

**Table 2**

Recipes of cement slurries for borehole conditions: temp. 50°C, pressure 18 MPa

Constant components: cement G HSR = 100%, microcement 20.0%, salinity of working water KCl = 3.0% (bwow), defoamer = 0.3% (bwoc), swelling agent = 0.3% (bwoc)											
No.	Composition (No.) ratio (w/c)	Anti filtration agent	Liquefier	Latex	Latex stabilizer	2G Anti migration poly- mer	Polymer increasing viscosity3X	4B Bentonite	5P Fine grain agent	6C Water dispersion of sodium silicate	Retarder
1.	No. 7 w/c = 0.50	0.20	0.10	10.0	1.0	-	-	-	-	-	0.10
2.	No. 8 w/c = 0.50	0.20	0.10	10.0	1.0	4.0	-	-	-	-	0.10
3.	No. 9 w/c = 0.50	0.20	0.10	10.0	1.0	-	0.15	-	-	-	0.10
4.	No. 10 w/c = 0.50	0.20	0.10	10.0	1.0	-	-	0.50	-	-	0.10
5.	No. 11 w/c = 0.50	0.20	0.10	10.0	1.0	-	-	-	0.50	-	0.10
6.	No. 12 w/c = 0.50	0.20	0.20	10.0	1.0	-	-	-	-	3.0	0.20

Technological parameters were analyzed for a group of 12 slurries presented in Table 1 (six recipes for temperature 30°C and 50°C). The obtained results are listed in Table 2.

The rheological parameter, determined with a rotary viscometer Ofite 900, were analyzed. The dependence between tangential stresses ( $\tau_i$ ), and shear rates ( $\dot{\gamma}$ ) were defined. The plastic viscosity and yield point were established and the obtained results presented in Table 2.

The filtration of cement slurries in dynamic conditions was determined with a filtration press HPHT 7120 by Chandler. The thickening time of the analyzed slurries was defined with a pressure consistometer OFITE 130. On this basis, the change of consistency could be measured and the time of the thickening of cement slurry determined.

The first group of slurries were designed for sealing casing columns seated at a depth of about 200 m to about 800 m, and sealed at a temperature of 30°C and pressure 5 MPa. These slurries were based on drilling cement G HSR. The analysis of the obtained results reveals that the density of slurries varied from 1800 kg/m<sup>3</sup> (slurry with admixed fine grained 5P) to 1885 kg/m<sup>3</sup> base slurry 1Baz (Fig. 3). The analysis of sedimentation stability of the analyzed slurries shows that fractioning could be observed only in base slurry 1, in the remaining cases the required sedimentation stability was obtained.

**Table 3**  
Technological parameters of cement slurries

No.	Slurry	Cement	Temp [°C]		Density [kg/m <sup>3</sup> ]	Sedimentation stability (density at measuring point) [kg/m <sup>3</sup> ]			Spillability [mm]	Water settling [%]	Filtration [cm <sup>3</sup> /30min]	Thickening time * [hrs - min]		Readouts from Fann apparatus [readout j.f.** at r.p.m.]					
			30°C	50°C		Top	Center	Bottom				30Bc	100Bc	600	300	200	100	6	3
1	1Baz	Cement G HSR	30°C	5 MPa	1885	1880	1885	1890	245	0.1	46	4:20	4:50	190	107	78	45	14	10
2	2G				1850	1850	1850	1850	155	0.0	7	2:10	2:45	290	265	205	140	61	52
3	3X				1880	1880	1880	1880	230	0.0	35	2:48	3:34	312	174	125	72	10	8
4	4B				1830	1830	1830	1830	220	0.3	36	2:35	3:12	217	128	97	65	30	28
5	5P				1800	1800	1800	1800	235	0.4	27	4:04	4:34	203	114	83	51	16	15
6	6C				1810	1810	1810	1810	235	0.0	25	2:20	3:00	202	114	83	51	13	12
7	1Baz	Cement G HSR	50°C	18 MPa	1840	1830	1840	1850	215	0.2	56	3:17	3:39	207	119	89	55	16	14
8	2G				1810	1810	1810	1810	205	0.0	10	5:27	6:24	312	170	120	68	8	6
9	3X				1840	1840	1840	1840	215	0.0	112	3:27	3:55	215	121	91	56	16	14
10	4B				1830	1830	1830	1830	205	0.4	126	2:45	3:13	217	128	96	62	20	19
11	5P				1810	1810	1810	1810	205	0.5	171	3:04	3:30	235	137	101	63	18	16
12	6C				1810	1810	1810	1810	190	0.0	14	1:50	2:06	204	122	94	63	26	24

\* time to reach temp: 30°C = 10 min; to temp. 50°C = 25 min

\*\* j.f. Fann unit

**Table 4**  
Technological parameters of cement slurries

Rheological model	Rheological parameters	Slurry											
		For temp. 30°C; pressure 5 MPa						For temp. 50°C; pressure 18 MPa					
		1Baz	2G	3X	4B	5P	6C	1Baz	2G	3X	4B	5P	6C
Bingham's model	Plastic viscosity [Pa·s]	0.0899	0.1227	0.1526	0.0947	0.0944	0.0953	0.0966	0.1535	0.1005	0.0994	0.1099	0.0900
	Yield point [Pa]	6.9568	44.1517	7.3285	15.6283	8.6391	7.7284	9.4213	5.5564	9.2112	12.0442	10.8404	14.5043
	Correlation coefficient [-]	0.9985	0.9142	0.9974	0.9989	0.9991	0.9982	0.9973	0.9883	0.9979	0.9968	0.9970	0.9972
Ostwald-Waale's model	Consistency coefficient [Pa·s <sup>n</sup> ]	2.0184	14.5727	1.1332	6.8483	2.9319	2.1719	2.7547	0.7911	2.7201	3.9319	3.1215	5.6918
	Exponent [-]	0.5263	0.3372	0.6981	0.3611	0.4770	0.5256	0.4952	0.5224	0.5013	0.4493	0.4963	0.3838
	Correlation coefficient [-]	0.9618	0.9855	0.9915	0.9298	0.9493	0.9646	0.9643	0.9939	0.9623	0.9591	0.9659	0.9465
Casson's model	Casson's viscosity [Pa·s]	0.0635	0.0648	0.1300	0.0504	0.0623	0.0675	0.0657	0.1360	0.0687	0.0630	0.0749	0.0504
	Yield point [Pa]	2.9038	24.2912	1.5163	9.5669	4.1442	3.1676	4.1373	0.8973	4.0336	5.9079	4.7122	8.3111
	Correlation coefficient [-]	0.9993	0.9568	0.9995	0.9962	0.9985	0.9997	0.9998	0.9996	0.9996	0.9997	0.9999	0.9988
Herschel-Bulkley's model	Yield point [Pa]	4.8629	3.1358	2.2632	13.7108	6.8416	5.1355	6.1903	1.5795	6.3393	8.3565	6.8915	11.4099
	Consistency coefficient [Pa·s <sup>n</sup> ]	0.1914	11.9352	0.4289	0.1842	0.1771	0.2271	0.2734	0.3525	0.2489	0.3099	0.3322	0.2612
	Exponent [-]	0.8916	0.3668	0.8519	0.9045	0.9097	0.8755	0.8509	0.8808	0.8699	0.8371	0.8414	0.8473
	Correlation coefficient [-]	0.9999	0.9863	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	0.9999	1.0000	1.0000	0.9999

The spillability was observed to considerably decrease (Tab. 3) in slurry 2G (admixture of anti migration polymer), which is connected with a considerable increase of consistency coefficient  $\tau_{HB}$  (Fig. 5). The filtration in this slurry is lowest (7.2 cm<sup>3</sup>/30 min) and the thickening time is short (2 hrs. 10 min for 30 Bc<sup>1</sup>, and 2 hrs. 45 min for 100 Bc). With this information in mind, the anti migration agent 2G lowers filtration, water settling and considerably increases rheological parameters of the slurry. This can be caused by the presence of a suspension based on working water and its increased viscosity. This and the necessity to additionally liquefy the slurry should be accounted for when designing the slurry. In the analyzed group of recipes (Tab. 3) we can observe a favorable operation of anti sedimentation admixtures on the stability of cement slurry. However, during complex analysis of technological parameters the admixture of 4B and 5P resulted in a higher water settlement. An excessive value of water settling may stem from the use of fine fractions, which cause a slight increase of the yield point  $\tau_{HB}$  (Fig. 5) and anti sedimentation activity; however in the top part of the measuring column it may slightly settle gravitationally, forming a visible water film on the slurry surface. The filtration in these slurries is at the required level (below 50 cm<sup>3</sup>/30 min).

In the second group are slurries designed for sealing casing columns at a depth of about 800 m to 1300 m, sealed at a temperature of 50°C and pressure 18 MPa. The slurries were also based on drilling cement G HSR. The density of these slurries oscillated between 1810 kg/m<sup>3</sup> (slurry with 2G, 5P, 6C) to 1840 kg/m<sup>3</sup> (base slurry 1Baz and 3X). In this group of slurries the required sedimentation stability was obtained after using admixtures. The fractioning was noted only in the base slurry. The analysis of the influence of the applied admixtures on the rheological parameters of the analyzed slurries shows the similar values of the rheological parameters (Fig. 5). A slight increase of the rheological parameters (plastic viscosity  $c_{ss}$  and consistency coefficient  $\tau_{HB}$ ) was visible only after applying admixture 2G, which also in the previous group of slurries increased the values of rheological parameters. The use of these admixtures also lowered filtration from 56 cm<sup>3</sup>/30 min (base slurry) to 10 cm<sup>3</sup>/30 min (slurry with 2G) – Table 3, Figure 5. However, in this group of slurries the admixture 2G (Fig. 4) strongly elongated the time of thickening (5 hrs. 27 min for 30 Bc and 6 hrs. 24 min for 100 Bc). Such behavior can be explained by the synergy of the anti-migration agent 2G and the applied thickening retarder. In the analyzed group of slurries the addition of fine grain admixtures 4B, 5P caused a considerable (126 cm<sup>3</sup>/30 min and 171 cm<sup>3</sup>/30 min, respectively) increase of filtration. The application of a polymer increasing viscosity 3X caused a higher filtration from 56 cm<sup>3</sup>/30 min in base slurry 1Baz to 112 cm<sup>3</sup>/30 min in a slurry containing 3X. These effects can be caused by the incompatibility of the administered admixtures and the retarder. These recipes should be additionally modified to reduce the filtration of the slurry. Analogous to the previous group of slurries, the fine grain admixtures 4B and 5P resulted in the slight precipitation of free water.

The analyses in hardened cement slurries covered mechanical parameters, i.e. compressive strength, adhesiveness to steel pipes, porosity and permeability. Strength analyses were performed with Chandler 4207. This machine allowed for static tests of bending, compression and adhesiveness of cement stone to various surfaces to be conducted. Experiments on the microstructure of hardened sealing slurries were realized with a mercury porosimeter MicroMetrics.

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<sup>1</sup> Bc – unit of Bearden's consistency of slurry while measuring time of thickening in consistometer



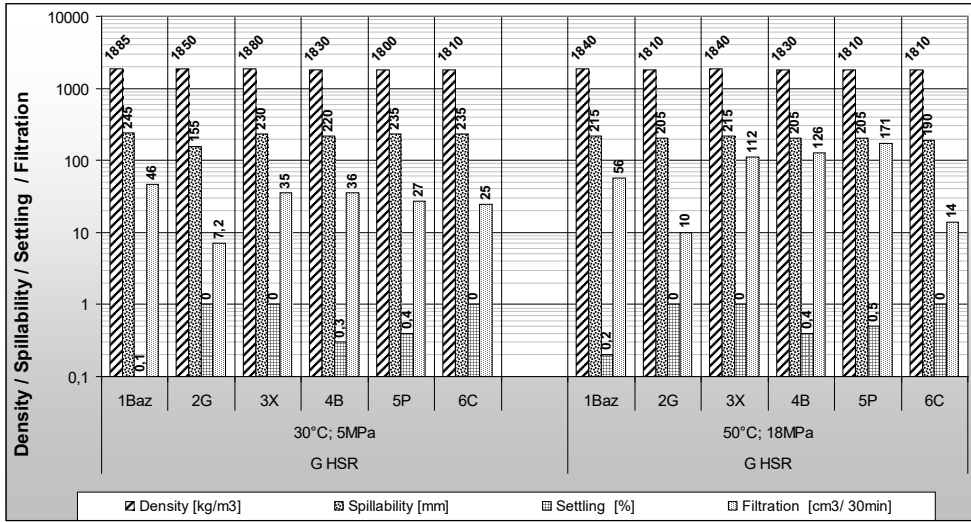


Fig. 3. Density, spillability, settling and filtration of cement slurries

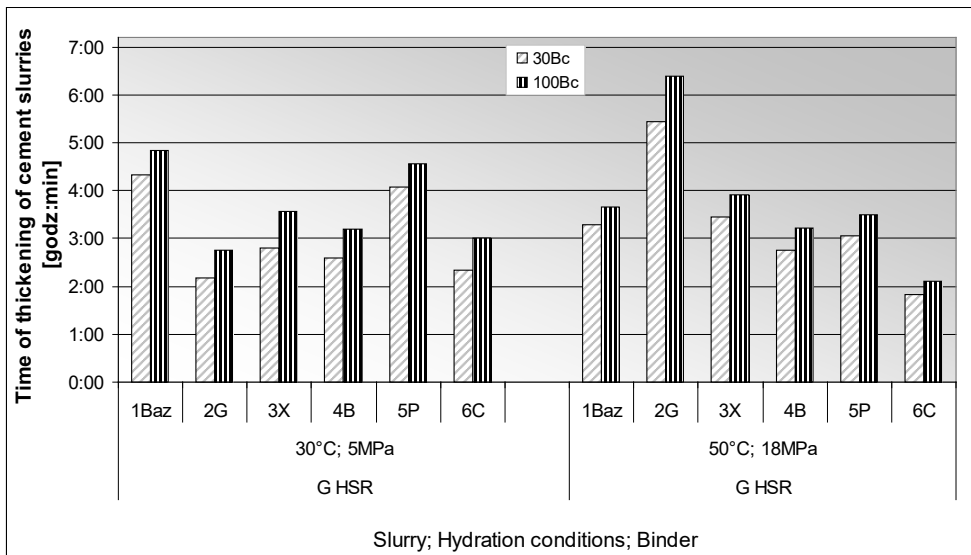


Fig. 4. Measurements of time of thickening of cement slurries

The analyses of hardened sealing slurries were mainly focused on mechanical parameters and porosity, which describe the microstructure of hardened cement slurry. Comparing the obtained strength results on compression in a group of slurries to be applied at temperature 30°C and pressure 5 MPa, we can observe an increase of compressive strength and adhesiveness to steel pipes after applying admixture 6C (Tab. 4).

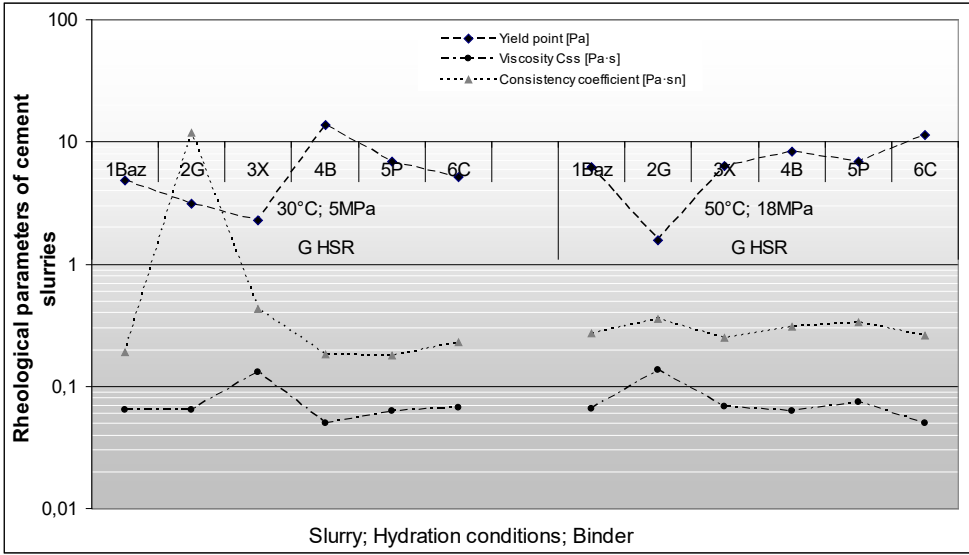


Fig. 5. Results of the measurement of the rheological parameters of cement slurries

Table 4  
Mechanical parameters of hardened slurries

No of sample	Admixture	Conditions of hydration [temp. pressure]	Analyzed parameter of hardened slurry							
			Compressive strength [MPa]			Adhesiveness to steel tubes [MPa]			Permeability [mD]	Porosity [%]
			Hydration time							
			2 days	7 days	28 days	2 days	7 days	28 days	28 days	
1	1Baz	30°C 5 MPa	9.3	11.6	13	6.2	7.5	8.7	0.12	37.15
2	2G		10.1	10.9	12.3	4.9	5.1	8.5	0.05	35.15
3	3X		10.6	11.2	11.9	3.8	5.9	7.5	0.07	35.81
4	4B		9.8	11.9	12.6	5	5.5	6.5	0.16	36.22
5	5P		9	11.4	11.8	5.1	6.8	8.6	0.09	39.42
6	6C		11.3	14.1	14.8	6.8	9	9.7	0.09	36.87
7	1Baz	50°C 18 MPa	16.4	16.8	17.2	6.8	7.9	9.1	0.14	36.92
8	2G		11	13.6	14.1	5.9	6.1	8.8	0.07	35.55
9	3X		12.8	13.7	14.3	4	6.9	8.7	0.09	35.77
10	4B		11.3	12.4	12.7	6.9	6.5	7.3	0.08	35.67
11	5P		8.6	11.4	12.1	4.1	5.8	7	0.15	38.88
12	6C		9.3	12.3	12.7	6.2	7.8	8.5	0.12	36.75

Such results can be explained by hydration reinforced by an admixture of water dispersion of sodium silicate. The comparison of the parameters describing the microstructure of the sample (porosity, permeability) shows that the strongest sealing of hardened cement slurry matrix was obtained after admixing anti migration polymer 2G (Tab. 4).

In the second group of slurries, the mechanical parameters were observed to slightly lower after applying agents improving sedimentation stability. This may be the reason to carry on modifications of the recipes. However the microstructure of the hardened slurry was improved as in the previous group of slurries. It resulted in the lowering permeability to gas and porosity after using admixture 2G. The results of analyses of mechanical parameters are listed in Table 5.

Concluding, anti sedimentation admixtures show a considerable influence on the technological parameters of cement slurry. Various types of admixture improve the sedimentation stability and also have an influence on the remaining parameters of cement slurry. The analyses confirm the very differentiated advantageous and disadvantageous operation of the applied agents. The use of fine grain admixtures favorably lowers the sedimentation of the slurry, but may also increase water settling and filtration. The use of an anti-migration polymer may result in higher rheological parameters of slurry and lower filtration and water settling, which is advantageous in view of anti sedimentation activity. However this agent may significantly elongate the thickening time. This should be accounted when planning slurry for given borehole conditions.

#### 4. CONCLUDING REMARKS

The following conclusions can be drawn from the obtained results of laboratory analyses:

- Some anti sedimentation admixtures (3X, 4B, 5P) advantageously influence the stability of slurry, but also may increase water settling and the filtration of slurry. This means that additional anti filtration agents have to be applied.
- Admixtures counteracting the sedimentation of slurry increase the viscosity of working water and slurry. The effect of the above is stronger counteracting forces of gravity affecting the heavier fractions in the cement slurry.
- The increase of plastic viscosity of cement slurry by using anti sedimentation admixture may lower pumpability, and so increase the pumping pressure. This is important for obtaining the required turbulent flow.
- Because of the increase of rheological parameters due to the applied anti sedimentation admixtures, sometimes a liquefier has to be used to lower the rheological parameters, maintaining the required sedimentation stability.
- The agents improving stability of slurry may sometimes result in a visible (over three times) increase of filtration. This behavior of admixtures prompts their further detailed analyses.
- The use of anti-sedimentation admixtures results in a homogeneous structure of the cement sheath. This is confirmed by lower permeability and porosity values in samples (admixture of 2G).
- Owing to the indicated significant influence of anti-sedimentation admixtures on the technological parameters of cement slurries, recipes modified with agents improving sedimentation stability should be analyzed laboratory on specialist apparatuses each time.

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