AGH DRILLING, OIL, GAS • Vol. 33 • No. 4 • 2016

http://dx.doi.org/10.7494/drill.2016.33.4.781

Marcin Kremieniewski*, Marcin Rzepka*, Stanisław Stryczek, Rafał Wiśniowski** Łukasz Kotwica***, Albert Złotkowski****

TECHNOLOGICAL PARAMETERS OF INNOVATIVE CEMENT SLURRIES USED FOR SEALING WELLBORES IN SHALE FORMATIONS****

1. INTRODUCTION

The major aim of sealing casing in wellbores drilled on shale gas fields and in conventional wells lies in obtaining maximum tightness of the drilled permeable zones and protection of groundwater. This imposes definite technological restrictions when working out recipes of slurry with appropriately selected parameters. The final result will be a tight cement sheath, resistant to the migration of fluids in the annular space. The properties of the slurry, and so the presence of various additives and admixtures, mainly depends on the geological and technical conditions in the wellbore, i.e. in the annular space where hydration and formation of hardened cement slurry take place. The type of the drilled geological layers, ultimate depth of drilling, static and dynamic temperature, as well as reservoir pressure and fracturing pressure were the most important factors [11, 13, 15, 16]. Works realized within Optidrilltec, aimed at working out innovative cement slurries, were preceded by a detailed analysis of the geological structure. Additional emphasis was put on the analyses of recipes of slurries already used in a given area. The analyses were focused on parameters of fresh and hardened slurries and on the final effect, i.e. cemented wellbore. The collected data, mainly on the slurry parameters, were used for modifying old recipes and working out new compositions. These works were undertaken to create a recipe of a slurry which would have optimum technological parameters for a particular geological structure. Such a cement slurry should eliminate the risk of badly performed cementing, and so the resulting complications and difficulties with removing consequences of possible outflows of the reservoir medium between

* Oil and Gas Institute – National Research Institute, Krakow, Poland
** AGH University of Science and Technology, Faculty of Drilling, Oil and Gas, Department of Drilling and Geoengineering, Krakow, Poland
*** AGH University of Science and Technology, Faculty of Materials Science and Ceramics, Department of Building materials Technology, Krakow, Poland
**** The work realized within the Blue Gas Program – Optidrilltec project financed by the National Center for Research and Development

and beyond the casing. Owing to the high cost of additional works, i.e. sealing up, innovative cement slurries for sealing casing in the perspective areas were worked. According to the Investor's Representatives, they seem to be a very advantageous option.

2. CRITERIA FOR SLURRIES TO BE USED FOR SEALING CASING IN SHALE FORMATION

Sealing of the casing in directional wells, especially the horizontal ones, when shale formations are drilled, is one of the most serious challenges for the engineers. These difficulties are connected with more restrictive standards for sealing fluids used in horizontal wells as compared to those used in their vertical counterparts [7]. Thus, the innovative cement slurries designed by AGH-UST and OGI-NRI, which will be used for sealing casing in shale formations, should meet the following criteria:

– Optimum injectivity in time needed to pump the cement slurry into the annular space. This time should be extended by a safety margin (time in which the thickening begins in HTHP (High Temperature Pressure High Pressure)conditions should equal to about 180 to 300 min, depending on the depth of the wellbore).
– The bounding should begin right after the slurry has been injected to the annular space. However, the time from the moment the cement slurry is injected and the annulus is filled with the material, to the moment the slurry solidifies and is able to tightly seal the space should be relatively short.
– Short transition time (TT) from 50 Pa to 250 Pa when the growing static structural strength (SSS) is measured [3, 6, 16].
– Optimum rheological parameters allowing for efficient displacing of the mud and buffering fluid from the wellbore, maintaining minimum resistance of flow. Additionally, the maximum radius of filling of the sealed medium can be obtained [8, 14].
– Sedimentation stability and no settling water.
– Filtration of this type of slurries in high temperature and high pressure conditions should not exceed 50 cm^3/30 min [4, 5, 10, 14, 16].

Hardened cement slurries in the wellbore should meet the following requirements:

– good interface insulation (high adhesiveness to steel and rock formation after bounding in the wellbore),
– low permeability and low porosity,
– high compressive strength in HTHP conditions,
– they should insulate the casing column against aggressive media and also against crushing by the swelling rock formation,
– no contraction during hydration [1, 2, 9, 10, 12, 16, 17].

When working out innovative recipes of cement slurries which meet the above criteria and which can be used for sealing casing columns in shale formations, the researchers also analyzed technological parameters of the currently used slurries [16]. Some of them have correct technological parameters, but owing to the high qualitative regimes for slurries which will be used for sealing casing columns in shale formations, innovative compositions based on new additives and admixtures were worked out.

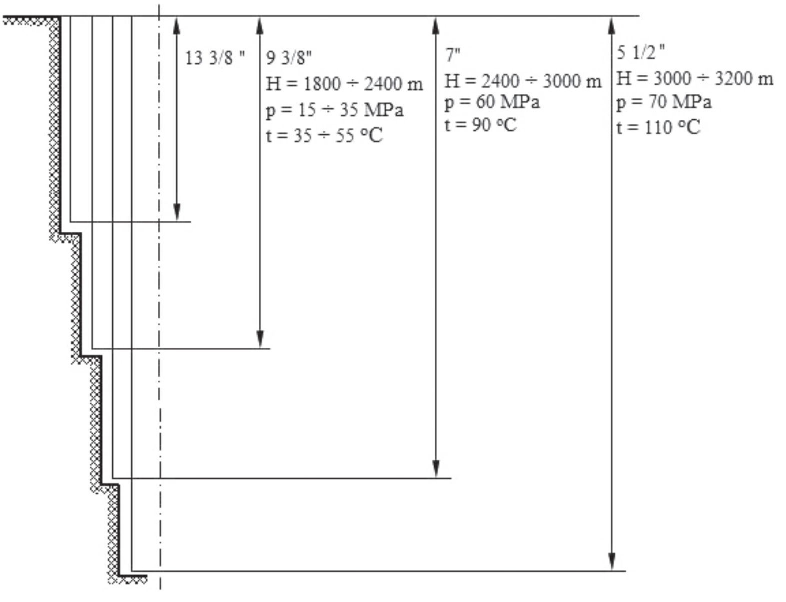


Fig. 1. Schematic of wellbore conditions

Among the main additives were dispersants, polymeric additives stopping gas migration and sedimentation of slurry, as well as fine-grain additive for lowering the density of the upper (light) slurry.

A schematic of the casing and wellbore conditions where the new slurries will be injected, i.e. depth of the well, temperature and pressure on the bottom as well as the diameter of the casing to be sealed, is visualized in Figure 1. The wellbore conditions (depth, temperature and pressure) for the analyzed slurry compositions are presented in Table 1 further in this paper.

3. LABORATORY TESTS

A series of experiments aimed at working out innovative recipes of cement slurries which will be used for sealing wells in shale formations was performed at the Laboratory of Sealing Slurries, the Drilling Technology Department, OGI-NRI and the Faculty of Drilling Oil and Gas, Department of Drilling and Geoengineering, AGH-UST. The experiments were conducted in compliance with the standards: API Spec 10 and PN-EN 10426-2 Oil and Gas Industry *Cements and materials for cementing wellbores – part 2*.

Cement slurries were analyzed with the high class measuring apparatuses owned by the Laboratory of Sealing Slurries, OGI-NRI (e.g. mixers, Baroid weight, pressure consistometer, filtration press, Fann viscosimeter, porosimeter, gaseous permeability meter, ultrasonic cement analyzer UCA + SGSM for measuring the mechanical and static structural strength of gelling cement slurries, strength testing machine for determining compressive strength and adhesiveness of cement stone to steel casing – produced by Chandler and Ofite).

While elaborating the innovative recipes, the technological parameters of 5 groups of slurries were analyzed. The first group consisted of slurries with an addition of dispresive agents, the second had an addition of agents fighting migration, the third and the fifth groups were based on latex polymers, and a counter-migration agent Gasblok. In the third groups the slurry was elabrated for a casing column 9 5/8", whereas the fifth for 7" and 5 ½" (HTHP). Slurries belonging to the fourth group were modified with light fine-grain additives. On the whole, 31 innovative recipes were analyzed, and 14 of which were selected and presented in Table 1.

Attention was drawn to the recipes of slurries to be used for sealing casing pipes 9 5/8". Besides new recipes of slurries were elaborated for casing columns 7" and 5 ½". The results of the analyses of selected slurries are presented in Table 2. For the sake of distinguishing the obtained results, the most advantageous values were marked in green and the least favorable – in red. The positive to negative values ratio indicating which of the recipes is optimum, is presented graphically in the lower part of the table. On this basis the best compositions in the analyzed group could be compared.

The rheological parameters, which were determined with the use of a rotary viscosimeter Ofite model 900 were analyzed. The dependence of tangential stress (τi) on shear rate ($\dot{\gamma}I$) was established. In line with standard PN-EN 10426-2, plastic viscosity (two point method) and yield point were measured and the obtained results are listed in Table 2.

The filtration of the slurry, measured with the filtration press (Chandler model 7120) was also analyzed. The filtration of cement slurries was conducted in dynamic conditions.

The second group of slurries (5 to 8) had in their composition an agent hindering gas migration. Tests were performed on these slurries to determine if the additives can limit gas migration through the bounding and bounded slurry structure. The static structural strength (SSS) and transition time (TT) measurements were performed for the SSS values from 50 Pa to 250 Pa. The analyses were performed with the Static Gel Strength Measurement device, thanks to which the increasing static structural strength of the sealing slurry could be measured while its was gelifying.

All the samples were measured for their time of thickening. The measurement was conducted with a pressure consistometer OFITE model 130; the changes in the consistency and time of thickening of the cement slurry were recorded.

The hardened cement slurry samples underwent mechanical tests for compressive strength, adhesiveness to steel pipes, porosity and permeability to gas. Strength tests were performed on a strength testing machine Chandler model 4207, where the bending tests, compressive tests and tests for adhesiveness of the cement stone to various surfaces could be performed.

The microstructure of hardened cement slurries was measured with a mercury porosimeter MicroMetrics, and permeability to gas with a permeability meter OFITE.

Statistical calculations (Tab. 3 and 4) were performed on the basis of the obtained results.

They were used for determining average compressive strength values, adhesiveness of particular samples to steel casing and confidence intervals (assumed value: $1 - \alpha = 0.95$). The analyzed recipes were analyzed for the compressive strength of hardened cement and adhesiveness to steel casing obtained for three samples (in line with PN-EN ISO 10426). The arithmetic mean of the analyzed parameters in MPa was calculated on this basis.

Table 1

Composition of analyzed slurries

Composition of slurry		1	2	3	4	5	6	7	8	9	10	11	12	13	14
Slurry label		s. 1	s. 2	s. 3	s. 4	s. 5	s. 6	s. 7	s. 8	s. 9	s. 10	s. 15	s. 20	s. 25	s. 29
Additive		LIQ				AMI				LATE	FINE	LATE			
Name of additive		P4	L5	L1	C4	GS	AD6	N6	PG	D5	PP	D5	D6	D5	D6
Casing column ["]		9 5/8	9 5/8	9 5/8	9 5/8	9 5/8	9 5/8	5 1/2	9 5/8	9 5/8	9 5/8	7	7	5 ½	5 ½
Temperature [°C]		50	50	50	50	50	50	50	50	55	55	90	90	110	110
Pressure [MPa]		15	15	15	15	15	15	15	15	24	27	60	60	70	70
Denotations: w/c – water/cement ratio; bwow – in relation to working water. Remaining components added in relation to the mass of cement															
Network water. w/c =		0.52	0.52	0.52	0.52	0.50	0.50	0.50	0.50	0.52	0.60	0.44	0.28	0.44	0.28
Bentonite (bwow)		–	–	–	–	–	–	–	–	–	1.0	–	–	–	–
KCl (bwow)		–	–	–	–	–	–	–	–	3.0	3.0	–	–	–	–
Defoamer		1.0	1.0	1.0	1.0	0.2	0.2	0.2	0.2	0.3	0.5	0.5	0.5	0.5	0.5
Antifiltration agent		0.2	0.2	0.2	0.2	–	–	–	–	0.5	0.3	0.2	0.1	0.1	0.1
Liquifier		0.1	0.1	0.1	0.1	0.4	0.4	0.4	0.4	0.3	0.3	0.2	0.3	0.3	0.3
Counter-migration additive		–	–	–	–	7.0	7.0	7.0	7.0	7.0	–	7.0	20.0	7.0	20.0
Retarder		0.15	0.2	0.15	0.15	–	–	–	–	0.1	0.3	0.25	0.1	0.7	0.2
Latex		10.0	10.0	10.0	10.0	–	–	–	–	–	10.0	–	–	–	–
Stabilizer of latex		2.0	2.0	2.0	2.0	–	–	–	–	–	2.0	–	–	–	–
Microcement		20.0	20.0	20.0	20.0	5.0	5.0	5.0	5.0	–	20.0	5.0	–	5.0	–
Fine-grain additive		–	–	–	–	–	–	–	–	–	2.0	–	–	–	–
Microsilica		–	–	–	–	–	–	–	–	–	–	25.0	25.0	25.0	25.0
Cement	CEM I 32.5R	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	–	–	–	–
	CEM G HSR	–	–	–	–	–	–	–	–	–	–	100.0	100.0	100.0	100,0
Swelling agent		–	–	–	–	0.1	0.1	0.1	0.1	0.1	0.3	–	–	–	–

* denotations: LIQ – dispersant (liquefier), AMI – anitmigration agent (polymer), LATE – latex (co-polymer) and anitmigration co-polymer Gasblok, FINE– fine-grain additive,

Table 2

Parameters of selected slurries

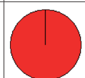
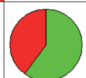
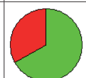
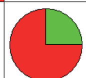
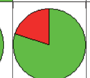
Parameter	Additive	LIQ				AMI				LATE	FINE	LATE			
Slurry composition		1	2	3	4	5	6	7	8	9	10	11	12	13	14
Label of sample used for strength calculations		s. 1	s. 2	s. 3	s. 4	s. 5	s. 6	s. 7	s. 8	s. 9	s. 10	s. 15	s. 20	s. 25	s. 29
Casing column ["]		9 5/8	9 5/8	9 5/8	9 5/8	9 5/8	9 5/8	9 5/8	9 5/8	9 5/8	9 5/8	7	7	5 1/2	5 1/2
Density [kg/m^3]		1790	1790	1790	1790	1800	1800	1800	1800	1760	1660	1930	1940	1930	1940
Spillability [mm]		255	310	270	230	260	270	265	250	210	215	265	220	240	245
Plastic viscosity [mPa·s]		73.5	84.0	108	76.5	76.5	33.0	33.0	48.0	229.5	150.0	265.5	154.5	202.5	133.5
Yield point [Pa]		6.0	3.36	3.36	8.88	3.6	1.44	1.92	4.32	18.48	15.36	15.6	14.16	15.12	1.28
Filtration [cm^3/30 min]		42.0	19	17	34	14	429	491	108	4.5	108	18.0	8.0	52	16
Transition time (TT) [hrs:min]		–	–	–	–	0–22	0–31	0–29	0–15	–	–	–	–	–	–
Thicken-ing time [hrs:min]	30 Bc	2–42	2–51	3–02	3–10	3–03	2–32	2–46	1–37	4–47	3–07	4–53	3–55	4–36	4–32
	100 Bc	3–08	3–19	3–47	4–04	4–11	3–20	3–19	3–41	5–16	4–16	5–26	4–08	4–33	4–42
Permeability (7 days) [mD]		0.060	0.050	0.065	0.060	0.045	0.065	0.075	0.055	0.031	0.023	0.060	0.040	0.038	0.035
Com-pressive strength [MPa]	24 hrs	10.4	9.8	8.2	9.2	9.6	10.6	11.8	12.2	10.1	18.0	24.3	22.0	26.9	26,6
	48 hrs	20.9	18.7	17.5	17.9	17.5	20.6	22.5	22.9	15.2	22.5	28.9	26.1	30.5	30,3
Adhesi-veness to casing	24 hrs	2.6	2.1	2.0	2.2	1.8	2.0	2.4	2.4	2.3	2.2	3.9	3.5	4.9	4,7
	48 hrs	4.4	4.2	3.8	3.8	3.6	3.8	4.6	4.6	3.0	2.2	4.4	4.3	5.5	5,4
Porosity		37.07	35.55	36.25	36.92	33.12	34.27	33.85	33.24	36.73	40.32	37.24	33.18	36.32	34.12
Positive to negative values ratio [%] (green to red)															

Table 3

Statistical calculation for the obtained compressive strength tests on hardened cement slurry samples placed in well-like conditions for 24 hrs and 48 hrs

Hardened cement slurry sample		Results of compressive tests (*Ws*) for three samples of hardened cement slurry [MPa],			Arithmetic mean *Ws* [MPa]	Standard deviation	Confidence interval (for 1 − α = 0.95)	Confidence interval (for 1 − α = 0.95)
Composition No.	Hydration time	Sample 1	Sample 2	Sample 3				
1	24 hrs	10.3	10.6	10.3	10.4	0.1414	0.4303	9.9697 – 10.8303
	48 hrs	21	20.8	20.9	20.9	0.0816	0.2484	20.6516 – 21.1484
2	24 hrs	9.3	9.4	10.7	9.8	0.6377	1.9403	7.8597 – 11.7403
	48 hrs	18.3	18.5	19.3	18.7	0.4320	1.3146	17.3854 – 20.0146
3	24 hrs	8.1	8.2	8.3	8.2	0.0816	0.2484	7.9516 – 8.4484
	48 hrs	17.5	17.6	17.4	17.5	0.0816	0.2484	17.2516 – 17.7484
4	24 hrs	9	9.3	9.3	9.2	0.1414	0.4303	8.7697 – 9.6303
	48 hrs	17.9	18	17.8	17.9	0.0816	0.2484	17.6516 – 18.1484
5	24 hrs	9.6	9.6	9.6	9.6	0.0000	0.0000	9.6000 – 9.6000
	48 hrs	17.5	17.6	17.4	17.5	0.0816	0.2484	17.2516 – 17.7484
6	24 hrs	10.6	10.5	10.7	10.6	0.0816	0.2484	10.3516 – 10.8484
	48 hrs	20.4	20.6	20.8	20.6	0.1633	0.4969	20.1031 – 21.0969
7	24 hrs	11.8	11.7	11.9	11.8	0.0816	0.2484	11.5516 – 12.0484
	48 hrs	22.5	22.5	22.5	22.5	0.0000	0.0000	22.5000 – 22.5000
8	24 hrs	12	12.2	12.4	12.2	0.1633	0.4969	11.7031 – 12.6969
	48 hrs	22	23	23.7	22.9	0.6976	2.1226	20.7774 – 25.0226
9	24 hrs	10	10.1	10.2	10.1	0.0816	0.2484	9.8516 – 10.3484
	48 hrs	15.2	15.3	15.1	15.2	0.0816	0.2484	14.9516 – 15.4484
10	24 hrs	18	18	18	18.0	0.0000	0.0000	18.0000 – 18.0000
	48 hrs	22.5	22.6	22.4	22.5	0.0816	0.2484	22.2516 – 22.7484
11	24 hrs	24.3	24.6	24	24.3	0.2449	0.7453	23.5547 – 25.0453
	48 hrs	29	28.5	29.2	28.9	0.2944	0.8957	28.0043 – 29.7957
12	24 hrs	21	22	23	22.0	0.8165	2.4843	19.5157 – 24.4843
	48 hrs	26	26	26.3	26.1	0.1414	0.4303	25.6697 – 26.5303
13	24 hrs	27	26.7	27	26.9	0.1414	0.4303	26.4697 – 27.3303
	48 hrs	30.5	30.5	30.5	30.5	0.0000	0.0000	30.5000 – 30.5000
14	24 hrs	26.5	26.5	26.8	26.6	0.1414	0.4303	26.1697 – 27.0303
	48 hrs	30	30.4	30.5	30.3	0.2160	0.6573	29.6427 – 30.9573

Table 4

Statistical calculation for obtained results of tests on the adhesiveness of hardened cement slurry to steel casing performed for hardened cement slurry samples placed in well-like conditions for 24 hrs and 48 hrs

Hardened cement slurry sample		Results of adhesiveness tests to steel casing (*Pr*) for three samples of hardened cement slurry [MPa]			Arithmetic mean *Pr* [MPa]	Standard deviation	Confidence interval (for 1 – α = 0.95)	Confidence interval (for 1 − α = 0.95)
Composition No.	Hydration time	Sample 1	Sample 2	Sample 3				
1	24 hrs	2.5	2.6	2.7	2.6	0.0816	0.2484	2.3516 – 2.8484
	48 hrs	4.2	4.4	4.6	4.4	0.1633	0.4969	3.9031 – 4.8969
2	24 hrs	2.1	2.1	2.1	2.1	0.0000	0.0000	2.1000 – 2.1000
	48 hrs	4	4.2	4.4	4.2	0.1633	0.4969	3.7031 – 4.6969
3	24 hrs	2	2	2	2.0	0.0000	0.0000	2.0000 – 2.0000
	48 hrs	3.6	3.8	4	3.8	0.1633	0.4969	3.3031 – 4.2969
4	24 hrs	2	2	2.6	2.2	0.2828	0.8606	1.3394 – 3.0606
	48 hrs	3.8	4	3.6	3.8	0.1633	0.4969	3.3031 – 4.2969
5	24 hrs	1.6	1.6	2.2	1.8	0.2828	0.8606	0.9394 – 2.6606
	48 hrs	3.6	3.4	3.8	3.6	0.1633	0.4969	3.1031 – 4.0969
6	24 hrs	2	2	2	2.0	0.0000	0.0000	2.0000 – 2.0000
	48 hrs	3.8	3.8	3.8	3.8	0.0000	0.0000	3.8000 – 3.8000
7	24 hrs	2.2	2.4	2.6	2.4	0.1633	0.4969	1.9031 – 2.8969
	48 hrs	4.4	4.6	4.8	4.6	0.1633	0.4969	4.1031 – 5.0969
8	24 hrs	2.2	2.4	2.6	2.4	0.1633	0.4969	1.9031 – 2.8969
	48 hrs	4.4	4.4	5	4.6	0.2828	0.8606	3.7394 – 5.4606
9	24 hrs	2.3	2.2	2.4	2.3	0.0816	0.2484	2.0516 – 2.5484
	48 hrs	3	2.8	3.2	3.0	0.1633	0.4969	2.5031 – 3.4969
10	24 hrs	2.2	2.2	2.2	2.2	0.0000	0.0000	2.2000 – 2.2000
	48 hrs	2	2	2.6	2.2	0.2828	0.8606	1.3394 – 3.0606
11	24 hrs	3.8	3.9	4	3.9	0.0816	0.2484	3.6516 – 4.1484
	48 hrs	4.2	4.4	4.6	4.4	0.1633	0.4969	3.9031 – 4.8969
12	24 hrs	3.5	3.4	3.6	3.5	0.0816	0.2484	3.2516 – 3.7484
	48 hrs	4.2	4.2	4.5	4.3	0.1414	0.4303	3.8697 – 4.7303
13	24 hrs	5	5	4.7	4.9	0.1414	0.4303	4.4697 – 5.3303
	48 hrs	5.5	5.8	5.2	5.5	0.2449	0.7453	4.7547 – 6.2453
14	24 hrs	4.7	4.7	4.7	4.7	0.0000	0.0000	4.7000 – 4.7000
	48 hrs	5.5	5.5	5.2	5.4	0.1414	0.4303	4.9697 – 5.8303

The following calculations have been performed (Tab. 3 and 4):
– standard deviation of sample;

$$\sigma(x) = \sqrt{\frac{\sum (x_i - \overline{x})^2}{n}} \tag{1}$$

– confidence interval*;

$$p_u = t_\alpha \cdot \frac{\sigma(x)}{\sqrt{n-1}} \tag{2}$$

– confidence interval*;

$$p\left\{\overline{x} - t_\alpha \cdot \frac{\delta(x)}{\sqrt{n-1}} < x < \overline{x} + t_\alpha \cdot \frac{\delta(x)}{\sqrt{n-1}}\right\} = 1 - \alpha \tag{3}$$

where:

n – number of samples,
x_i – results,
$\overline{x}$ – arithmetic mean,
t_α – Student t value read out from the table of distribution for $n - 1$ degrees of freedom,
p – probability that compressive strength of cement stone equals to $1 - \alpha$ in the interval described with equation (3).

* assumed confidence interval equaled to $1 - \alpha = 0.95$

4. WELLBORE CONDITIONS IN SHALE GAS AREAS

Recipes of slurries, which will be used for sealing casing columns in shale formations were designed for the casing of diameter 9 5/8", 7" and 5½". Based on the results of the analyses, the pressure from 15 MPa to 27 MPa and temperature from 50°C to 55°C were assumed for casing columns 9 5/8", pressure of 60 MPa and temperature of 90°C for casing 7", and pressure of 70 MPa and temperature of 110° for casing 5 1/2" tripped to over 3000 m of depth. A graphical representation of temperature and pressure for the analyzed wellbore conditions is presented in Figure 2.

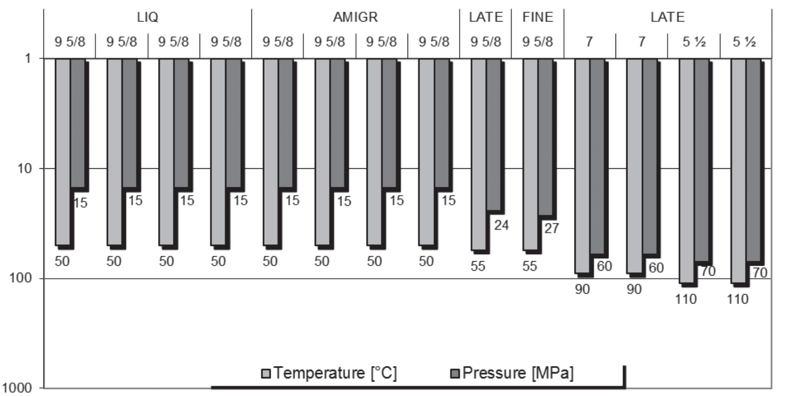


Fig. 2. Temperature and pressure for analyzed slurry recipes

5. RECIPES OF INNOVATIVE CEMENT SLURRIES FOR SEALING CASING COLUMNS IN SHALE FORMATIONS

For casing columns 9 5/8" there were prepared slurries with an addition of new and presently applied dispersing agents (compositions 1 to 4, Tab. 1). In the second group of recipes the slurries contained an agent hindering gas migration (compositions 5 to 8, Tab. 1). The third and the fourth group consisted of a slurry with new latex additive or Gasblok, and a slurry with an addition of fine-grain material, lowering the density of the slurry. The fifth group consisted of slurries based on new polymeric-latex antimigration agents, used for sealing casing columns 7" and 5 ½".

Slurries containing liquefiers, belong to the first group and are referred to as LIQ in Table 1. They contained a defoamer (1%) and antifiltration agent (0.2%). The required time of thickening in view, a retarding material was used (0.15% to 0.2%). A 10% latex additive was used and 20% of microcement to seal up the cement matrix. The water/cement ratio for all recipes equaled to 0.52 and the slurries were based on Portland cement CEM I 32,5 R. For the sake of comparing the operation of innovative dispersing agents, 0.1% of liquefier was used in each of the recipes.

The second group of slurries, denoted as AMI, is represented by compositions containing an agent hindering gas migration. These slurries contain 0.4% of liquefier and 0.2% of defoamer. For the sake of filling the intergranular spaces, 5% of microcement was added. This quantity sufficed as additionally 7% of antimigration polymer was used. The slurries were based on Portland cement CEM I 32,5 R; the water/cement ratio equaled to 0.5. For the sake of eliminating negative changes of volume of the slurry in the course of hydration processes, 0.1% of swelling agent was used.

In the third group (LATE) only one recipe containing a 7% latex addition was selected. The slurry also contained 3% KCl, 0.3% of defoamer; 0.5% of antifiltration agent and 0.3% of liquefier. A retarder was also used to elongate the time of thickening. The slurry was based on Portland cement CEM I 32,5 R. The water/cement ratio equaled to 0.52. A swelling agent was also added.

In the fourth group (FINE) there is also one composition containing 2% of fine-grain material, a light additive which lowers density of the slurry. The water/cement ratio in this recipe equaled to 0.6. In this recipe 1% of bentonite was used for making a suspension counteracting slurry fractioning. Besides, the slurry contains 3% of KCl, 0.5% of decalcifier and antifiltration agent, liquefier and retarder (0.3% of each). Moreover, 10% of latex and 20% of microcement were used to fill the intergranular spaces in the cement sheath. Also, in this case the slurries were based on Portland cement CEM I 32,5 R.

The fifth group of slurries (LATE) for sealing casing columns 7" and 5½" contains recipes with an admixture of new latex and antimigration polymers resistant to HTHP[1] conditions. These slurries were based on drilling cement HSR class G. For the sake of increasing the resistance of slurry to high temperature, a 25% admixture of microsilica was applied. All slurries belonging to this group contained 0.5% of defoamer in their compositions. The antifiltration agent was added (0.1% to 0.2%), and a liquefier (0.2% to 0.3%) to lower the rheological parameters. Besides, 7% of additive D5 was used. Slurries containing this

[1] HTHP – High Temperature, High Pressure.

agent (recipe[2] s. 15 and s. 25) also contained 5% of microcement, and the water/cement ratio equaled to 0.44. The recipes containing 20% of polymer D6 were not added any microcement, and the water/cement ratio equaled to 0.28. For the sake of obtaining optimum time of thickening, the 0.1% to 0.7% of retarder was used. Detailed compositions of innovative recipes of slurries are listed in Table 1.

6. TECHNOLOGICAL PARAMETERS OF FRESH SLURRIES GAIN

The density of innovative cement slurries to be used for sealing casing columns in shale formations ranged between 1760 kg/m^3 and 1940 kg/m^3, only the density of light slurry with the addition of fine-grain material equaled to 1660 kg/m^3 (Tab. 2, Fig. 3).

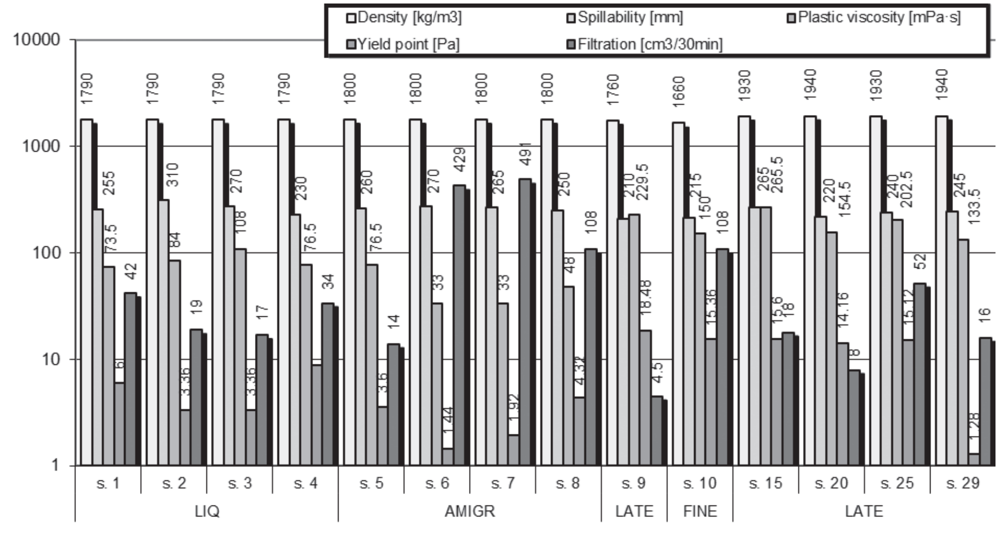


Fig. 3. Parameters of fresh slurries worked out for wells in shale formations

The obtained spillability values ranged from 210 mm for slurry s. 9 containing latex to 310 mm for slurry s. 2 with liquefier added. The remaining rheological parameters, i.e. plastic viscosity ranged from 33 mPa·s (slurries s. 6 and s. 7 with antimigration agent) to 229.5 mPa·s for slurry s. 9 with latex additive, which had a lower spillability in the previous measurement. The yield point equaled from 1.3 Pa (composition s. 29) to 18.5 Pa (composition s. 9). The results are presented in Figure 2 and Table 2. The analysis of the obtained rheological parameters reveals that among slurries for sealing casing column 9 5/8" the most advantageous properties had s. 2 and s. 6 (spillability equal to 310 mm and 270 mm, respectively). The lowest plastic viscosity in sealing slurries for casing 9 5/8" had slurries s. 6 and s. 7, (33 mPa·s, and recipe labeled as s. 8 with viscosity of 48 mPa·s). The best parameters

---

[2] E.g.: s. 15, s. 25 – denote composition no. 15 and no. 25, respectively. Analogous denotations are used a in this paper

among slurries belonging to the fifth group has slurry s.29, with plastic viscosity equal to 133 mPa·s, and yield point totaling to 1.2 Pa. Too high rheological parameters were observed in slurries s. 3, s. 9, s. 10 (for sealing casing 9 5/8") and s. 15 for slurries belonging to the fifth group.

The lowest filtration value in slurries to be used for sealing casing 9 5/8" was observed in recipes s. 2, s. 3, s. 5 and s. 9 (filtration ranged between 4.5 cm^3/30 min and 19 cm^3/30 min). Among slurries from the fifth group the lowest filtration was found in slurries s. 15, s. 20 and s. 29 (8 cm^3/30 min to 18 cm^3/30 min). The lowest values were noted in slurries s. 6 and s. 7 (filtration 429 cm^3/30 min and 491 cm^3/30 min, respectively). The described parameters are listed in Table 2 and in Figure 3.

The analysis of the increasing static structural strength of slurries in the second group of recipes (AMI) and transition time TT revealed that the designed compositions have very good properties hindering gas migration (values are presented in Table 2, and the plot of increasing SSS in Figs. 4 to 7). The values of time TT range between 15 min and 31 min, which is lower than the boundary value, i.e. 60 min[3].

The time of thickening of the analyzed groups of sealing slurries for casing columns 9 5/8" equaled from the 3 hrs for 30 Bc to about 4 hrs for 100 Bc. The differences were observed in slurry s. 8, which was 30 Bc after 1 hrs 40 min, and 100 Bc after 3 hrs 41 min and in slurry s. 9, for which 30 Bc was after 4 hrs, 47 min, and 100 Bc after over 5 hrs. Among slurries belonging to the fifth group the time of thickening ranged between almost 4 hrs for 30 Bc (composition s. 20) to almost 5 hrs for slurry s. 15. The value of 100 Bc was obtained after the time from 4 hrs 33 min to 5 hrs 26 min. A graphical representation of all results is presented in Figure 8.

7. PHYSICO-MECHANICAL PARAMETERS OF HARDENED SLURRIES

The hardened slurry, based on the analyzed recipes, underwent mechanical tests, i.e. compressive test. The remaining physico-mechanical parameters, i.e. adhesiveness to steel casing, permeability to gas after 7 days of hydration, and porosity of samples were defined. On this basis the quality of microstructure of the cement sheath could be determined.

The analysis of mechanical parameters of hardened slurries revealed that the highest compressive strength values were obtained both for 24 hrs and 48 hrs after hydration in well-like conditions for samples representing the second (AMI), third (FINE) and fifth group of slurries (Fig. 9). Such values could be obtained in the group of AMI slurries thanks to the added antimigration polymer, in the presence of which the hydrating cement grains adhered more closely to themselves. The high compressive strength obtained in the fourth group (FINE) was due to the presence of light pozzolana material, which filled the intergranular spaces, and its pozzolana properties could increase the hydration force of the cement grains. High mechanical values in the fifth group of slurries can be a consequence of using drilling cement class G, and also microsilica, and also low water/cement ratio.

---

[3] According to the literature data [3], TT should not exceed 60 min so that gas does not have time to enter the annular space filled with cement slurry

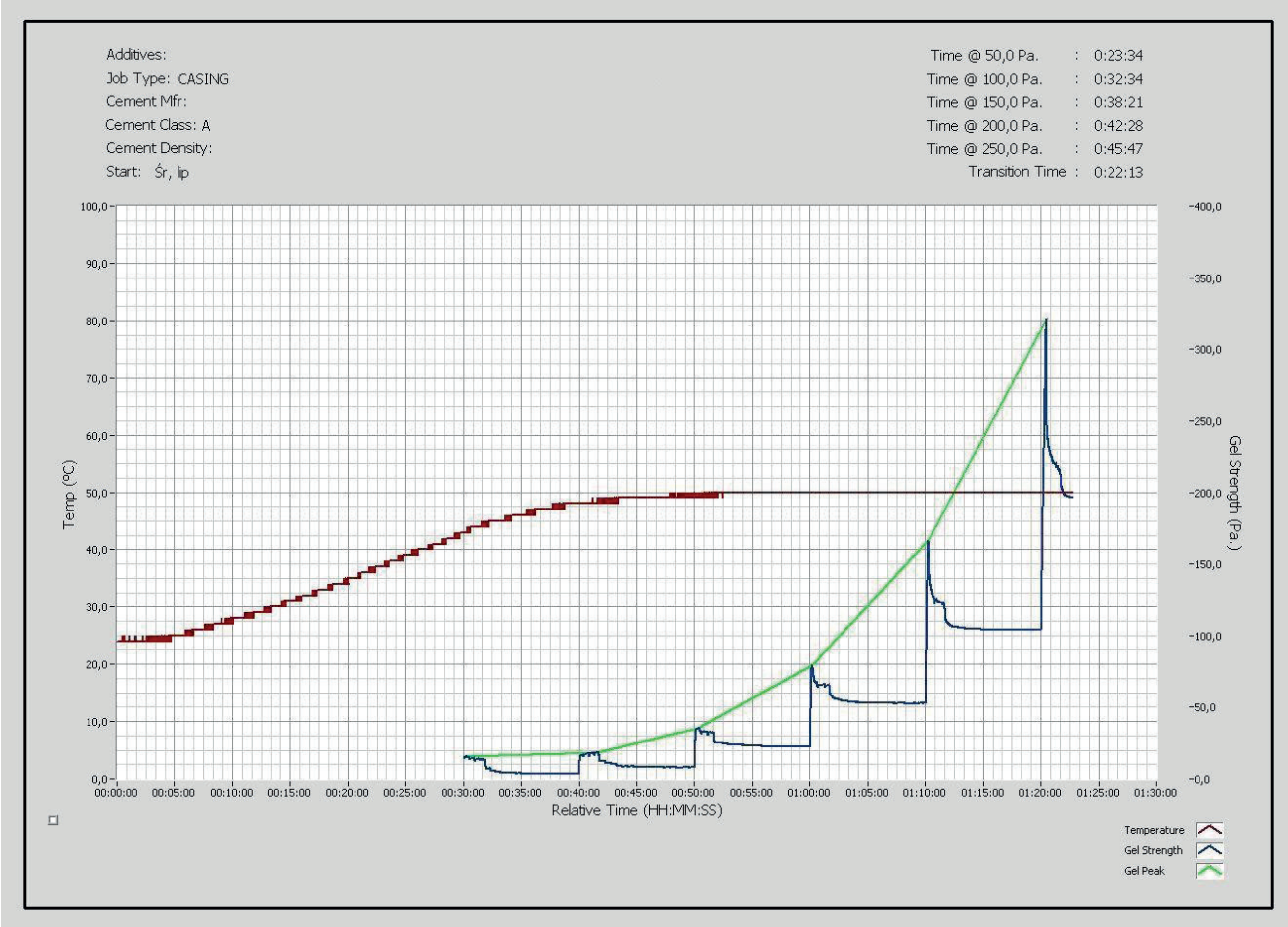


Fig. 4. Increasing static structural strength and transition time for slurry no. 5, to be used for sealing casing column 9 5/8"

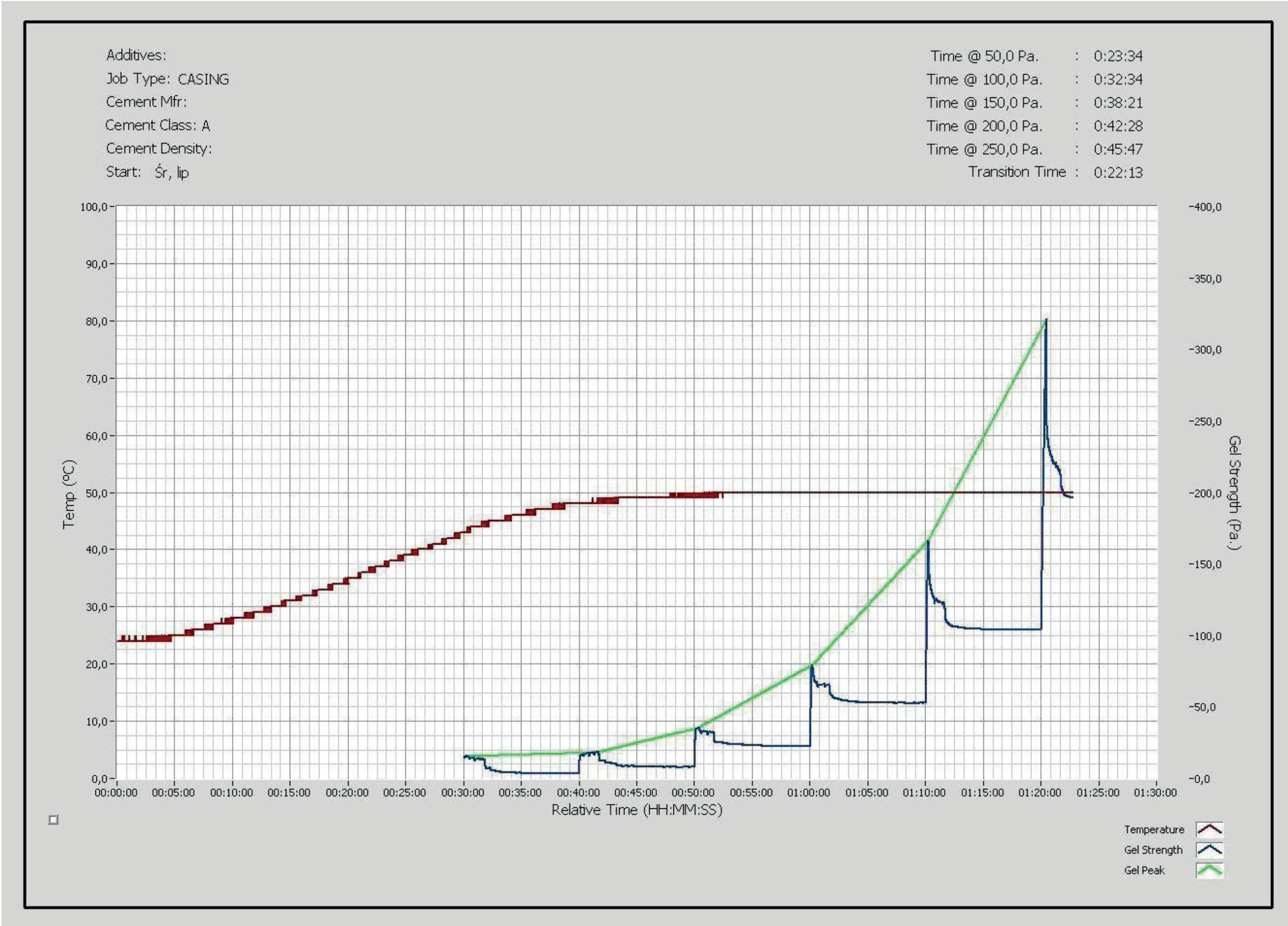


Fig. 5. Increasing static structural strength and transition time for slurry no. 6, to be used for sealing casing column 9 5/8"

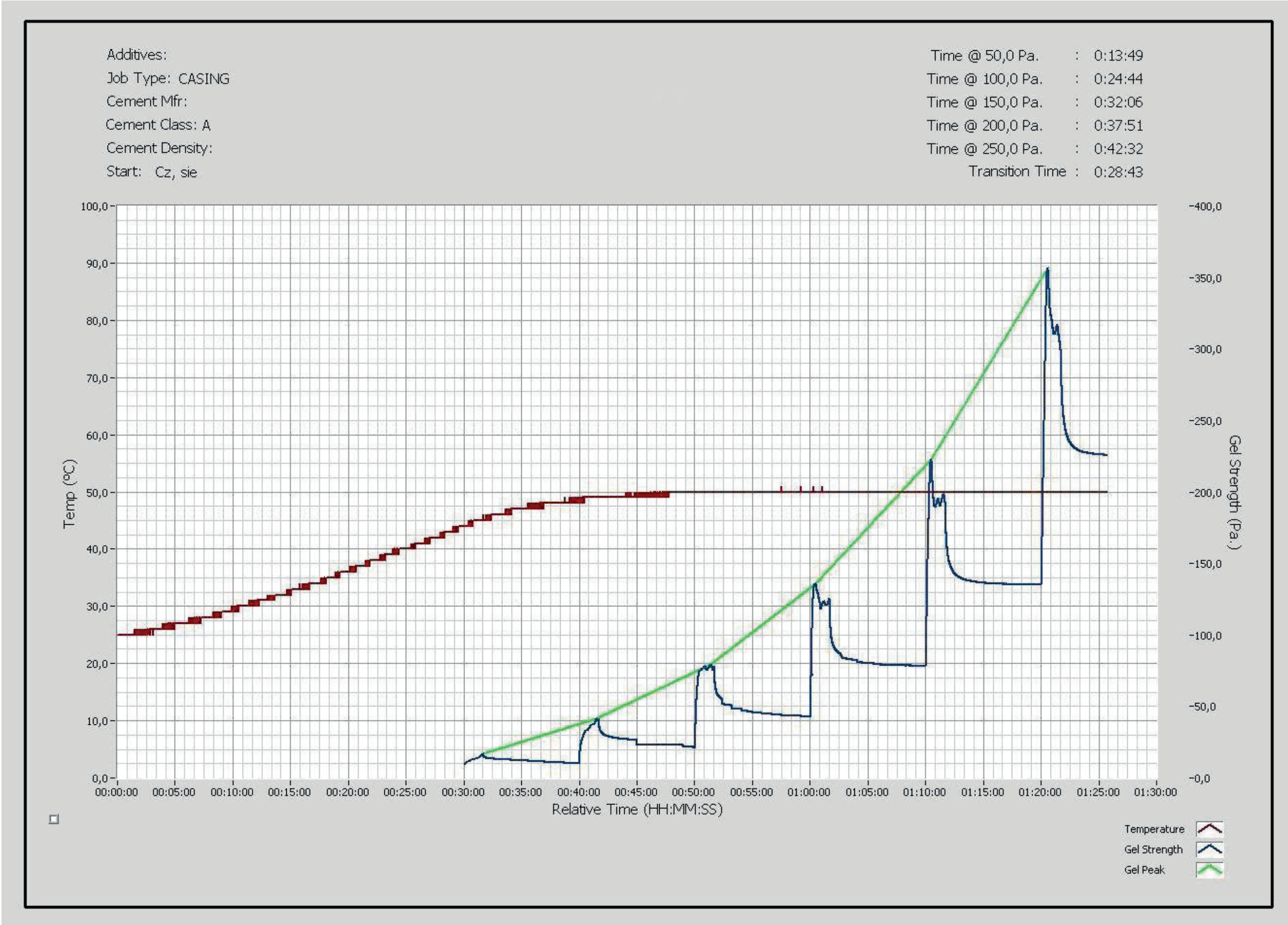


Fig. 6. Increasing static structural strength and transition time for slurry no. 7, to be used for sealing casing column 9 5/8"

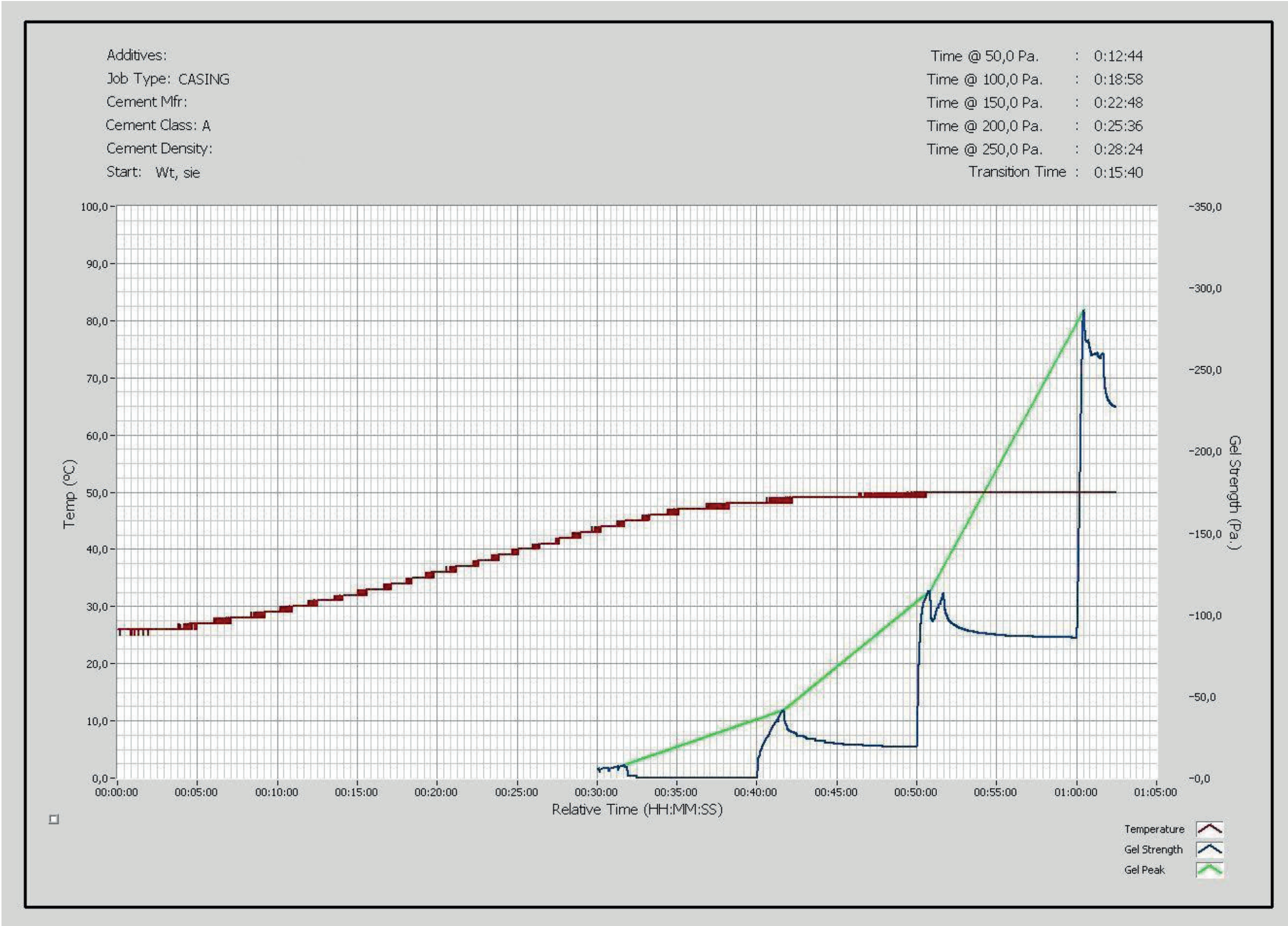


Fig. 7. Increasing static structural strength and transition time for slurry no. 8, to be used for sealing casing column 9 5/8"

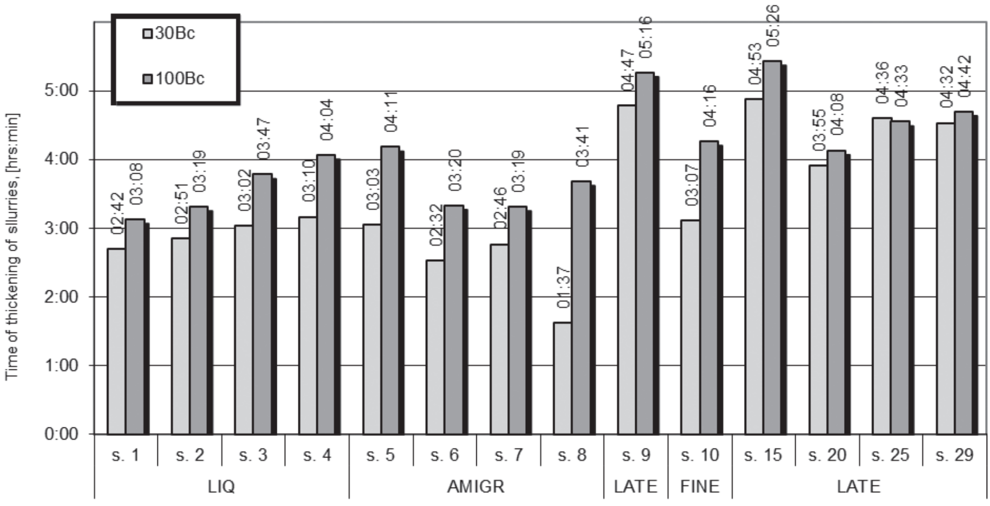


Fig. 8. Time of thickening of analyzed slurries

Another factor is the hydration conditions (pressure 60 MPa to 70 MPa), thanks to which better compaction of cement grains was obtained. The adhesiveness of hardened cement slurry to steel casing was observed to have a similar tendency. The highest values were obtained for samples belonging to the second group (AMI), fourth group (FINE) and fifth group (LATE) – for slurries representing the fifth group. The results of the analyses are presented in Figure 9.

In the course of the analyses of hardened slurry samples, the obtained porosity values ranged from minimum 33.12% to maximum 40.32%. Low porosity values were observed for samples from the third group of slurries, which during previous tests had higher compressive strength values than their counterparts. In the group of hardened slurries worked out for slurries of the fifth group, the lowest values were obtained for samples prepared on the basis of recipes s. 20 and s. 29 (33.18% and 34.12%, respectively). Such low values can be connected with the fact that a very small quantity of water was used for the slurry (w/c = 0.28), and 20% of latex polymer and antimigration agent. The highest porosity (40.32%) obtained for a slurry with a thinning agent (fine grained material), could be caused by the use of considerable quantities of working water (w/c = 0.60), thanks to which much less cement was present in the slurry than in the remaining samples. The discussed results are visualized in Figure 9.

The permeability of slurry samples ranged from 0.023 mD for sample s. 10 where light fine-grained material was added, to 0.075 mD in sample s. 7. Due to the fact that the permeability to gas was determined for samples dried out to solid mass, and that the structure of the cement sheath reveals the presence of bounded water, the obtained values do not create a hazard for the flow of reservoir media.

Along with partial reports, a group of innovative recipes were worked out on the basis of an analysis of slurry parameters and geological-technical conditions. These slurries were planned for sealing casing columns at a depth of 1,800 m to 3,200 m in shale formations. While working out new recipes attention was mainly focused on the technological regimes for these slurries and microstructure of the formed cement sheath. A group of slurry recipes can be successfully used for sealing casing columns in prospecting and production wells for shale gas. Slightly exceeded parameters of slurries can be corrected with appropriate dispersant. The set of recipes in Table 1 constitutes a slurry recipe base, thanks to which the efficiency of sealing of the annular space can be increased.

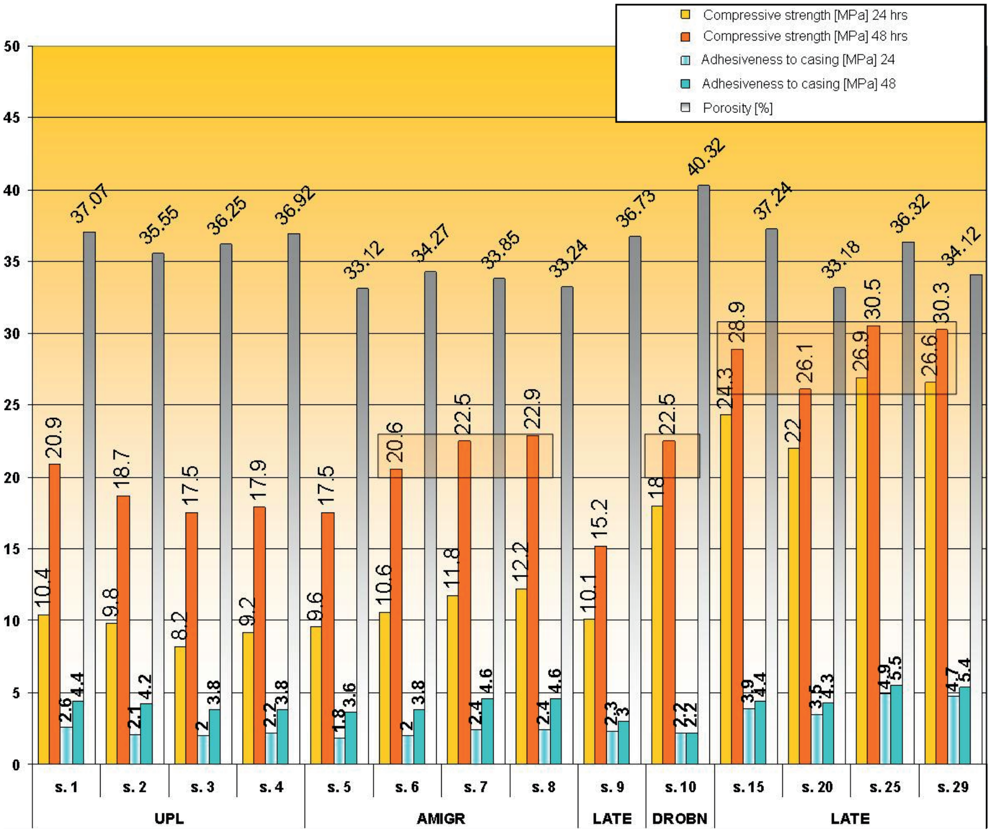


Fig. 9. Parameters of hardened cement slurries based on recipes,
to be used for wells in shale formations

8. CONCLUSIONS

When working out innovative cement slurries to be used for sealing casing in wells in shale formations, the following conclusions were drawn:

- Cement slurries designed for sealing annular space in wells in shale formations reveal good technological parameters deciding about the efficiency of sealing of the casing.
- The percent participation of advantageous to negative slurry parameters (last row in Tab. 2) reveals that a majority of slurries have proper parameters to be used in wellbore conditions.

– For the sake of eliminating possible inaccuracies of the assumed parameters, the analyzed recipes should be modified and re-analyzed prior to using in cementing jobs.
– From among the analyzed recipes, the weakest parameters were observed for slurry s. 4 (as far as recipes of the whole group are concerned), however, during the general interpretation (all compositions), the parameters of this slurry meet the requirements and the slurry can be used in wellbore conditions.
– The use of innovative additives and admixtures contributed to the improvement of technological parameters of fresh and hardened sealing slurries, and so to the higher efficiency of sealing casing columns in shale gas wells.

REFERENCES

[1] Bensted J.: *Cementy wiertnicze* (*Oilwell Cements*). Cement, Wapno, Beton, nr 6, 2002, pp. 249–265.

[2] Bensted J., Smith J.: *Cementy wiertnicze z historycznej perspektywy* (Oilwell Cements. Part 6. An Historic Perspective). Cement, Wapno, Beton, nr 3, 2008.

[3] Dębińska E.: *Wyznaczanie statycznej wytrzymałości strukturalnej i wczesnej wytrzymałości mechanicznej zaczynów cementowych*, Nafta – Gaz, nr 2, 2013, pp. 134–142.

[4] Gawlik P., Szymczak M.: *Migracje gazowe w przestrzeniach międzyrurowych otworów realizowanych na Przedgórzu Karpat*, Nafta – Gaz, nr 7–8, 2006, pp. 349–358.

[5] Kremieniewski M., Rzepka M.: *Badania zaczynów cementowych przeznaczonych do uszczelniania otworów wiertniczych*. Wiadomości Naftowe i Gazownicze, nr 4, 2012, pp. 9–13.

[6] Kremieniewski M., Rzepka M., Dębińska E.: *Statyczna wytrzymałość strukturalna zaczynów przeznaczonych do uszczelniania otworów o podwyższonym ryzyku występowania migracji gazu*. Nafta – Gaz, nr 9, 2014, pp. 608–616.

[7] Kremieniewski M.: *Ograniczenie ekshalacji gazu w otworach wiertniczych poprzez modyfikację receptur oraz kształtowanie się struktury stwardniałych zaczynów uszczelniających*. Prace naukowe INiG – PIB nr 199, 2016.

[8] Kremieniewski M.: *Wpływ dodatku superplastyfikatora na zwiększenie efektywności uszczelniania kolumn rur okładzinowych*. Nafta – Gaz nr nr 3, 2016, pp. 169–176.

[9] Nelson E.B.: *Well Cementing*. Schlumberger Educational Service, Houston, Texas USA, 1990.

[10] Rzepka M., Kremieniewski M., Dębińska E.: *Zaczyny cementowe przeznaczone do uszczelniania eksploatacyjnych kolumn rur okładzinowych na Niżu Polskim*. Nafta – Gaz, nr 8, 2012, pp. 512–522.

[11] Rzepka M., Stryczek S., Kremieniewski M., Wiśniowski R., Dębińska E.: *Recipes of cement slurries for sealing casing in deep wellbores*. AGH Drilling, Oil, Gas, vol. 33, no. 2, 2016, pp. 455–471.

[12] Stryczek S., Gonet A.: *Wymagania odnośnie zaczynów uszczelniających stosowanych w technologiach wiertniczych*. Sympozjum Naukowo-Techniczne „Cementy w budownictwie, robotach wiertniczych i inżynieryjnych oraz hydrotechnice", Piła – Płotki 2001.

[13] Stryczek S., Małolepszy J., Gonet A., Wiśniowski R., Kotwica Ł., *Wpływ dodatków mineralnych na kształtowanie się właściwości technologicznych zaczynów uszczelniających stosowanych w wiertnictwie i geoinżynierii*. Wydawnictwo S.C.M.R., Kraków 2011.
[14] Stryczek S., Wiśniowski R., Gonet A., Złotkowski A.: *The influence of time of rheological parameters of fresh cement slurries*. AGH Drilling, Oil, Gas, vol. 31, no. 1, 2014, pp. 123–133.
[15] Stryczek S., Wiśniowski R., Kotwica Ł., Złotkowski A., Rzepka M., Kremieniewski M., Skrzypaszek K.: *Analysis of technological parameters of cementing slurries for horizontal casing works in Pomeranian Basin*. AGH Drilling, Oil, Gas, vol. 32, no. 2, 2015, pp. 431–442.
[16] Stryczek S., Wiśniowski R., Kremieniewski M., Rzepka M., Kotwica Ł., Złotkowski A.: *Analysis of technological parameters of cement slurries used for sealing casing in the Lublin Basin area*. AGH Drilling, Oil, Gas, vol. 33, no. 1, 2016, pp. 145–159.
[17] Such P.: *Przestrzeń porowa skał łupkowych*. Nafta – Gaz, nr 9, 2012, pp. 561–565.