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ANALYSIS OF TECHNOLOGICAL PARAMETERS OF CEMENTING SLURRIES FOR HORIZONTAL CASING WORKS IN POMERANIAN BASIN****

1. INTRODUCTION

Casing works in directional drilling, especially in case of horizontal wells, which are common in case of drilling in shale formations are one of the most demanding challenges for engineers. Among others, this is due to the fact, that drilling fluids used in horizontal drilling should comply to much more strict requirements, comparing to typical horizontal drilling. Main parameter which should be taken into account during designing and mix-proportioning of slurries for horizontal casing works is the stability of the slurry. Two critical parameters for stability of slurry are free water and sedimentation. For slurries used in casing operations in vertical drilling free water content should not exceed 1.4%, while in case of casing works in bore holes with slope larger than 60^{o}, free water should be eliminated. This requirement is caused by the specific behavior of cementing slurry in horizontal well. The difference in behavior of slurries in wells with various inclination are such an important issue, that for the same slurry free water content can be 1% in case of vertical measurement while in case of measurement with 45^{o} inclination, in can be even about 7%. Additionally, temperature is to be an important parameter influencing free water content of slurries. For example if measurement is conducted in ambient temperature shows 1% of free water, the same slurry tested

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in 80°C would give about 9%. Another important parameter in terms of horizontal drilling is settlement of cementing slurries used in casing operations. Settlement phenomenon is difficult to observe in case of vertical wells, because it takes place on the distance of many meters, while in case of horizontal wells, settlement is a process which takes place on the distance of a few dozens of centimeters – often the diameter of casing well. Stable cementing slurries exhibit constant density thorough whole height of cemented body. Results of all measurements: in top, middle and bottom points should be equal. If the difference between the highest and the lowest measurement of density is more than 60 kg/m^3 it means that the slurry is unstable. Hardened slurry formed from unstable fresh slurry is characterized by non-uniform strength (lower on the top, higher on the bottom) as well as increased porosity in top sections, caused by low density of fresh slurry and thus lower cement concentration. Increased porosity results in higher permeability and thus lowered durability. If sedimentation is accompanied by water loss, than in upper part of horizontal well the released water will be accumulate, and later, after hardening of slurry, it will form a channel which will allow the gas migration through the casing.

2. EXPERIMENTAL

Six cementing slurries were investigated. All investigated slurries were used for casing cementing in horizontal drilling for shale gas in Pomerania Basin. Detailed destination of slurries is described in Table 1. Composition of slurries is presented in Table 2. The mix proportioning was made the way to obtain appropriate properties of fresh slurries. All slurries has got similar flow in the range 250–270 mm. What is especially important, all slurries were design to be extremely stable, due to reasons presented in introduction. From all slurries, settlement was 0. After mixing, slurries were transferred to 20 × 20 × 100 mm moulds (see Fig. 1) and immediately transferred to high pressure curing device. Setting, hardening and maturing were performed in conditions corresponding to that within bore-holes i.e. elevated temperature and pressure. Table 3 gives precise information on the conditions of curing for each slurry. Conditions were based on parameters measured within the well.

Table 1

Destination of slurries investigated

Sample	Well localization	Type of casing
Z1	Opalino 2	7" pipes
Z2	Lubocino 2H	5½" pipes
Z3	Lubocino 1	7" pipes
Z4	Lubocino 3H	7" pipes
Z5	Opalino 3	7" pipes
Z6	Borcz 1	7" pipes

The heat of hardening was measured in the non-isothermal – non-adiabatic microcalorimeter (of our own laboratory construction, from commercially available elements, equipped with computer controlled registration and data refinement) on the pastes produced from 20 g of cement with appropriate amount of additives and admixtures (see Tab. 2). Calorimetric measurement allows to trace the rate of hydration of a given paste measuring the heat evolution of hydrating paste. Calorimetric measurements were done at 20°C. Phase composition of investigated pastes was characterized with the use of XRD and thermal analysis. Samples of appropriate size were crushed with mortar and pestle and dried in dessicator under high vacuum. Next, they were ground with agate mortar and pestle until whole sample passed 63 μm sieve. So prepared samples were subjected to XRD analysis. XRD patterns were collected using Philips PW 1130 apparatus with Cu cathode.

Table 2

Compositions of investigated cementing slurries

Compound	Content [wt. % of cement mass]					
	Z1	Z2	Z3	Z4	Z5	Z6
Cement class G	100	100	100	100	100	100
Silica	–	–	–	–	–	10
Water	47	45	50	52	45	44
Defoamer 1	0.30	0.30	–	–	–	0.50
Dispersant 1	0.30	0.30	–	–	–	–
Fluid loss additive 1	0.50	0.25	–	–	–	0.30
Retarder 1	0.05	0.15	–	–	–	–
Gas Seal®	–	4.00	–	–	–	–
NaCl	–	4.50	–	–	–	4.40
Fluid loss additive 2	–	–	0.70	0.60	0.60	–
Retarder 2	–	–	0.20	0.15	0.20	–
Dispersant 2	–	–	0.30	0.30	0.20	–
Defoamer 2	–	–	0.50	0.50	0.50	–
Defoamer 3	–	–	–	–	–	0.30
Dispersant 3	–	–	–	–	–	0.30
Polymer latex	–	–	–	–	–	10

Beam parameters were 16 mA and 35 kV. Step was 0.05° theta and acquisition time was 3 s/step. Thermal measurements were performed with Netzsch STA 449 F3 Jupiter apparatus. Thermal curves were obtained with the rate of heating equal to 10°C/min in helium. TG and

DTG techniques were used. Mass loss of water due to calcium hydroxide and calcium carbonate was determined using DTG curve and used for calcium hydroxide and calcium carbonate content calculations. Hydrated slurries were observed using FEI Nova NanoSEM 200 scanning electron microscope. Morphology of hydration products was observed in secondary electron mode. Samples were not dried prior to observations.

Table 3

Curing conditions applied for investigated samples

Sample	Well localization	Temperature [°C]	Pressure [MPa]
Z1	Opalino 2	85	35
Z2	Lubocino 2H	85	35
Z3	Lubocino 1	90	42
Z4	Lubocino 3H	80	49
Z5	Opalino 3	80	49
Z6	Borcz 1	80	42

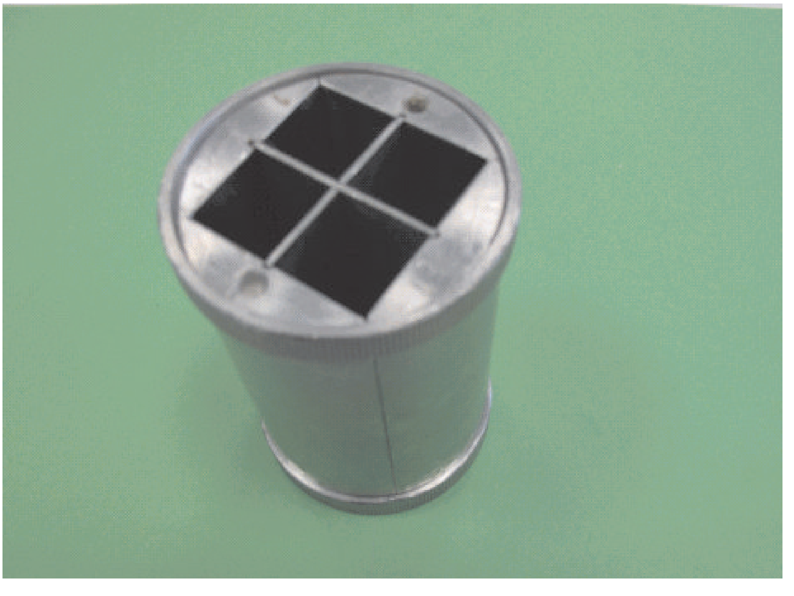

Fig. 1. Mould used for preparation of samples for strength tests

Small pieces of hardened slurries were taken from the bars and placed on the holder, then transferred to the coater and covered with thin carbon layer to avoid charging. Samples were observed in low vacuum mode at 60 Pa water vapour pressure. Microstructure of hardened paste was observed in backscatter mode. Compressive strength of samples after 24 and 48 hours was tested with OFI Ultrasonic Cement Analyzer. Measurements were conducted in environments described in Table 3. Tensile and compressive strength after 2, 7 and 28 days of curing in elevated temperature and pressure were measured on 20×20×100 mm bar samples in hydraulic press CHANDLER Model 4207.

3. RESULTS

3.1. Calorimetry

The results illustrating the rate of heat evolution changes vs. time, calculated per 1g of cement in the mixture, as well as the heat evolved values are plotted in Figures 2 and 3 respectively. Results obtained indicates, that investigated slurries can be divided into two groups. In the first group there are slurries Z1 and Z2 which exhibit lower dynamic of heat evolution comparing to other slurries forming second group, which gather slurries of higher heat evolution dynamics. It suggests that the action of retarder 1 is more effective, comparing to retarder 2. From the point of view of total heat evolved by hydrating slurries, the differences are negligible in case of slurries Z1-Z4 and for that slurries total heat evolved is in the range between 346 and 361 J/g. Slurries Z5 and Z6 exhibit higher total amount of heat evolved (382 and 422 J/g respectively) (Tab. 4).

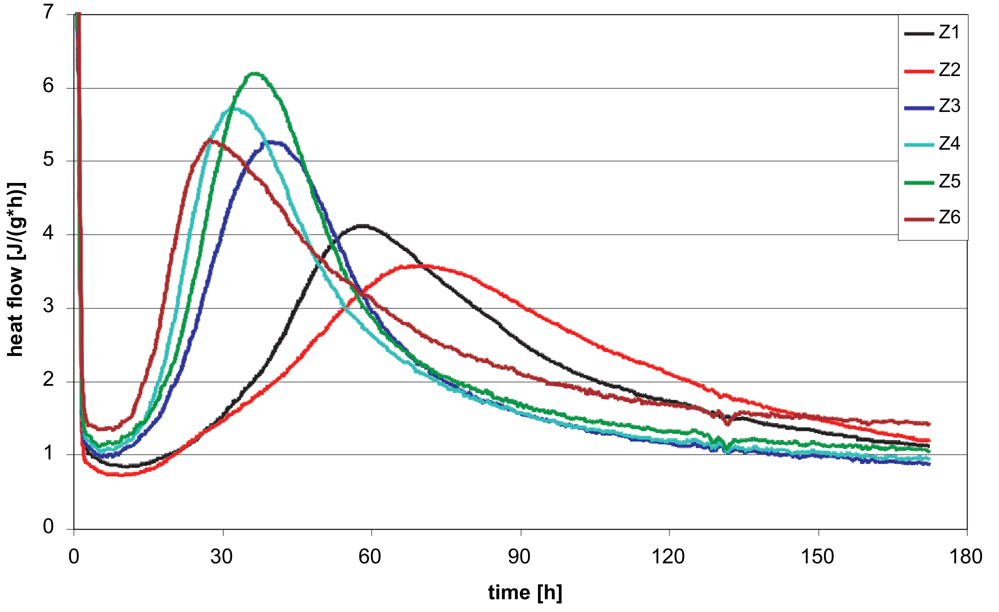

Fig. 2. Rate of heat evolution curves for slurries investigated

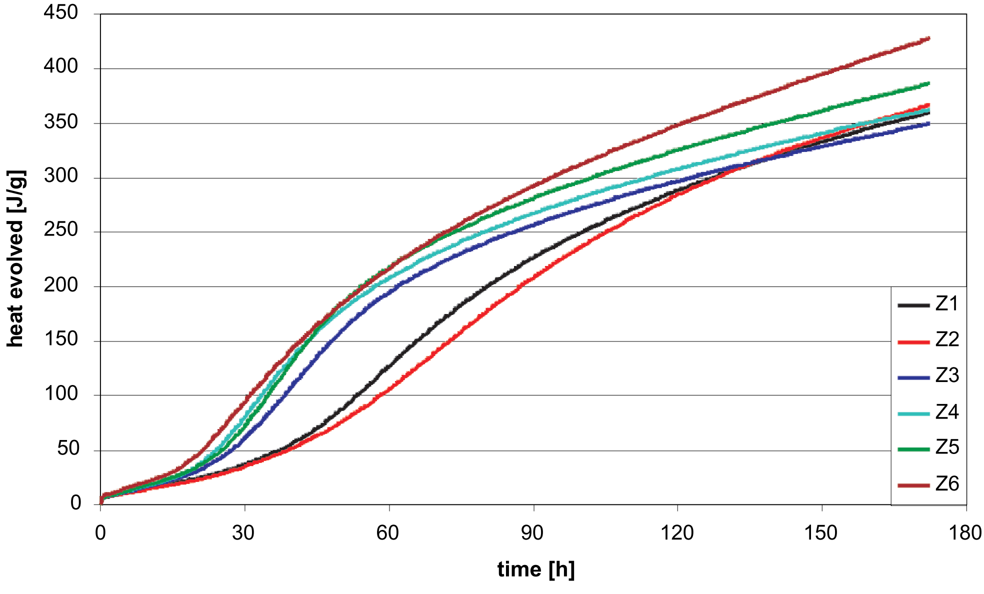

Fig. 3. Cumulative heat evolution curves for slurries investigated

Table 4

Values of total heat evolved after a given periods of hydration by slurries investigated

Sample	Heat evolved after [J/g]						
	24 h	48 h	72 h	96 h	120 h	144 h	168 h
Z1	29	80	173	241	288	325	355
Z2	27	70	148	226	284	328	361
Z3	40	150	225	266	297	323	346
Z4	50	171	236	277	308	335	359
Z5	46	175	247	291	326	355	382
Z6	63	176	251	305	348	386	422

3.2. Mechanical strength

Tables 5 and 6 present values of tensile and compressive strength respectively for slurries cured in elevated temperature and pressure. Obtained results indicate that slurry Z1 shows definitely highest compressive strength values, as well as strength evolution dynamics. Basically, all slurries cured at elevated temperatures exhibit high tensile/compressive strength ratios.

Table 5

Tensile strength of slurries cured in elevated temperature and pressure

Sample	Compressive strength [MPa]		
	1 day	2 days	28 days
Z1	9.0	12.0	12.0
Z2	9.0	12.0	12.0
Z3	12.0	13.5	13.5
Z4	12.0	13.5	13.5
Z5	9.0	12.0	12.0
Z6	9.0	12.0	13.5

Table 6

Compressive strength of slurries cured in elevated temperature and pressure

Sample	Compressive strength [MPa]		
	2 days	7 days	28 days
Z1	34.8	36.5	39.5
Z2	26.1	28.2	30.0
Z3	20.8	23.5	26.0
Z4	17.0	23.5	25.8
Z5	25.9	34.2	34.5
Z6	12.8	31.0	33.5

In addition, test of early strength were performed on slurries. Ultrasonic method was applied on slurries cured in elevated temperature and pressure. Results are presented in Table 7.

Table 7

Compressive strength of slurries cured in elevated temperature and pressure obtained after 24 and 48 hours with the use of ultrasonic method

Sample	Compressive strength [MPa]	
	1 day	2 days
Z1	22.3	26.4
Z2	18.5	24.1
Z3	15.7	18.6
Z4	11.2	15.4
Z5	18.2	23.5
Z6	13.1	15.0

In general results obtained with ultrasonic method are consistent with those obtained with direct, mechanical method. The average difference between measurement of the same slurries with different methods is about 15%.

3.3. Phase composition

Phase analysis performed with the use of XRD and thermal analysis techniques allowed to identify main phases in hydrated cementing slurries. On the basis of XRD following phases were found in all slurries:

- alite (tricalcium silicate) (after 7 and 28 days of hydration),
- C-S-H phase (after 7 and 28 days of hydration),
- portlandite (after 7 and 28 days of hydration),
- iron rich hydrogarnet $Ca_3AlFe(SiO_4)(OH)_8$ (after 7 days of hydration).

Additionally in samples Z2 and Z6 after 7 days of hydration Friedel's salt was found.

Alite presence results from incomplete hydration of cement (alite is main phase compound of class G cement used in experiments). Analysis of peaks intensities showed that the intensity of alite peaks is decreasing between 7th and 28th day of hydration, what indicated, that the rate of hydration in that period is increasing, despite relatively low strength gains in that period. C-S-H phase and portlandite are main cement hydration products. Presence of iron-rich hydrogarnet is connected with elevated temperature of hydration, and on the other hand composition of well cements, containing usually large amounts of aluminoferrite phase (brownmillerite). Friedel's salt present in slurries Z2 and Z6 results from the NaCl presence, as one of slurry compounds.

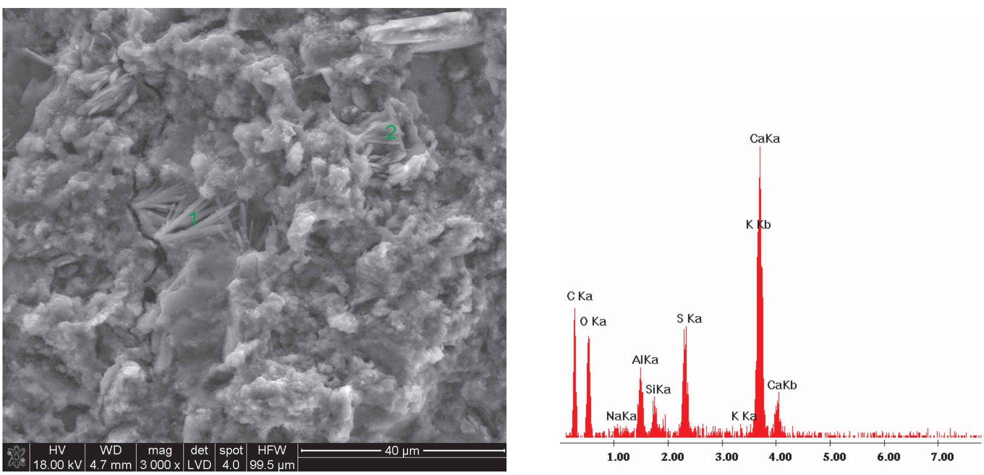

Fig. 4. Ettringite needles visible within C-S-H phase (left).
Chemical analysis of ettringite crystals (point 1), confirms identification

Thermal analysis measurements showed presence of calcium carbonate within hardened slurries. SEM observations revealed the presence of ettringite in investigated samples (see Fig. 4). Its amount was under the limit of detection for XRD.

3.4. Microstructure

SEM observation of fresh fractures revealed presence of typical, compacted microstructure of hardened cement paste both after 7 as well as 28 days. C-S-H phase after 7 days is compacted, with small amounts of fibrous microstructure (see Fig. 5). After both 7 and 28 days massive agglomerates of portlandite can be observed.

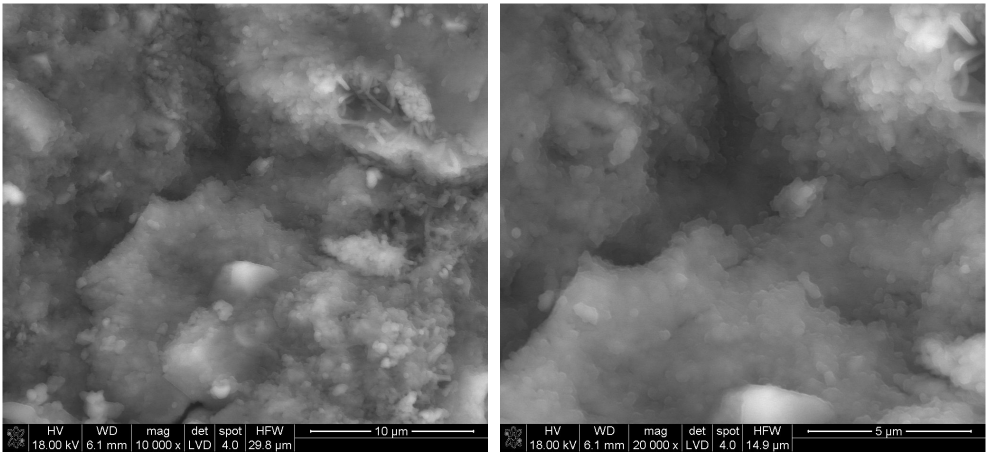

Fig. 5. Microstructure of C-S-H phase. Compacted gel is visible, with small amount of fibers

Backscatter SEM observations were performed on polished sections of hardened slurries and thus allowed to analyze microstructure in more representative way, comparing to SE mode. In that section, samples cured for 28 days in elevated temperature and pressure were compared with samples cured for 7 days in laboratory conditions (20^{o}C/95%RH). Observations showed, that the degree of hydration of cement is higher in case of 28 days samples. In Figure 6 microstructure of both slurries is compared. It can be seen that 7-day old slurry contains much more unreacted cement grains (bright sports – see details in Fig. 7). Especially small grains are absent in case of 28-days old slurry.

The most important conclusion which can be drawn from backscatter SEM observations is that on the microstructure of hardened cement matrix, mainly C-S-H phase. In Figure 8 microstructure of 7 days old and 28 days old samples were compared. Analysis of micrographs (see example in Fig. 8) revealed differences in porosity of C-S-H phase. IN case of samples cured in elevated temperature C-S-H appears to be more porous. The distribution of porosity is more uniform comparing to samples cured in ambient temperature, but still there are more pores in hot cured slurries. This observations are consistent with literature data. Investigations of Galucci et al. showed, that curing temperature strongly influence the properties of C-S-H phase being formed. The increase in temperature causes changes in chemical composition of C-S-H phase. First of all, the amount of bound water is decreasing. It causes the increase in C-S-H density. Increase in density of C-S-H phase results in increase of porosity of hardened cement paste. It influence the macroscopic properties of hardened slurry. It may increase the permeability of hardened slurry towards chloride ingress. Litera-ture data indica-tes, that using mineral admixtures, both pozzolanic (silica fume) as well as hydraulic (ground granulated blast furnace slag) it is possible to improve the microstructure of C-S-H phase cured in high temperatures. Investigations made by Campbell and Detwiler showed that using appro-priate amounts of silica fume as active pozzolana and slag as a hydraulic additive, it is possible to reduce the chloride diffusion coefficient even up to 50 times.

7 days At 20^{o}C/95%RH　　　　28 days at 80^{o}C/42 MPa

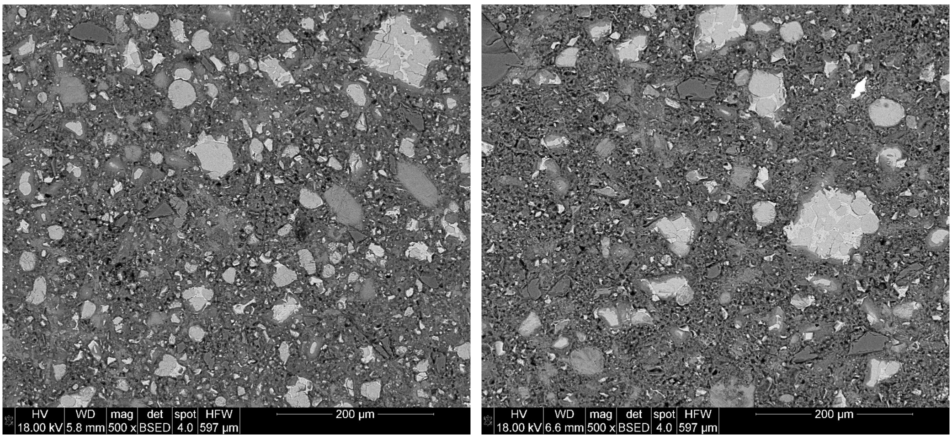

Fig. 6. Microstructure of hardened slurries cured for 7 days at 20^{o}C (left) and 28 days in 80^{o}C (right)

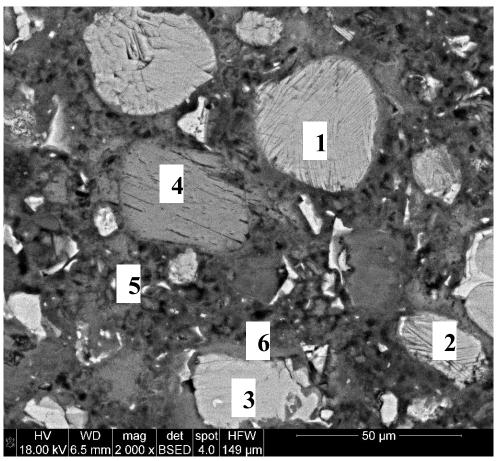

Fig. 7. Details of hardened slurry microstructure: 1, 2 – unreacted belite grains, 3 – unreacted alite grain, 4 – portlandite, 5 – C-S-H phase – outer product, 6 – C-S-H phase – internal product

7 days At 20°C/95%RH 28 days at 80°C/42 MPa

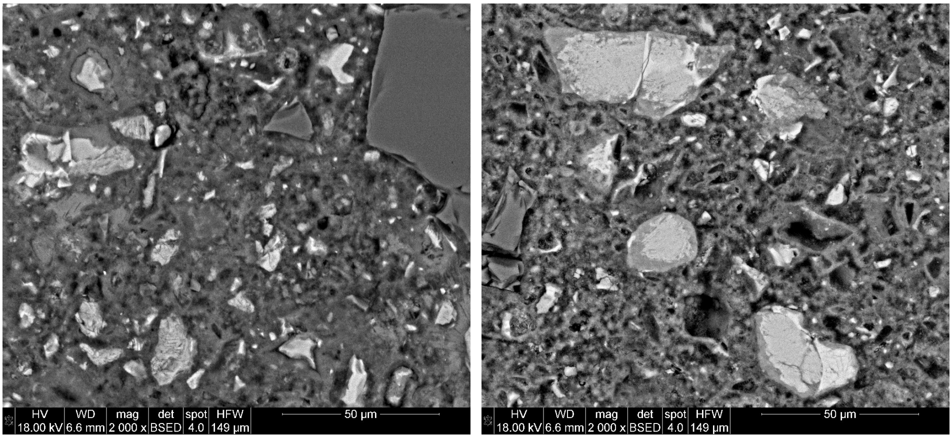

Fig. 8. Microstructure of hardened slurries cured for 7 days at 20°C (left) and 28 days in 80°C (right)

4. CONCLUSIONS

1. Investigated slurries allowed to successfully complete casing operations in horizontal wells
2. Slurries Z1 and Z2 exhibit lowest heat evolution rate among all investigated slurries
3. Slurries prepared with the use of NaCl after 7 days of hydration contain Friedel's salt, which later disintegrates
4. Slurries cured at elevated temperatures exhibit higher porosity of C-S-H phase and thus, hardened cement paste. It may result in increased permeability

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