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Analysis of the Influence of Technological Factors on Free Water Separation During Production of Oil-Well Cement in Industrial Conditions

ABSTRACT

This article present the results of production-scale studies conducted on cement tube mills with ball loading in open and closed cycles, which produced oil-well cement without additives (PCTI-100). The article analyzes the factors that affect the properties of the finished product, and especially the water separation of the cement obtained during grinding. For this purpose, correlation coefficients were obtained from processed experimental data. Analysis of the correlation coefficients shows that in order to achieve the normalized water separation index, it is necessary to adhere for the corresponding values of specific surface area of the cement, gypsum stone and hemihydrate gypsum. The analysis identified factors with a significant impact on free water separation. The correlation analysis revealed that hemihydrate gypsum content exhibits a strong negative correlation with water separation (r = -0.85), particularly in open-cycle grinding. This suggests that a higher hemihydrate content may facilitate faster hydration reactions, reducing free water. Meanwhile, the specific surface area showed a consistent negative correlation with water separation across both grinding modes, underscoring the importance of fineness in controlling bleeding. Regression equations for predicting free water separation were developed.

Keywords: *residue on a 45 μm sieve, hemihydrate gypsum, anhydrite, water separation, specific surface area of cement.*

1. INTRODUCTION

The first type of oil-well cement (PCTI-100) is characterized by absence of additives and consists only of clinker and gypsum [1]. Water separation is one of the main indicators of the quality of first type oil-well cement, since free water can escape during casing installation in a well, and form a path through which gas can move. It is recommended that cement paste placed in the well shows no signs of water separation [2]. In accordance with integrated standard for requirements for oil well cement, water separation should not exceed 8.7 ml. Reducing water separation is an urgent technological problem; its solution would improve the quality of oil well cement pastes. Existing studies on free water separation have mainly focused on cement with additives.

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It shows that in order to reduce the water separation of oil-well cement, it is advisable to introduce additives, which are usually wastes from the chemical industry [3], as well as polyfunctional additives [4], metakaolin and zeolite [5], ash mixtures [6], manganese ore, blast furnace slag, organic resin [7], polypropylene fibers [8], and cellulose-based polymers [9]. However, introducing additives during cement grinding or post-production presents several risks. One of them is the instability of the chemical composition of the additives. If additives are introduced during grinding of clinker to reduce water separation, there are many factors that can negatively affect other, but no less important, properties of cement paste. This makes it difficult to predict their impact on cement properties.

Laboratory studies are not fully representative of the grinding process, as they not account for the full range of that affect during grinding in a production by cement mill. One of the significant differences is the temperature inside of the cement mill. While expected mill temperatures are around 70-80 °C, during grinding in tube mills can generate localized heat, creating a high probability of gypsum phase transition ($CaSO_4 \cdot 2H_2O$) and the formation of hemihydrate ($CaSO_4 \cdot 0{,}5H_2O$) at the temperature 100-150 °C and anhydrite ($CaSO_4$) at the temperature over 150 °C. In previous works [10], the effect of dihydrate and hemihydrate gypsum was studied in laboratory conditions, however, the specifics of industrial grinding require clarification.

The current regulatory documents in Ukraine DSTU B V.2.7-88-99 [1], API SPEC 10A:2019+ADD1:2019 [11] and technical literature [2] do not regulate cement fineness or its influence on the water separation index.

This work shows that it is possible to reduce the water separation of cement paste obtained by grinding in both open and closed cycles by achieving a sufficient fineness of grinding, characterized by both the appropriate residue on a sieve with a hole size of 45 μm and the specific surface area. The aim of this study is to analyze the individual and combined effects of the main technological factors on the water separation of cement paste obtained in both open and closed cycles and propose a calculation method for its prediction and minimization.

2. MATERIALS AND METHODS

The clinker used in this study had the following mineralogical composition (%): C_3S – 54.15; C_2S – 26.82; C_3A – 2.18; C_4AF – 11.71. Gypsum stone, with a dihydrate content of 94.2%, was used. Grinding was performed on cement ball mills 3.2x15 m (Fig. 1). During grinding in a closed cycle, the mill was equipped with an air separator IV-th generation TSV DN3200 (Fig. 2). The loading coefficient of two chambers of the cement mills was the same. During cement grinding in a closed cycle, the grinding fineness was regulated by changing the speed of the separator turbine. Spreading and water separation were determined according to DSTU B V.2.7-86-99 [12] (Fig. 3 and Fig. 4). Correlation coefficients of the influence of the studied factors and the linear regression equations were obtained using Microsoft Excel.

Figure 1. Cement tube mill 3,2x15 m

Figure 2. Air separator TSV DN3200 used in closed-cycle cement grinding

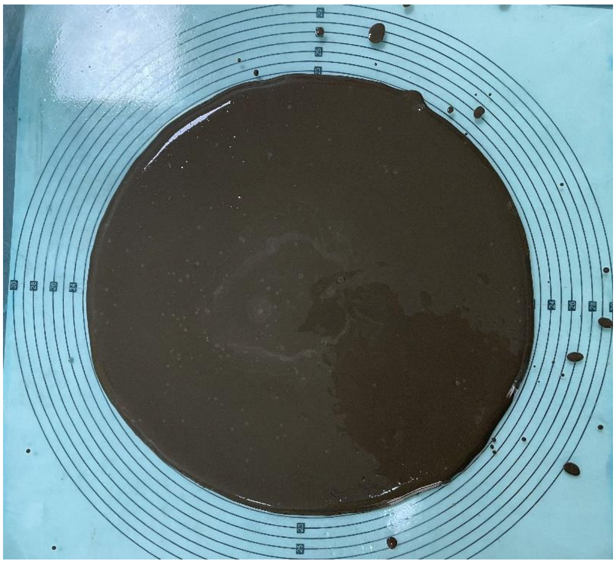

Figure 3. Determination of spreading

Figure 4. Determination of water separation

3. RESULTS AND DISCUSSIONS

The results of studies of the influence of grinding parameters on water separation in open cycle are given in table 1. The higher water separation is observed at the lowest specific surface area of cement. At a specific surface area of cement of about 3100 Blaine, the water separation is 7.0 ml, while at a specific surface area of cement of 3700 Blaine, the water separation reaches only 4.0 ml.

Statistical processing of the open-cycle production results showed that several parameters had correlation coefficients with an absolute value greater than 0.7. This indicates that parameters with a strong negative correlation ($r < -0.7$) are associated with a significant decrease in free water separation. Correlation analysis performed shows a significant interaction between the content of hemihydrate gypsum, residue on a 45 μm sieve, specific surface area and the total SO_3 content by assessing their influence on water separation, which is visible in table 2 and fig. 5.

Figure 5 graphically shows the quantity of the correlation coefficients. Statistically significant coefficients ($|r| > 0.7$) are highlighted.

Table 1. Results of a production experiment on an open-cycle cement mill

Number of cement samples	Spreading, mm (X_1)	Specific surface area, cm^2/g (X_2)	Residue on a 45 μm sieve, % (X_3)	SO_3 content, % (X_4)	Mineralogical composition of clinker			Gypsum,% (X_8)	Hemihydrate, % (X_9)	Anhydrite, % (X_{10})	Free water separation, mL (Y)
					C_3S, % (X_5)	C_2S, % (X_6)	C_3A, % (X_7)				
1	190	3642	20.03	3.69	51.14	29.13	1.61	0.04	3.36	0.16	4.0
2	195	3723	16.99	3.38	54.92	27.46	2.22	0.04	2.70	0.06	4.0
3	210	3306	14.90	2.96	54.83	27.27	1.50	0.04	2.65	0.06	5.0
4	210	3233	14.35	3.01	54.20	28.47	2.21	0.04	2.42	0.11	6.0
5	210	3019	14.31	2.86	53.77	28.81	1.63	0.04	2.26	0.13	7.0
6	190	3132	14.67	2.89	54.56	27.38	1.73	0.04	1.82	0.15	7.5
7	200	3293	14.12	3.04	51.42	29.85	1.44	0.04	2.34	0.07	6.0
8	210	3337	15.82	3.32	54.37	27.18	1.50	0.04	3.04	0.10	5.0
9	195	3427	12.41	3.41	51.94	29.06	1.44	0.09	2.98	0.20	5.5

Table 2. Correlation coefficients of the influence of the studied parameters on water separation during open cycle grinding

	Spreading, mm	Specific surface area, cm2/g	Residue on a 45 μm sieve, %	SO3 content, %	C_3S, %	C_2S, %	C_3A, %	Gypsum, %	Hemihydrate, %	Anhydrite, %
Free water separation, mL (Y)	0.14	-0.91	-0.64	-0.81	0.15	0.09	-0.12	-0.02	-0.85	0.25

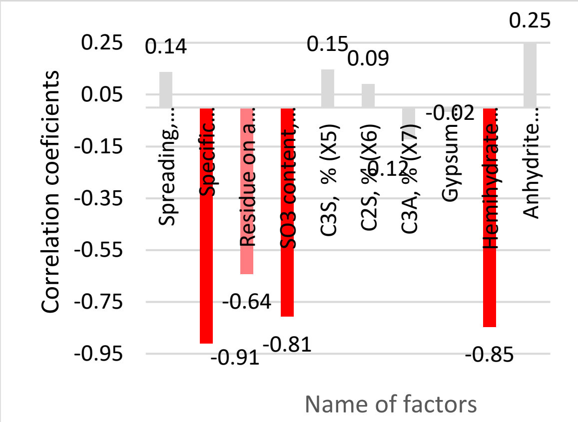

Figure 5. Histogram of correlation coefficients of the influence of the studied parameters on water separation obtained during open-cycle grinding

The other parameters are not strongly correlated and could be excluded from the calculation. The decrease in water separation with higher hemihydrate content is attributed to its effect on water demand. As hemihydrate is highly reactive and undergoes rapid hydration, it binds a significant amount of mixing water, which has a positive effect on water separation.

Using the experimental results a regression model of water separation on an open cycle was obtained (Eq. 1)

$$Y=18{,}16-0{,}0053X_2-0{,}075X_3+3{,}815X_4-2{,}28X_9 \quad (1)$$

According to this calculation the R^2=0.995, adjusted R^2=0.99, RSE=0.12, significance F= 6.56316E-05 and p-values for each parameter are at the table 3. The R^2 and adjusted R^2 values are close to 1.0, indicating that the regression model is a good fit, RSE is low which mean that suggesting the model's predictions are generally close to the actual data points. From table 3 it is clear that all of p-values are less than 0,05, indicating that all parameters are statistically significant. The Variance Inflation Factor (VIF) scores in Table 3 are all below 5, indicating moderate multicollinearity that is unlikely to severely bias the model.

Table 3. P-values and VIF scores of the main parameters at open cycle

	P-value	VIF score
Intercept	1,31956E-05	
Specific surface area, cm^2/g (X_2)	0,000164492	4,36
Residue on a 45 μm sieve, % (X_3)	0,039285109	1,71
SO_3 content, % (X_4)	0,001089606	4,81
Hemihydrate, % (X_9)	0,000316278	4,92

Taking into account all the studied parameters in open cycle, the regression coefficients are arranged in descending order (Eq. 2).

$$X_4 > X_2 > X_3 > X_9 \quad (2)$$

During cement mixing, hemihydrate rapidly rehydrates to form dihydrate gypsum. Dihydrate gypsum reacts with C_3A and forms calcium hydrosulfoaluminate, which slows down hydration, which affects the setting time of cement. When the ratio C_3S to C_3A decreases, a drop in water separation is observed.

The results of studies of the influence of grinding parameters on water separation in close cycle are given in table 4.

Statistical processing of the closed-cycle production results showed that several parameters had correlation coefficients with an absolute value greater than 0.7. This indicates that parameters with a strong negative correlation ($r < -0.7$) are associated with a significant decrease in free water separation and strong positive correlation ($r > 0.7$) are decreasing water separation index.

Performed correlation analysis of the process in close cycle shows a significant interaction between the total SO_3 content, specific surface area and spreading of cement paste by assessing their influence on water separation, which is visible in table 5 and fig. 6.

Table 4. Results of a production experiment on a closed-cycle cement mill

Number of cement samples	Spreading, mm (X_1)	Specific surface area, cm²/g (X_2)	Residue on a 45 µm sieve, % (X_3)	SO_3 content, % (X_4)	Mineralogical composition of clinker			Gypsum, % (X_8)	Hemihydrate, % (X_9)	Anhydrite, % (X_{10})	Free water separation, mL (Y)
					C_3S, % (X_5)	C_2S, % (X_6)	C_3A, % (X_7)				
1	220	3992	3.69	3.91	52.96	25.65	2.35	5.47	0.53	0.00	3.0
2	250	3706	4.08	3.31	53.00	27.20	2.50	3.58	0.59	0.00	5.0
3	250	3245	13.11	2.69	56.88	25.59	1.84	0.35	1.50	0.13	7.5
4	250	3455	12.15	3.38	54.01	28.01	1.88	0.47	2.46	0.01	7.0
5	235	3902	10.26	3.35	52.01	29.63	1.57	0.06	2.31	0.13	2.0
6	220	3486	8.49	3.41	48.85	31.22	2.10	0.49	3.06	0.00	5.0
7	230	3619	3.90	3.45	50.87	30.34	2.51	2.64	1.57	0.06	5.5
8	240	3389	3.11	3.30	53.16	27.44	2.22	2.01	1.60	0.05	7.0
9	245	3396	4.01	2.92	50.61	30.43	2.40	2.36	1.50	0.00	7.5
10	260	2829	4.89	1.53	54.74	29.37	1.69	0.09	0.84	0.02	9.0
11	195	4578	4.00	4.11	53.36	27.56	2.39	0.58	3.06	0.15	2.0

Table 5. Correlation coefficients of the influence of the studied parameters on the water separation of cement obtained by closed-cycle grinding

	Spreading, mm	Specific surface area, cm²/g	Residue on a 45 µm sieve, %	SO_3 content, %	C_3S, %	C_2S, %	C_3A, %	Gypsum, %	Hemihydrate, %	Anhydrite, %
Free water separation, mL (Y)	0.78	-0.91	0.1	-0.78	0.3	0.1	-0.19	-0.21	-0.32	-0.4

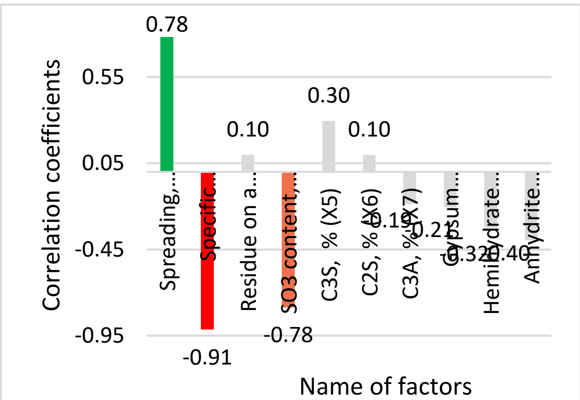

Figure 6. Histogram of correlation coefficients of the influence of the studied parameters on the water separation of cement obtained by closed-cycle grinding

Figure 6 graphically shows the quantity of the correlation coefficients. Statistically significant coefficients (|r| > 0.7) are highlighted.

The other parameters are not strongly correlated and could be excluded from the calculation.

During evaluating model reliability of closed cycle, the high level of p-value of parameters of spreading and total SO_3 content can be seen (table 6). These parameters are not statistically significant. In table 6 there are VIF scores coefficients which shows that all parameters have value close to 5 and moderate multicollinearity, which may not be severe.

Table 6. P-values and VIF scores of the main parameters at closed cycle

	P-value	VIF score
Intercept	0,1522	
Spreading, mm (X_1)	0,9225	3,61
Specific surface area, cm^2/g (X_2)	0,0398	4,86
SO_3 content, % (X_4)	0,8818	4,2

The increased number of particles smaller than 45 microns significantly increases the influence of grinding fineness compared to other parameters, which allows them to be excluded from the regression equation.

After excluded total SO_3 content and spreading of cement paste the p-values can be seen at table 7. The R^2= 0.836, adjusted R^2= 0.818, RSE=1.00, significance F= 8.09287E-05. The R^2 and adjusted R^2 values are close to 1.0, indicating that the regression model is a good fit, RSE is low which mean that suggesting the model's predictions are generally close to the actual data points.

Table 7. P-values of the main parameters at closed cycle after excluded spreading and SO_3 content

	P-value
Intercept	9,48839E-06
Specific surface area, cm^2/g (X_2)	8,09287E-05

Using the experimental results a regression model of water separation for close cycle was obtained (Eq. 3).

$$Y= 22,68 -0,00478X_2 \quad (3)$$

In both equations (2 and 3) there are low coefficients for specific surface area because the number of X_2 is represented in thousands.

Comparing open and closed cycles there is a difference between the temperature inside of the mill and therefore the phase transition of dihydrate gypsum to hemihydrate. In regression model of closed cycle hemihydrate gypsum doesn’t have impact to the water separation because not full transition of gypsum. During cement grinding in a closed cycle, a significant increase in specific surface area is achieved (compared with an open cycle) and it becomes the only factor that can be taken into account in the regression model of water separation.

The results of grinding in open and close cycles method shows that residue on a 45 μm sieve and specific surface area are not strongly correlated. The specific surface area of cement inherently related to the mean size of cement grains [13] which can fluctuate during grinding process in the production cement mills. The high value of the regression coefficient for hemihydrate gypsum (Fig. 7) is noteworthy. Figure 7 shows the XRD diffractogram of the cement, with a peak corresponding to hemihydrate gypsum indicated by an arrow.

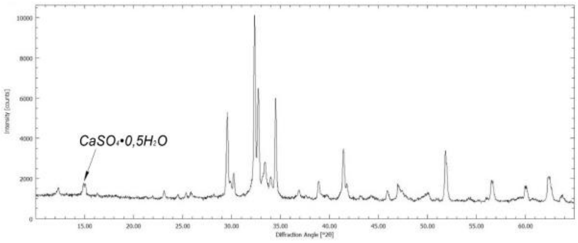

Figure 7. XRD diffractogram of cement with hemihydrate gypsum

However, notice its small amount in cement, the effect on water separation is relatively low. During mixing cement with water, hemihydrate gypsum turns into dihydrate, which reduces the total water content and has a positive effect on reducing water separation. There is also a strong direct correlation with water separation in the amount of cement paste spreading, which is logical, because it is related to the consumption of water for mixing.

The regression equation enables prediction and adjustment of water separation index of cement obtained during grinding in order to achieve it in a minimum value.

Considering the production method and mineralogy of the clinker, the water separation index can be adjusted by the fineness of grinding and gypsum content.

Regression equations for water separation can be considered as calculation formulas for predicting and regulating water separation, taking into account a set of basic technological parameters.

To test the effectiveness of the equations, the additional production experiments were conducted to compare the experimental results of determining the water separation under the considered fluctuations of the technological parameters shows at table 8 and table 9 for open and close cycles. These regression equations give the practical instrument for decreasing free water separation during the operational process. At the open cycle average deviation is 9,3% and at the close cycle average deviation is 14,9%. Comparisons of measured and predicted results show that in an open cycle of production Root Mean Square Error RMSE=0.56, and in closed cycle RMSE=1.03.

Table 8. Comparing results of production experiment on an open-cycle cement mill with calculated water separation

Number of cement samples	Spreading, mm (X_1)	Specific surface area, cm^2/g (X_2)	Residue on a 45 μm sieve, % (X_3)	SO_3 content, % (X_4)	Mineralogical composition of clinker			Gypsum, % (X_8)	Hemihydrate, % (X_9)	Anhydrite, % (X_{10})	Free water separation, mL (Y)	Calculated free water separation, mL	Prediction error of free water separation, %
					C_3S, % (X_5)	C_2S, % (X_6)	C_3A, % (X_7)						
1	210	3021	11,48	2,89	55,56	26,43	1,66	0,04	2,48	0,27	6	6,8	13,1
2	210	2985	15,03	3,06	52,02	29,1	1,42	0,04	2,76	0,16	6	6,7	12,0
3	220	3163	17,32	3,3	53,12	28,04	1,95	0,04	3,4	0,14	5	5,1	1,4
4	210	3231	16,58	3,17	51,96	28,45	2,18	0,04	3,7	0,3	4,5	3,6	20,3
5	220	2996	14,87	3,1	53,53	27,6	1,66	0,04	3,09	0,06	5,5	6,1	10,4
6	210	3118	14,49	3,06	52,01	28,41	1,93	0,04	3,45	0,2	5	4,5	10,2
7	210	2990	15,24	3	52,95	27,66	1,89	0,04	3	0,16	6	5,9	1,7
8	200	3130	13,71	3,04	54,66	26,13	2,39	0,04	3,43	0,11	5	4,5	11,0
9	210	3395	15,48	3,17	53,87	27,62	1,69	0,04	2,81	0,22	5	4,8	3,3

Table 9. Comparing results of production experiment on a close-cycle cement mill with calculated water separation

Number of cement samples	Spreading, mm (X_1)	Specific surface area, cm^2/g (X_2)	Residue on a 45 μm sieve, % (X_3)	SO_3 content, % (X_4)	Mineralogical composition of clinker			Gypsum, % (X_8)	Hemihydrate, % (X_9)	Anhydrite, % (X_{10})	Free water separation, mL (Y)	Calculated free water separation, mL	Prediction error of free water separation, %
					C_3S, % (X_5)	C_2S, % (X_6)	C_3A, % (X_7)						
1	230	3896	3,03	4,09	51,44	27,55	2,11	4,68	1,14	0,15	4	4,1	4,6
2	250	3807	2,89	3,43	50,66	29,43	1,58	2,83	1,13	0,17	3	4,5	8,4
3	230	3806	2,77	3,86	50,56	29,49	2,05	3,05	1,83	0,03	4	4,5	14,6
4	235	3935	2,86	3,25	52,26	28,87	2,67	2,21	1,3	0,01	4	3,9	3,2
5	220	3831	2,14	3,63	55,91	25,01	2,27	2,13	1,8	0,06	5,5	4,4	20,3
6	230	4042	1,22	3,93	55,44	25,57	2,2	1,31	2,48	0,03	6	3,4	42,6
7	230	3895	2,18	3,23	57,03	24,62	3,01	2,28	1,34	0,09	4	4,1	1,1
8	240	3871	2,65	3,06	57,13	24,47	2,74	0,1	2,5	0,2	4	4,2	4,2
9	230	3862	2,34	3,12	56,58	24,76	3,05	0,27	2,44	0,12	5	4,2	16,3
10	220	4128	2,31	3,52	57,46	24,69	2,7	0,05	2,67	0,1	4,5	3,0	35,1
11	220	3771	2,47	3,1	57,07	24,66	2,55	0,6	2,48	0,03	4	4,7	14,7

From the closed cycle experiments it can be argued that hemihydrate gypsum work for decreasing water separation. But this parameter is not included for the regression model because of low amount of hemihydrate gypsum at the cement as a result of not high temperature of cement.

Analysis of the laboratory experiment in order to achieve the same parameters (different specific surface area) as in production (table 10) shows that higher finesses of cement decreases water separation from 4 ml to 1,6 ml.

Table 10. Results of laboratory tests

Specific surface area, cm²/g	Free water separation, mL	Spreading, mm	Residue on a 45 µm sieve, %	SO_3 content, %	C_3S, %	C_2S, %	C_3A, %	Gypsum, %	Hemihydrate, %	Anhydrite, %	Flexural streght in 1 day, MPa
3020	4,0	200	9,28	3,4	53,77	28,81	1,17	3,19	0,53	0,03	5,30
4406	1,6	200	4,38	3,36	56,88	29,63	1,26	3,21	0,62	0,41	7,31

At the same time flexural strength (Fig. 8) at the age of 1 day is higher for 27 % with higher finesses which is important value for the oil-well cement too.

Figure 8. Determining of flexural strength

4. CONCLUSION

1. Maintaining a stable mineralogical composition of clinker and other components, as well asconsistent grinding parameters, is essential for industrial production of oil-well cement.
2. Cement fineness has the greatest impact on the water separation. The SO_3 content and hemihydrate gypsum concentration also significantly influence the water separation index.
3. Application of the regression models reduced the free water separation index by 1 mL (19%) in the open cycle (from 5.5 to 4.5). In closed cycle it was reduced for 0,3 mL (4%) (from 5.5 to 5.3). The process was stabilized in both cycles. The average deviation (AD) for the open cycle was 9.3%, compared to 15.0% for the closed cycle. During the experiments, production cement mills with in-stream cement sampling were used. This may be a methodological problem as some cement analyses from table 9 have large deviations. During the analyses, the parameters of thickening time at a consistency of 100 Bc were not investigated due to the lack of appropriate equipment.
4. The obtained regression equations can be used as calculation formulas for predicting water separation, which will allow regulating the water separation index in production conditions. The conducted production research demonstrates that regression analysis can be used to predict the influence of key factors, allowing for process adjustments to achieve and maintain the normalized water separation value at a minimum level.
5. Along with water separation, the quality of oil-well cement pastes is affected by a number of other properties, which are also influenced by the characteristics of grinding in open and closed cycles. The characterization of the influence of grain composition of oil-well cement according to the density and integral particle distribution requires additional research.

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IZVOD

ANALIZA UTICAJA TEHNOLOŠKIH FAKTORA NA SEPARACIJU SLOBODNE VODE TOKOM PROIZVODNJE CEMENTA ZA NAFTNE BUŠOTINE U INDUSTRIJSKIM USLOVIMA

Ovaj članak predstavlja rezultate studija u proizvodnom obimu sprovedenih na mlinovima za cementne cevi sa punjenjem kuglica u otvorenom i zatvorenom ciklusu, koji su proizvodili cement za naftne bušotine bez aditiva (PCTI-100). Članak analizira faktore koji utiču na svojstva gotovog proizvoda, a posebno na odvajanje vode iz cementa dobijenog tokom mlevenja. U tu svrhu, koeficijenti korelacije su dobijeni iz obrađenih eksperimentalnih podataka. Analiza koeficijenata korelacije pokazuje da je, da bi se postigao normalizovani indeks odvajanja vode, potrebno pridržavati se odgovarajućih vrednosti specifične površine cementa, gipsanog kamena i poluhidratnog gipsa. Analiza je identifikovala faktore sa značajnim uticajem na odvajanje slobodne vode. Analiza korelacije je otkrila da sadržaj poluhidratnog gipsa pokazuje jaku negativnu korelaciju sa odvajanjem vode (r = -0,85), posebno kod mlevenja u otvorenom ciklusu. Ovo sugeriše da veći sadržaj poluhidrata može olakšati brže reakcije hidratacije, smanjujući slobodnu vodu. U međuvremenu, specifična površina je pokazala konzistentnu negativnu korelaciju sa odvajanjem vode u oba načina mlevenja, što naglašava važnost finoće u kontroli krvarenja. Razvijene su regresione jednačine za predviđanje odvajanja slobodne vode.

Ključne reči. ostatak na situ od 45 mm, gips hemihidrata, anhidrit, odvajanje vode, specifična površina cementa.

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