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To cite this article: L D Shahova et al 2018 IOP Conf. Ser.: Mater. Sci. Eng. 327 032049

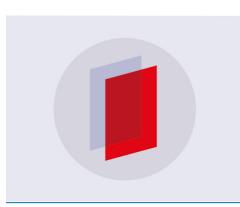
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Flowability of cement powder

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Abstract. The paper considers principal factors influencing fluid properties of cement powder. Analysis with a correlation regression method has shown that a variable factor which has not been previously quantitatively evaluated, influences the fluid properties.

1. Introduction

Diminishing flowability (mobility) of cement during large-scale handling operations with road and railroad transport in bulk or in tare creates significant problems, increasing time and labor costs of handling operations [1]. This may lead to quite significant delays.

Flow of powders is a combination of physical properties of material, environmental conditions and equipment used for processing and storage of the materials [2]. Behavior of a stream of powdered material has a complex nature and depends on many physical characteristics.

Causes, leading to deterioration of cement powder flowability, are not yet completely understood. The principle cause, according to many researchers, is a complex relationship between surface energy of single grains of cement and the stream of the same grains. Surface energy depends on multiple factors arising during the cement production process: fineness of cement, a type of grinding machinery, a rotary kiln type and a production method (wet or dry), burning and cooling temperatures, a cooling rate, etc. [3].

These factors play an important role in shaping other properties of cement, such as packed density, an angle of natural repose, caking ability in silos, duration of handling from storage and transportation reservoirs. Even slight change in the value of these factors may lead to significant changes in powder mobility: a reduction in grain size leads to lower flowability of the powder. To effectively manage and predict flowability values, it is important to understand its role and significance in cement handling and storage processes, as well as controlling mechanisms for this parameter.

There are several empirical regularities: hydrophobic powders flow better than hydrophilic ones; monodisperse powders flow better than polydisperse ones [4-5]. According to these empirical regularities, cements produced in closed-cycle mills with separators shall have higher flowability than those produced in open-cycle mills that have the same specific surface area. As evident from practice, open-cycle cement starts aggregating earlier. This is due to the fact that small particles are not removed from the grinding bodies action zone, thus they adhere to the grinding bodies and reduce grinding efficiency. The supplied energy is expended not only upon milling, but upon destruction of

newly-formed aggregates as well. That is why open-cycle mills do not allow for production of cements with specific surface area above $400 \text{ m}^2/\text{kg}$ [6].

Challenges in evaluating influence from a multitude of factors onto cement flowability lay in impossibility to quantify the flow under concrete process conditions [7-9]. Thus, in these experiments the flowability was determined in the lab in accordance with the ASTM C1565-09 method with consideration of parameters that may be quantified.

2. Materials and methods

Samples of cement were taken at different points to study influence of different factors onto its flow: at unloading from silos (set 1) and right after an open-cycle mill (set 2).

Set 1 - number of observations 92. For unloaded batch of CEM II/A-Sh 32.5B cement, the following parameters were determined: flowability as per ASTM C1565-09, rest on sieve no.008, Blaine specific surface area, moisture content, bulk density and maximum packed density.

Set 2 – number of observations 30. For Set 2, cement samples were taken during industrial test of a grinding-intensifying process additive of a Litoplast AI series, produced by LLC Polyplast Novomoskovsk. The samples were taken from an output chute of a 4x13.5 m open-cycle cement grinding mill, at equal intervals during 2 days. The grinding intensifier was supplied to a clinker transporter at a rate of 200 g per 1 tonne of cement when producing CEM II/A-Sh 32.5B type cement. For the samples taken, the main parameters were analyzed: cement temperature, chemical and material composition, fineness by specific surface and grain-size distribution. Cement temperature after the mill was measured with an integrated transducer and indicated at the mill operator's panel. The grain-size distribution of the cement was determined with a special instrument ANALYSETTE 22 manufactured by FRITSCH. As a result of the analysis, the weight ratio was determined for grains up to 3 micron, from 3 to 30 micron, from 30 to 80 micron and over 80 micron. Cement powder flowability was tested in accordance with ASTM C1565-09 directly after the mill and after cooling the sample in the lab to room temperature.

To study correlations between cement flowability and the multitude of factors, a correlation regression analysis was used, as implemented in STATISTICA software package. This method considers intercorrelation between parameters when dependency between them is not strictly functional and is distorted by irrelevant random factors.

To select the most informative regression model, a method of stepwise (ridge) regression was applied, pertaining to methods of a dimensionality reduction. It is applied when there is an excess of data and independent variables correlate with each other, that is, there is a multicollinearity [10].

Strength of relationship in the correlation analysis was determined with the Chaddock scale.

3. Results of statistical analysis

Set 1

Descriptive statistics for Set 1 data with the statement of variable parameters x_i and output parameter y are shown in Table 1. Output of the Multiple Regression module of STATISTICA software is given in Figure 1.

Value	Output	Variable parameters											
	parameter Y1 – cement flow, %		x2 – Blaine specific surface area, cm ² /g		ratio of	x5 – loose bulk density, g/l;	x6 – maximum compacted bulk density, g/l;	x7 – caking degree					
Average	25.54	8.376	293.45	0.028	10.59	1132.26	1591.83	0.712					
Minimum	16	5.5	267	0.01	2.1	1046	1327	0.63					

 Table 1. Descriptive statistics for Set 1

Maximum	31	13.3	335	0.05	19.7	1236	1788	0.851
Standard deviation	2.83	1.65	14.56	0.00785	3.59	49.47	90.63	0.037
Correlation coefficient between x _i and y		-0.114	-0.22	-0.06	-0.017	-0.075	-0.044	-0.033

	Regression Summary for Dependent Variable: Y (текучесть) (регрессия текучесть) R= ,31287695 R?= ,09789198 Adjusted R?= ,03421377 F(6,85)=1,5373 p<,17587 Std.Error of estimate: 2,7718												
	b*												
N=92		of b* of b											
Intercept			52,43706	10,88447	4,81761	0,000006							
Х1 (Ост 008)	-0,214230	0,124124	-0,36496	0,21146	-1,72593	0,087993							
Х2 (Уд.пов.)	-0,301510	0,110260	-0,05841	0,02136	-2,73453	0,007601							
X3 (W,%)	0,019902	0,113730	7,14229	40,81522	0,17499	0,861503							
Х4 (шлак)	0,010453	0,112744	0,00821	0,08850	0,09271	0,926349							
Х5 (насыпной вес)	-0,091392	0,110832	-0,00521	0,00632	-0,82460	0,411906							
X7	-0,020373	0,108200	-1,53505	8,15247	-0,18829	0,851096							

Figure 1. Results of regression analysis in Statistica.

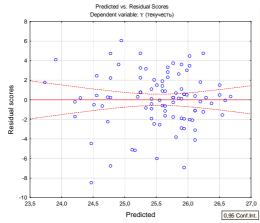
As evident from the results, only one parameter $-X_2$ has a significant influence over the output variable (flow). The rest of the regression coefficients are insignificant, except for b_2 ,. The value of determination coefficient is $R^2 = 0.098$. A regression model selected with it as the most informative from the point of view of explaining dispersion, includes only one factorial attribute - X_2 (cement powder specific surface area) (see Fig. 1):

$Y = 52.44 - 0.058 X_2$

Accuracy of the obtained single-factor regression equation in describing the output variable, namely, cement powder flow, may be shown in Figure 2.

Thus, the scatter chart characterized the shift of regression residue estimations. A determination coefficient is also one of the regression equation significance evaluation and it characterizes the degree of intensity of inter-variable link. Figure 3 illustrates co-dependency of considered parameter Y and factor X2.

The estimation of determination coefficient R^2 (0.098), obtained while implementing the method, shows what ratio of the variable variation is due to variation in the factor attribute; it shows that changes in cement powder flowability are completely determined by influence of factors which are still unaccounted for in the model.



Y = 56.8493-0.1684*x+0.0002*x^2 40 20 0 36 34 32 30 28 Y (текучесть) 26 24 22 20 18 16 14 12 L 300 320 340 20 40 260 280 360 0 Х2 (Уд.пов.)

Figure 2. Distribution of regression residues depending on predicted values of flow.

Figure 3. Results of correlation analysis in Statistica.

Set 2

Descriptive statistics for Set 2 data with the statement of variable parameters x_i and output parameters y_1 and y_2 are shown in Table 2.

			Table2. I	Descriptiv	ve statistic	s for Set 2	2					
Value	Output pa	arameters y	Variable parameters x _i									
	y1 - cement flowabilit y after mill	y2 - cement flowability at room temperatur e $25^{\circ}C$	x1 – cement temperature after mill, ⁰ C	mass ratio in	specific	x4 - weight ratio of grains under 3 micron	ratio of	x6 - weight ratio of grains between 30 and 80 micron	ratio of grains over			
Average	41.22	41.26	127.9	16.58	299.2	10.25	49.89	30.71	9.05			
Minimum	28.5	25.5	98	7.37	237	7.8	45.1	25.9	5			
Maximum	50	56	169	25.06	433	13.9	55.2	34.7	12.8			
Standard deviation	5.34	5.41	15.8	3.74	43.5	1.35	2.85	2.1	1.84			
Correlation coefficient between x _i and y1	l		0.05	0.31	0.01	0.05	0.034	-0.04	-0.02			
Correlation coefficient between x _i and y2	I		-0.36	0.33	0.08	-0.09	-0.08	-0.2	-0.03			

Results of correlation analysis have shown that linear correlation coefficients for two variables varied from -0.04 (factor x6) to 0.31 (factor x2) for output variable y1 and from -0.36 (factor x1) to 0.33 (factor x2) - for variable y2. That is, all the correlation coefficients demonstrate weak dependency between output variables y1, y2 and variable factors (xi) according to the Chaddock scale.

The output of the Multiple Regression module for output parameter y1 is shown in Figure 4, a).

															05 0 0
	Regression	Summary	for Depend	dent Variab	le: Текуче	сть посл	е после мельницы, оС	Regression Summary for Dependent Variable: Текучесть при 25оС, %							
	R= ,41173697 R?= ,16952733 Adjusted R?=								R= ,49142259 R?= ,24149616 Adjusted R?= ,00015403						
F(7,22)=,64156 p<,71714 Std.Error of estimate: 5,4915								F(7,22)=1,0006 p<,45700 Std.Error of estimate: 5,3136							
1	b*	Std.Err.	h b	Std.Err.	t(22)	p-value			b*	Std.Err.	b	Std.Err.	t(22)	p-value	
	U		D		(22)	p-value		N=30		of b*		of b	()	praido	
N=30		of b*		of b						010	75 00570		0.05505	0.705744	
Intercept			-35,7520	218,9875	-0,163260	0,871804		Intercept				211,8935		0,725714	
X1	0,160759	0,222581	0,0541	0,0749	0,722247	0,477750	2	X1	-0,238484	0,212718	-0,08129	0,0725	-1,12113	0,274323	
X2	0.474773	0,231120	0,6736	0,3279	2,054227	0.052024	2	X2	0,257157	0,220879	0,36942	0,3173	1,16425	0,256795	
X3	-0.255494	0.279819	-0.0313	0.0343	-0.913070	0.371104		X3	-0,111891	0,267420	-0,01389	0,0332	-0,41841	0,679704	
X4	0.397416	0,662677	1,5546	2,5922	0,599713	0,554825	2	X4	-0,025614	0,633313	-0,10145	2,5083	-0,04044	0,968103	
X5	0.324288	1,164906	0,5891	2,1163	0,278381	0,783320		X5	-0,254631	1,113287	-0,46836	2,0478	-0,22872	0,821200	
X6	0,262728	0,974081	0,6416	2,3787	0,269719	0,789890	2	X6	0,175880	0,930918	0,43486	2,3017	0,18893	0,851878	
X7	0,124519	0,856279	0,3590	2,4685	0,145419	0,885704	2	X7	-0,546556	0,818336	-1,59526	2,3885	-0,66789	0,511152	
		<u></u>)						b)							
		a)											b)		

Figure 4. Results of correlation analysis in Statistica. a) for output parameter y1; b) for output parameter y2

As evident from the data, all the regression coefficients are insignificant.

Determination coefficient R^2 calculated in the software shows that the ratio of dispersion of the dependent variable (cement flowability after mill), determined by influence from factors X1-X7 is just 0.169. The rest of the variation in flowability shall be explained with other factors, not covered by this experiment.

Similar results were obtained in regression analysis for influence from factors X1-X7 onto cement powder flowability at 25°C (Figure 4, b).

All the regression coefficients are insignificant, determination coefficient $R^2 = 0.24$ is just a little greater than the value obtained for cement flowability after mill.

Results of regression analysis for Set 2 have shown that out of all the studied factors the highest influence onto flowability after the mill and at room temperature are shown by slug content (x2) and grain size composition (x4-x7). All the coefficients in the regression equation are characterized by

very low values, that is, almost every variable has insignificant influence over the output parameter. This fact is further supported with low values of determination coefficient R^2 , whose value does not exceed 0.241 for total influence of the seven factors. That is, there is 24.1% probability that these factors influence the cement flow, the remaining 75.9% are covered by unknown factors.

4. Conclusion

As it has been demonstrated, the authors failed to find a dependency between cement flowability and all the variable parameters the authors had selected. It is an evidence of the fact that cement flowability under considered conditions is influenced by other random parameters, not accounted for in this experiment and requiring additional studies.

5. Acknowledgments

The article was prepared within the development program of the Flagship Regional University on the basis of Belgorod State Technological University named after V.G. Shoukhov, using equipment of High Technology Center at BSTU named after V.G. Shukhov.

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