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Analysis of variance for testing method of cement in determination of strength

Hong HUANG, Xiao-dong SHEN

State Key Laboratory of Materials-Oriented Chemical Engineering, College of Material Science and Engineering, Nanjing University of Technology, Nanjing 210009, China

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Abstract: The statistical tools such as descriptive statistics, full factorial design and analysis of source of variation were used to identify the potential factors that impact the validity of testing method for determining the strength of cement. The results showed that personal error impacted both accuracy and precision of test greatly. Experimental time associated with temperature fluctuation resulted in strength variation but did not impact the precision of test in all curing ages. Different compactions did not impact the precision of test but resulted in the strength variation on 3 d and 28 d significantly. Different methods for the initial moist air curing significantly impacted the precision of testing method and resulted in the strength variation of cement on 1 d.

Key words: testing method of cement; analysis of variance; strength; accuracy; precision

1 Introduction

Testing method for determining the strength of cement, also called mortar test, is widely used to evaluate the mechanical property of finished cement and related cementitious materials. R&D works such as innovation of cement additive or other activities for improving the quality of cement also use this testing method to verify the experimental effectiveness. Currently in China, GB/T 17671—1999 which was derived from EN 196—1, is used as the national code for determining the strength of cement [1−2]. As a matter of fact, there are several potential factors that can impact the validity of this testing method significantly. When massive mortar tests are needed, it is necessary to evaluate the accuracy and the precision of testing method under certain conditions. Recently, the design of experiments, which is a statistical methodology, was used successfully for the chemical admixture innovation in cementitious materials [3−5]. It compared the experimental effectiveness versus testing errors. Thus, the smaller the testing error, the more powerful the methodology is.

In order to enhance the validity of mortar test, this work devised a laboratorial measurement system analysis to identify the potential factors that significantly impacted the accuracy and the precision of testing method [6]. Descriptive statistics, full factorial design and analysis of source of variation (SOV) were used as the tools to quantify personal, systematic and the environmental errors involved in experimental procedures [6, 7].

2 Materials and testing method

Two types of cement were used. They were a 32.5 fly ash cement and a 52.5R cement, respectively. The chemical compositions of cements were analyzed by X-ray fluorescence (XRF) and are shown in Table 1.

The mortar test strictly followed the procedures regulated in the national code (GB/T 17671—1999). The procedures for preparation and strength determination of cement mortar include materials weighing, mixing, casting and compacting, sleeking, initial curing in moist air, demoulding, curing in water and final prism testing. According to the national code, the strength tests of mortar on 3 days and 28 days are compulsive. However, the 1 d strength was also tested in this study since it is an important criterion to evaluate the early performance of cementitious materials [8, 9].

Generally, strength variation of mortar test may come from six aspects (Fig. 1). Testing machine for determining the strength of cement mortar is assumed to contribute little variation since it is well calibrated before

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Corresponding author: Xiao-dong SHEN; Tel: +86-25-83587234; E-mail: 20855309@qq.com; xdshen@njut.edu.cn

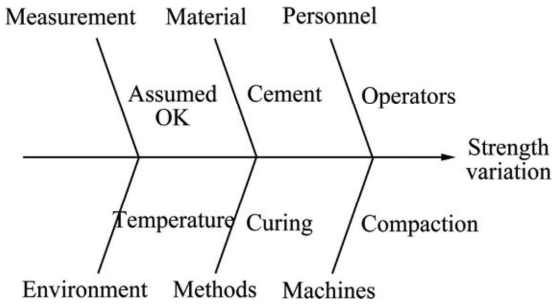

Fig. 1 Cause and effect diagram for strength variation of cement

it is in use. Materials are weighed by electronic balance with precision of 0.1 g, thus it is also considered that the weighing process is capable. Personal error, which comes from experimental operators, is considered to affect the reproducibility of experiment. In compaction process of fresh mix, the jolting apparatus is used. However, a type of vibrating table mentioned in appendixes of both EN 196—1 and GB/T 17671—1999 is used as the alternative equipment for compaction. The difference of jolting apparatus and vibrating table that may result in strength variation was investigated. As for the initial curing in moist air chamber after compaction, it is prescribed differently in EN196—1 and GB/T 17671—1999. That is, the former one regulates a plate of glass, steel or other impermeable material which does not react with cement, which should be placed on the mould after fresh mix is cast and compacted, but the latter does not and it allows the surface of specimen to directly contact the moist air in curing chamber. It is considered that covering or not on mould affects the moisture condensation on the surface of specimen, so that it may results in undesirable strength variation. At last, the environmental factor that tends to cause strength variation from day to day is taken into account. The cement mortar is prepared at different times since the temperature fluctuation of laboratorial environment may result in the temperature differences of materials including cement, standard sand and the water for mixing. It is assumed low room temperature in the mornings and relative higher temperature in the afternoons.

$$\mathrm{CV} = \frac{\sigma}{\mu} \tag{1}$$

where σ is the standard deviation (StDev) and μ is the arithmetic mean.

Coefficient of variation (CV) is a normalized measure of dispersion of a probability distribution. CV was used to estimate the precision of mortar test influenced by certain experimental factors.

3 Full factorial design

The full factorial design was used to quantify the systematic and environmental errors including cement type, compaction, initial curing and the experiment time on strength variation. In this investigation a 2^k (k=4) factorial model was used, which investigated both main effects and the possible interactions among the factors. Table 2 shows the four factors. The levels of factors are coded as −1 and 1, respectively, for convenient representation in factorial matrix.

When fitting the factorial model, analysis of variance (ANOVA) was used to judge the statistical significance of the terms. The *p*-value determines the appropriateness of rejecting the null hypothesis in a hypothesis test and it ranges from 0 to 1. The smaller the *p*-value, the smaller the probability that rejecting the null hypothesis is a mistake [10]. The *t*-value coming from Student *t*-test represents the significance of regression coefficient. The larger the value of *t* and the smaller the value of *p*, the more significant the corresponding coefficient term is [10]. The alpha (α) level in this study was defined as 0.05, thus items in ANOVA with *p*-value less than 0.05 are considered significant.

Table 1 Chemical compositions of 32.5 and 52.5R cements (mass fraction, %)

Cement	CaO	SiO_2	Al_2O_3	Fe_2O_3	K_2O	Na_2O	SO_3	MgO	LOI
32.5	62.7	21.3	5.5	3.2	0.9	0.1	2.1	2.3	1.28
52.5R	63.9	19.8	4.4	3.1	0.7	0.1	3.8	1.0	2.8

Table 2 Actual and coded values of factors

Factor	Symbol	Low level		High level	
		Coded	Actual	Coded	Actual
Cement	X_1	−1	32.5	1	52.5R
Compaction	X_2	−1	Jolting apparatus	1	Vibrating table
Initial curing	X_3	−1	Uncovered	1	Covered
Experiment time	X_4	−1	Morning	1	Afternoon

4 Results

4.1 Determination of personal error

In order to investigate the influence of personal error on the validity of mortar test, 10 technical operators were required to conduct cement mortar preparation by using the 32.5 fly ash cement. The whole experimental process was finished in 2 h, thus the variations of room temperature and the temperature of materials were considered to be negligible. The compressive strength of mortar was given by the arithmetic mean of 6 determinations made on a set of 3 prisms. 10 sets of data were collected and the statistical results are presented in Table 3.

Table 3 1 d compressive strength of cement mortar prepared by 10 different operators

Operator	Compressive strength/MPa		CV/%
	Mean	StDev	
A	7.1	0.7	10.1
B	7.1	0.2	2.8
C	6.2	0.3	4.2
D	6.2	0.5	7.4
E	6.7	0.7	11.0
F	6.5	0.2	3.3
G	7.5	0.3	3.7
H	6.7	0.7	10.4
I	7.4	0.2	2.5
J	6.9	0.4	5.8

It can be seen that the mean values of 10 sets of data varied greatly, from minimal 6.2 MPa to maximal 7.5 MPa. Meanwhile, the standard deviation (StDev) as well as the coefficient of variation of each data set was significantly different. The CVs of mortar prepared by operator A, E and H are greater than 10%, the tests of from whom considered as invalid, and the rest of CVs were acceptable according to national code. The operator I gave the most precise test (CV=2.5%) and thus was arranged to conduct the following experiment.

4.2 Determination of systematic and environmental errors

The influences of four factors including cement type, compaction, initial curing and the experimental time on the strength variation of cement mortar were studied by full factorial design. The design matrix of factors and the corresponding mortar strength are listed in Table 4. The compressive strength of mortar was given by means of the arithmetic mean of four determinations made on a set of two prisms. The statistical analysis of strength data is presented in Table 5.

Without doubt, the cement type was the most significant factor that resulted in the strength variation in all curing ages. Excluding the influence of cement type, the strength variations resulting from the rest of factors could be ordered by judging the *t*-value of each item. They were $X_4>X_3>X_1X_4>X_1X_2$ for 1 d strength, $X_4>X_2$ for 3 d strength and $X_2>X_4$ for 28 d strength.

The influences of significant factors resulting in the strength variation of cement in different curing ages are illustrated in Fig. 2. It can be noted that the experimental time induced strength variation in all curing ages. The

Table 4 Design matrix of factors and strength of cement

Run No.	Factor (Coded)				Mean compressive strength/MPa			StDev of compressive strength/MPa			CV/%		
	X_1	X_2	X_3	X_4	1 d	3 d	28 d	1 d	3 d	28 d	1 d	3 d	28 d
1	−1	−1	−1	−1	7.4	18.3	35.8	0.3	0.4	0.6	3.5	2.0	1.7
2	−1	−1	1	−1	7.6	18.2	35.6	0.1	0.1	0.2	1.7	0.7	0.7
3	−1	1	−1	−1	7.3	18.2	37.8	0.2	0.5	0.5	2.8	2.6	1.4
4	−1	1	1	−1	7.5	18.8	38.5	0.1	0.3	0.5	1.7	1.8	1.2
5	1	−1	−1	−1	14.9	33.5	59.4	0.7	0.5	0.9	4.9	1.6	1.5
6	1	−1	1	−1	15.0	34.4	61.5	0.3	0.7	0.9	2.0	2.0	1.4
7	1	1	−1	−1	15.1	34.3	64.2	0.3	0.9	0.9	2.3	2.6	1.5
8	1	1	1	−1	15.4	34.5	64.0	0.6	0.6	0.6	3.7	1.7	0.9
9	−1	−1	1	1	7.7	19.2	37.9	0.2	0.4	0.7	2.4	2.3	1.7
10	−1	−1	−1	1	7.9	19.1	37.7	0.2	0.4	0.9	2.2	1.8	2.4
11	−1	1	1	1	7.8	19.4	39.0	0.2	0.4	0.5	2.9	1.9	1.3
12	−1	1	−1	1	7.8	19.8	39.3	0.1	0.5	0.4	1.6	2.4	0.9
13	1	−1	1	1	15.8	33.8	63.9	0.3	1.0	0.7	2.0	3.1	1.1
14	1	−1	−1	1	16.0	34.5	63.8	0.4	0.5	1.1	2.5	1.5	1.8
15	1	1	1	1	15.8	35.4	66.2	0.5	0.7	1.4	3.4	2.1	2.1
16	1	1	−1	1	16.2	35.2	66.6	0.3	0.8	1.1	1.8	2.3	1.7

Table 5 ANOVA for full factorial design

Item	1 d strength		3 d strength		28 d strength	
	t	*p*	*t*	*p*	*t*	*p*
X_1	171.60	0	93.60	0	95.30	0
X_2	1.91	0.115	3.59	0.016	9.18	0
X_3	4.76	0.005	1.86	0.122	1.34	0.237
X_4	12.93	0	4.56	0.006	8.06	0
$X_1 X_2$	2.59	0.049	1.41	0.218	2.14	0.085
$X_1 X_3$	1.09	0.326	0.66	0.540	0.68	0.529
$X_1 X_4$	5.17	0.004	−1.37	0.229	2.40	0.062
$X_2 X_3$	0.95	0.385	−0.51	0.634	−0.17	0.870
$X_2 X_4$	−0.41	0.700	1.37	0.229	−1.98	0.104
$X_3 X_4$	0.27	0.796	−0.66	0.540	−0.93	0.396
Main effect		0		0		0
2-way interaction		0.035		0.451		0.156
R^2		99.98%		99.94%		99.95%
$R_{(adj)}^2$		99.95%		99.83%		99.84%

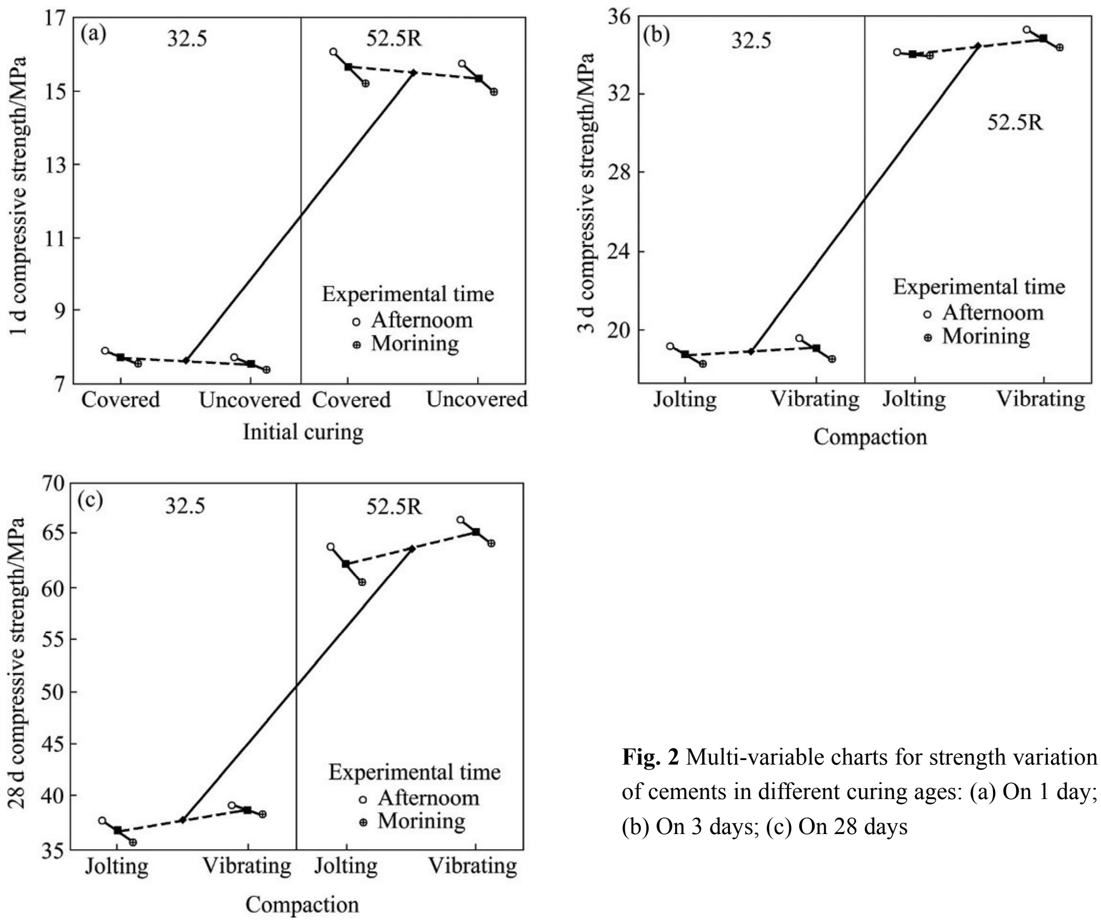

Fig. 2 Multi-variable charts for strength variation of cements in different curing ages: (a) On 1 day; (b) On 3 days; (c) On 28 days

strength of mortars prepared in the afternoon was always higher than that of mortars prepared in the morning. The difference in initial curing directly caused strength variation on 1 day (Fig. 2(a)). Specimens covered by impermeable material tended to have higher strength than that of uncovered specimens. Experimental time tended to affect the 1 d strength of 52.5R cement more significantly. The compaction method and the experimental time were considered the significant factors that resulted in the strength variations (Figs. 2(b) and

2(c)). Specimens compacted by vibrating table tended to have higher strength than that of specimens compacted by jolting apparatus.

4.3 Analysis of sources of variation

This study also investigated the influence of source of variation (SOV) on the precision of testing method. The distribution of the coefficient of variation (CV) was used as the criterion to evaluate the precision of experiment. The comparison of CVs regarding different SOVs was drawn by ANOVA (Fig. 3). The precision of 1 d strength data of the cement mortar prepared by single operator and a group of operators was significantly different, which was judged by *p*-value (0.000) in Fig. 3(a). It showed that the distribution of data obtained from mortar prepared by single operator was far more concentrated than that of mortar prepared by different operators. The comparison of CVs regarding different cements is presented in Fig. 3(b). It showed that the comparison between 32.5 fly ash cement and 52.5R cement was not significantly different, which suggests the precision of mortar test was not sensitive to cement type. The ways for compaction were compared (Fig. 3(c)). The ANOVA also showed that compaction method itself did not influence the precision of test. The comparison of CVs between the two initial curing methods was significantly different (Fig. 3(d)), in which the *p*-value is 0.027. The mean value of CV of mortar covered by the impermeable material was significantly smaller than that of uncovered mortar during the initial

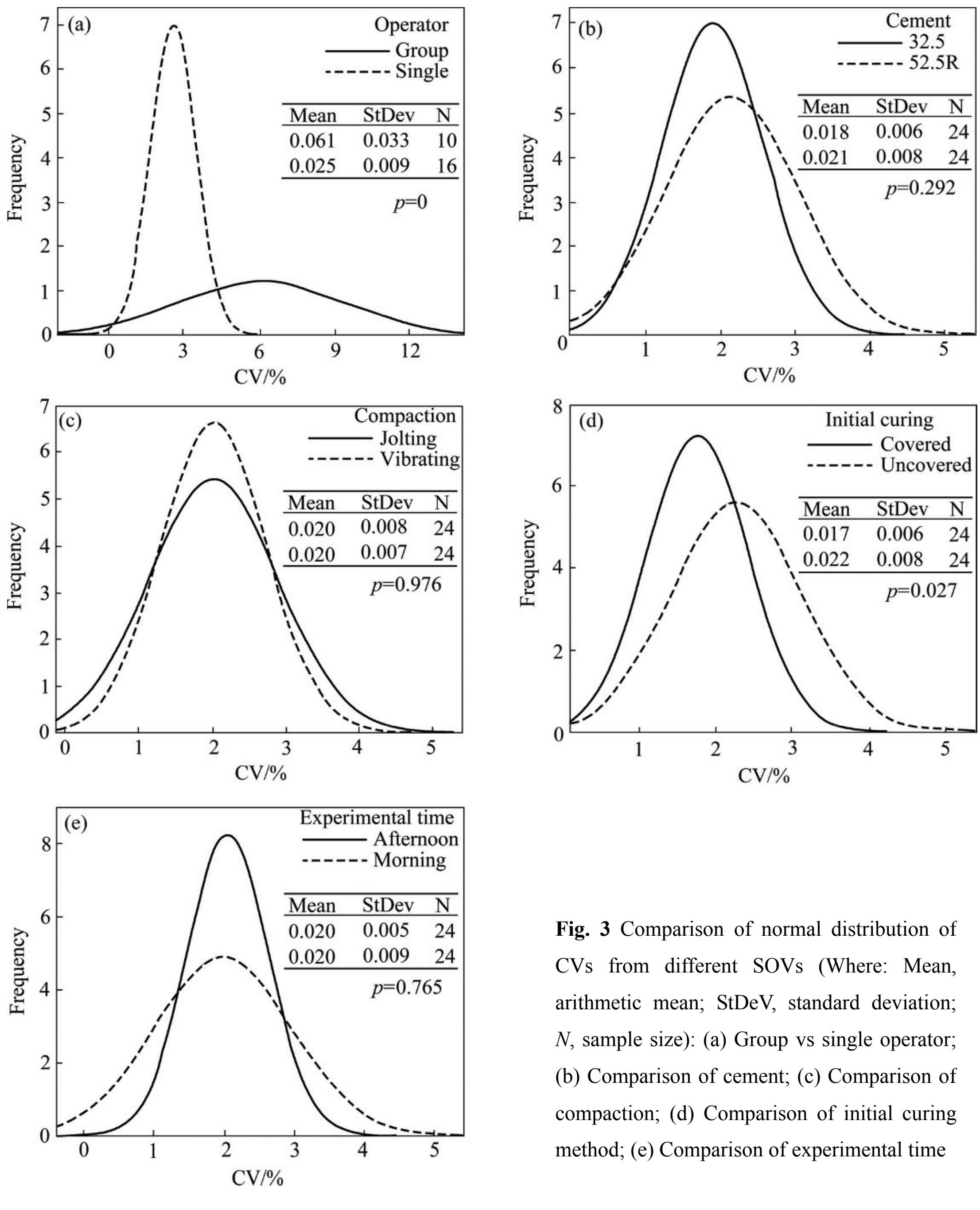

Fig. 3 Comparison of normal distribution of CVs from different SOVs (Where: Mean, arithmetic mean; StDeV, standard deviation; *N*, sample size): (a) Group vs single operator; (b) Comparison of cement; (c) Comparison of compaction; (d) Comparison of initial curing method; (e) Comparison of experimental time

curing in moist air. At last the comparison of different experimental time was drawn in Fig. 3(e). It showed that the experimental time did not impact the precision of experimental data since the *p*-value for the comparison is 0.765.

5 Discussion

According to statistical analysis it can be seen that the personal error seriously impacted both accuracy and precision of mortar test. It may be due to the different manners of operators conducting the mortar preparation even the same required procedures were followed such as casting and sleeking. It is suggested that the operators participated in the mortar preparation should be well trained and the mortar test especially for comparative study must be prepared by a single fixed operator.

The full factorial design determined the significant factors that resulted in the strength variation in different curing ages, while the comparison of the CVs from different SOVs indicated the factors that impacted the precision of mortar test. Experimental time resulting in temperature fluctuation significantly contributed to the strength variations of cements in all curing ages. It indicated that it is important to keep all the materials including cement, standard sand and mixing water at a constant temperature and to guarantee the temperature consistency of materials every time before mortar is prepared. The interaction between experimental time and cement type occurred after initial curing, resulting in the strength variation on 1 d, which indicated the early strength of high strength grade cement was more sensitive to the temperature variation. Thus, the temperature requirement for the materials in EN 196—1 is reasonable. The mortar being covered or not during initial curing in moist air chamber directly generated the strength variation on 1 d as well as impacted the precision of test in all curing ages. It indicated that the coverage of fresh mix is essential to enhance the validity of mortar test. As for the vibrating table mentioned in both appendixes of EN 196—1 and GB/T 17671—1999, it was showed that mortars compacted by vibrating table tended to have higher strength on 3 d and 28 d than the mortars compacted by jolting apparatus. However, both of the equipments did not impact the precision of the test. Considering the convenient operation of vibrating table, it is recommended to use when large scale of comparative mortar test needs to be accomplished.

6 Conclusions

In order to enhance the validity of testing method for determining the strength of cement, the potential factors that resulted in the strength variation of cement and impacted the precision of test were investigated. The results showed that personal error impacted the accuracy and the precision of cement mortar test seriously. Cement type and the compacting methods did not impact the precision of testing method. However, the mortar compacted by vibrating table tended to have higher strength on 3 d and 28 d. The difference in initial curing only affected the strength on 1 d but it influenced the precision of test significantly. The mortars covered by the impermeable material tended to have higher 1 d strength and the testing data from which was more concentrated. The experimental time associated with temperature fluctuation did not impact the precision of test but resulted in strength variation of cements in all curing ages.

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