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Drying shrinkage behavior of mortars made with ternary blends

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ABSTRACT

Ternary cementitious blends are widely used in today's concrete mixtures, particularly, when high performance is needed. In this paper, drying shrinkage behavior of mortar mixtures made with various ternary blends is discussed. Ternary blends consisting of different combinations of portland or blended cement, slag, fly ash and silica fume were considered. The amounts of slag, fly ash and silica fume ranged between 15-35%, 13-30%, and 3-10% by mass of cementitious materials, respectively. Mortar bars were made with the ternary blends and subjected to drying (i.e., T = 73 ± 3 °F and RH = 50 ±4%) after standard moist curing for 28 days. Free shrinkage of the bars was assessed at 56 days of age after 28 days of drying. A response surface analysis was done to examine the effects of blend proportions on shrinkage behavior of the mortars. To validate this model, an independent group of mortar mixtures with different ternary combinations was cast, and the measured values were compared with the predicted shrinkage values. The results indicated that among the three supplementary cementitious materials in the ternary blends studied, slag showed a dominant effect on increasing mortar shrinkage. Contribution of class C fly ash to the shrinkage was slightly less than that of slag. Increasing silica fume content slightly increased free shrinkage, and similarly an increase in class F fly ash content slightly increased free shrinkage. There is a good correlation between the measured shrinkage strain and the strain predicted from the shrinkage model developed from the response surface analysis.

Keywords: ternary blend, supplementary cementitious materials, shrinkage

INTRODUCTION

Supplementary cementitious materials (SCMs), such as fly ash, slag cement (referred to as slag hereafter), natural pozzolans and silica fume improve concrete performance by nature of their pozzolanic or combined pozzolanic and cementitious properties [1]. SCMs not only improve concrete performance but also have the benefit of reducing environmental impact. Ternary mixtures (i.e., concrete mixtures that contain two different SCMs and portland cement as binder) are used due to their potentially improved performance compared to plain portland cement or binary systems.

Shrinkage can be one of the major causes of concrete distress. Shrinkage cracking can affect structural capacity in pavements, and can increase water penetrability giving rise to durability problems. The component of concrete that shrinks is the cement paste when water is removed from the capillaries. Water in the larger voids (>50 nm) is considered to be "free water" since its removal does not result in volume change. However, water in small capillary voids (5 to 50 nm) may result in large shrinkage strains when it is forced to leave the system [2]. The size and volume of the capillary voids are determined by the initial water-to-cement ratio and the degree of hydration of the mixture. The microstructure is also modified also by the binder system used. The effects of SCMs on shrinkage have not been clearly articulated and there are conflicting findings in the literature. For instance, silica fume was found to increase drying shrinkage in a binary system [3-5] but decrease shrinkage strain when used in combination with slag and portland cement [6].

Little has been reported on the shrinkage behavior of ternary blends. The work presented in this paper aimed at developing statistical models for the drying shrinkage behavior of portland cement, slag, fly ash and silica fume when used in ternary combinations. Shrinkage of mortar mixtures was determined as 28 days of drying after 28 days of moist curing. 50 ternary blends were used to construct the models and another 12 mixtures to verify the validity of the models. .

EXPERIMENTAL PROGRAM

This study was conducted in two phases: developing the shrinkage models based on statistical analysis of previous experiment work and verification of the proposed models. The statistical analysis was conducted using data published by Tikalsky et al. [7] along with the supplementary data obtained from the mortar mixtures prepared in the current work. In the second phase a series of mortar bars was cast to verify the model obtained in the first phase.

Phase I

A total of 50 mixtures were used for the statistical model: 36 from reference 7 and 14 newly mixed. Mortars were prepared with the same mix parameters used in reference 7 aggregate-to-cementitious materials ratio of 2.75 and a water-to-cementitious ratio of 0.45. The fine aggregate used in reference 7 had a specific gravity of 2.61 and a fineness modulus of 2.90, while the fine aggregate used in the additional lab study had a specific gravity of 2.62 and a fineness modulus of 3.09. A sucrose-based water reducing admixture was used to maintain a flow between 80 and 120% in accordance with ASTM C 1437 [8]. Chemistry of the cementitious materials used in both sets of laboratory work is given in Table 1.

The ternary mixtures are grouped into three series:

Series I - Type I portland cement, slag (15-35%), class F fly ash (13-30%);
Series II - Type I portland cement, slag (15-35%), class C fly ash (15-30%);
Series III - Type I portland cement, slag (15-35%), silica fume (3-10%).

The combinations are given in Table 2(a), 2(b), and 2(c). Portland cement, slag, class C ash, class F ash and silica fume are denoted as PC, S, CFA, FFA and SF, respectively. The ternary cementitious systems were either separately blended or a combination of a manufacturer blended Type IP or IS with an added SCM.

Four $1 \times 1 \times 11$ ¼ inch mortar bars were prepared for each mixture in accordance with ASTM C 157 [9]. The mortar bars were moist cured for 28 days, then, transferred to the drying room maintained at $50 \pm 4\%$ relative humidity and a temperature of 73 ± 3 °F. Shrinkage measurements were taken up to 28 days from the start of drying.

TABLE 1 Chemical and Physical Properties of Cementitious Materials

Chemical, %	Phase I										Phase II		
	Type I-1	Type IS(20)	Type IP(20)	Type IP(7)	CFA	FFA1	FFA2	S100	S120	SF	Type I-2	S	SF2
CaO	61.71	58.19	50.88	59.15	27.18	3.78	13.15	36.86	36.77	0.42	63.22	42.25	0.46
SiO_2	19.80	23.53	28.88	24.91	34.02	45.05	51.40	37.40	36.81	97.90	20.97	38.82	94.32
Al_2O_3	6.18	5.29	8.19	4.38	18.20	23.71	16.21	8.98	9.66	0.18	4.47	7.27	0.28
Fe_2O_3	2.50	2.97	3.70	3.12	6.59	16.43	6.73	0.76	0.61	0.07	2.93	0.81	0.31
MgO	2.76	4.34	1.60	1.36	5.06	0.88	4.41	10.60	10.03	0.21	2.30	9.02	0.75
K_2O	0.74	0.58	0.90	0.56	0.35	1.46	2.33	0.40	0.35	0.59	0.60	0.50	0.48
Na_2O	0.36	0.13	0.35	0.22	1.56	0.80	2.86	0.29	0.31	0.12	0.16	0.31	0.08
SO_3	2.63	2.88	2.74	3.33	2.70	0.68	0.80	n/a	n/a	0.17	2.63	n/a	0.05
P_2O_5	0.21	0.09	0.22	0.11	1.29	0.24	0.15	0.02	0.01	0.12	0.12	0.03	0.09
TiO_2	0.28	0.41	0.44	0.29	1.57	1.15	0.63	0.38	0.49	n/a	0.31	0.41	0.01
SrO	0.24	0.04	0.20	0.10	0.50	0.18	0.33	0.04	0.05	0.01	0.08	0.05	n/a
Mn_2O_3	0.11	0.50	0.20	0.18	0.06	0.03	0.05	0.73	0.39	0.03	0.04	0.52	n/a
LOI	2.37	0.70	1.14	1.60	0.27	5.39	0.05	n/a	n/a	n/a	2.17	n/a	2.58
Specific gravity	3.04	2.95	3.11	3.08	2.62	2.37	2.41	2.82	2.96	2.21	3.15	2.87	2.25
Pozzolanic Index	n/a	n/a	n/a	n/a	108	86	107	97	112	125	n/a	n/a	n/a

Note: S100 and S120 denote different grades of slag cement.

TABLE 2 (a) Cementitious Percentages of the Blends in Series I

ID	FFA1	FFA2	FFA in IP(20)	S100	S120	Slag in IS(20)	Type I-1
I-1	30	-	-	20	-	-	50
I-2	30	-	-	-	20	-	50
I-3	-	30	-	20	-	-	50
I-4	-	30	-	-	20	-	50
I-5	15	-	-	35	-	-	50
I-6	-	15	-	35	-	-	50
I-7	15	-	-	-	35	-	50

I-8	-	15	-	-	35	-	50
I-9	20	-	-	20	-	-	60
I-10	20	-	-	-	20	-	60
I-11	-	20	-	20	-	-	60
I-12	-	20	-	-	20	-	60
I-13	-	-	13	35	-	-	52
I-14	-	-	13	-	35	-	52
I-15	25	-	-	-	-	15	60
I-16	-	25	-	-	-	15	60
I-17	-	-	16	20	-	-	64
I-18	-	-	16	-	20	-	64
I-19	15	-	-	-	-	17	68
I-20	-	15	-	-	-	17	68

TABLE 2 (b) Cementitious Percentages of the Blends in Series II

ID	CFA	S100	S120	S in IS(20)	Type I-1
II-1	30	20	-	-	50
II-2	30	-	20	-	50
II-3	15	35	-	-	50
II-4	15	-	35	-	50
II-5	20	20	-	-	60
II-6	20	-	20	-	60
II-7	25	-	-	15	60
II-8	15	-	-	17	68
II-9	22	-	22	-	56
II-10	22	22	-	-	56
II-11	15	-	15	-	70
II-12	15	-	25	-	60
II-13	15	25	-	-	60
II-14	25	15	-	-	60
II-15	25	-	15	-	60

TABLE 2 (c) Cementitious Percentages of the Blends in Series III

ID	S100	S120	S in IS(20)	SF1	Type I-1
III-1	35	-	-	5	60
III-2	-	35	-	5	60
III-3	35	-	-	5	60
III-4	-	35	-	5	60
III-5	35	-	-	3	62
III-6	-	35	-	3	62
III-7	-	-	19	5	76
III-8	-	-	19	3	78

III-9	-	30	-	5	65
III-10	-	25	-	10	65
III-11	-	30	-	10	60
III-12	-	28	-	7	65
III-13	28	-	-	7	65
III-14	-	20	-	10	70
III-15	-	23	-	7	70

Phase II

In order to validate the models obtained in phase I, verification mixtures were prepared and tested. Four verification mixtures were used for each of the three series. The ternary percentages within the valid range of the model were randomly selected. The proportions of the cementitious materials are given in Table 3. Chemistry of the cementitious materials used in this phase is provided in Table 1.

TABLE 3 Cementitious Percentages of the Blends Used for Model Verification in Phase II

ID	FFA1	S	CFA	SF2	Type I-2, %
V-I-1	15	30	-	-	55
V-I-2	15	35	-	-	50
V-I-3	22	22	-	-	56
V-I-4	25	25	-	-	50
V-II-1	-	18	18	-	64
V-II-2	-	25	25	-	50
V-II-3	-	15	30	-	55
V-II-4	-	28	28	-	44
V-III-1	-	30	-	5	65
V-III-2	-	25	-	4	71
V-III-3	-	25	-	10	65
V-III-4	-	15	-	8	77

RESULTS AND DISCUSSION

A commercially available statistical analysis software [10] was used to develop a quadratic response surface model for each series of ternary mixtures – PC+S+FFA, PC+S+CFA and PC+S+SF. A certain SCM type may represent materials from different sources with varying chemistry or physical characteristics. The aim was to use a spread of material sources for each type to improve the validity of prediction models, and the models did not take individual sources into account. The prediction models are valid for the ranges studied (i.e. slag: 15-35%, class F fly ash: 13-30%, class F fly ash: 15-30%, and silica fume: 3-10%).

Ternary Mixtures Series I: Portland Cement, Class F Fly Ash and Slag

Figure 1 gives the response surface fitted to the data. The prediction equation [1] whose R^2 value was 74%, is given below:

$$\varepsilon_{free} = -5051 \times FFA + 16041 \times S + 1605 \times PC - 17561 \times FFA \times S + 6645 \times FFA \times PC - 34835 \times S \times PC \qquad [1]$$

where ε_{free} = drying shrinkage at 28 days, μ-strain;

FFA = Class F fly ash content;

S = slag cement content;

PC = Type I portland cement content.

T-tests showed that the S content and the product of PC and S contents in series I mortar mixtures have statistically significant effects on 28-day drying shrinkage strain. Figure 1 (a) shows that for a given S replacement level between 15% and 25%, shrinkage strain increases with the increasing FFA content, whereas S content higher than 25%, shrinkage strain has a decreasing trend with the increasing FFA content. The findings seem to be in conflict with FFA effects in binary blends systems reported previously [1, 11]. The results suggest that the presence of S as a ternary component alters the FFA behavior. Furthermore, FFA effect might depend on the S content in the system. The contour lines developed from the response surface analysis in Figure 1 (b) show that S has a significant effect on drying shrinkage strain compared to FFA ash content. Moreover, it is observed that drying shrinkage increases with increased S content up to approximately 25% and then decreases up to 35% regardless the FFA content. A similar trend has been reported in the previous studies [11-13]. It is interesting that the maximum shrinkage occurs in the range in which slag is regularly used.

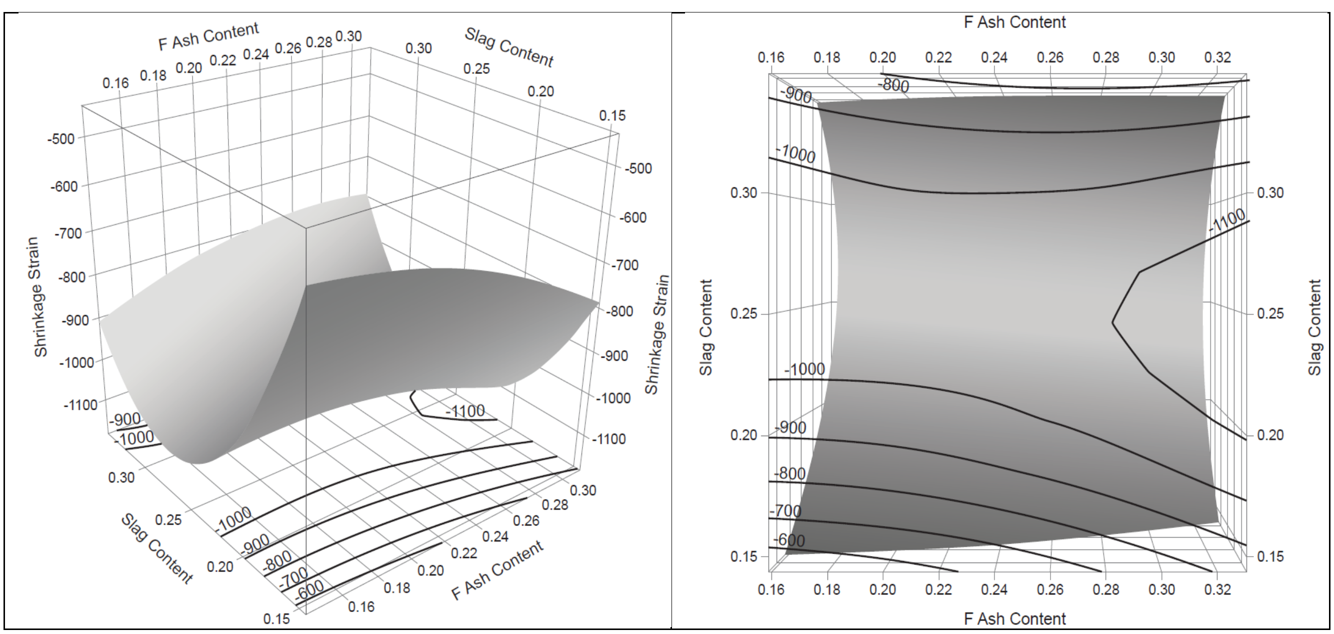

FIGURE 1 (a) response surface plot for series I (b) 2-D contour plot

Ternary Mixtures Series II: Portland Cement, Class C Fly Ash and Slag

The R^2 value for the fitted curve (Figure 2) is 79% and the equation [2] is given below:

$$\varepsilon_{free} = 6595 \times CFA + 5303 \times S - 370 \times PC - 30030 \times CFA \times S + 7043 \times CFA \times PC - 8303 \times S \times PC \quad [2]$$

where ε_{free} = drying shrinkage at 28 days, μ-strain;

CFA = Class C fly ash content;

S = slag cement content;

PC = Type I portland cement content.

Based on t-tests conducted, S content and the product of CFA and S contents have a statistically significant effect on 28-day drying shrinkage strain. Figure 2 (a) shows that for the S content below 20%, shrinkage decreases as CFA content increases and when the S content is above 20%, the shrinkage tends to increase but not significantly. Figure 2 (b) is a contour plot developed from the response surface analysis to illustrate the observations from response surface plot of Figure 2 (a) in a 2-D view. At high S contents, CFA seems to have a more pronounced effect on the shrinkage behavior as compared to its effect when S content is low (i.e., below 20%).

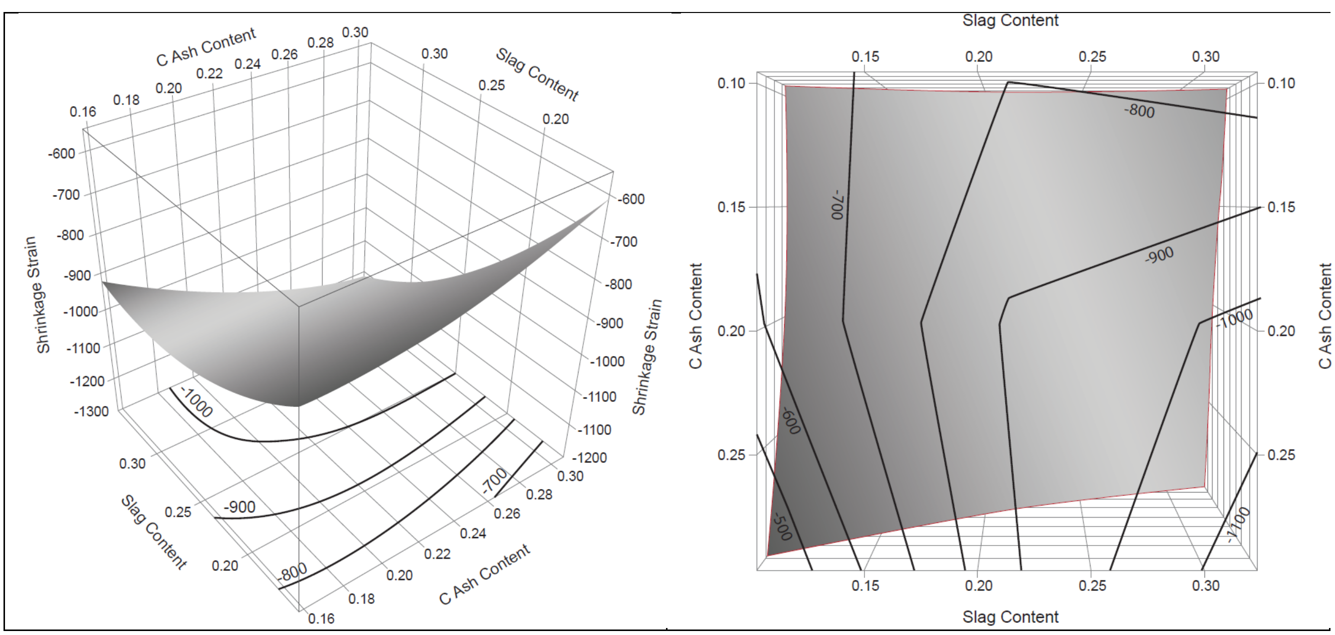

FIGURE 2 (a) response surface plot for series II (b) 2-D contour plot

Ternary Mixtures Series III: Type I Cement, Silica Fume, and Slag

The prediction equation [3] derived for the PC-SF-S blend is given below:

$$\varepsilon_{free} = -7613 \times SF + 10477 \times S + 940.1 \times PC - 2713 \times SF \times S + 5039 \times SF \times PC - 22725 \times S \times PC \quad [3]$$

where ε_{free} = drying shrinkage at 28 days, μ-strain;

SF = silica fume content;

S = slag cement content;

PC = Type I portland cement content.

The R^2 value was determined to be 66%. T-tests found that S and the product of PC and S have statistically significant effects on 28-day drying shrinkage strain. Figure 3 (a) shows that for a given S replacement level up to 30%, shrinkage strain increases with increasing SF content. This is in agreement with the literature that SF increases shrinkage with increased replacement levels [3, 6, 11, 14, 15]. On the other hand, for a given SF replacement similar behavior to CFA as in series I is observed: shrinkage peaks as the S content increases and then decreases as the S percentage further increases. The 2-D contour lines developed from the response surface analysis in Figure 3 (b) shows that S content has a significant effect on drying shrinkage strain compared to SF content. It also provides a quantitative view on how S content affects drying shrinkage at different SF replacement levels. For a given replacement level of S between 15% and 28%, shrinkage increases with the increasing SF content, whereas S content higher than 28%, shrinkage has a decreasing trend with the increasing SF content.

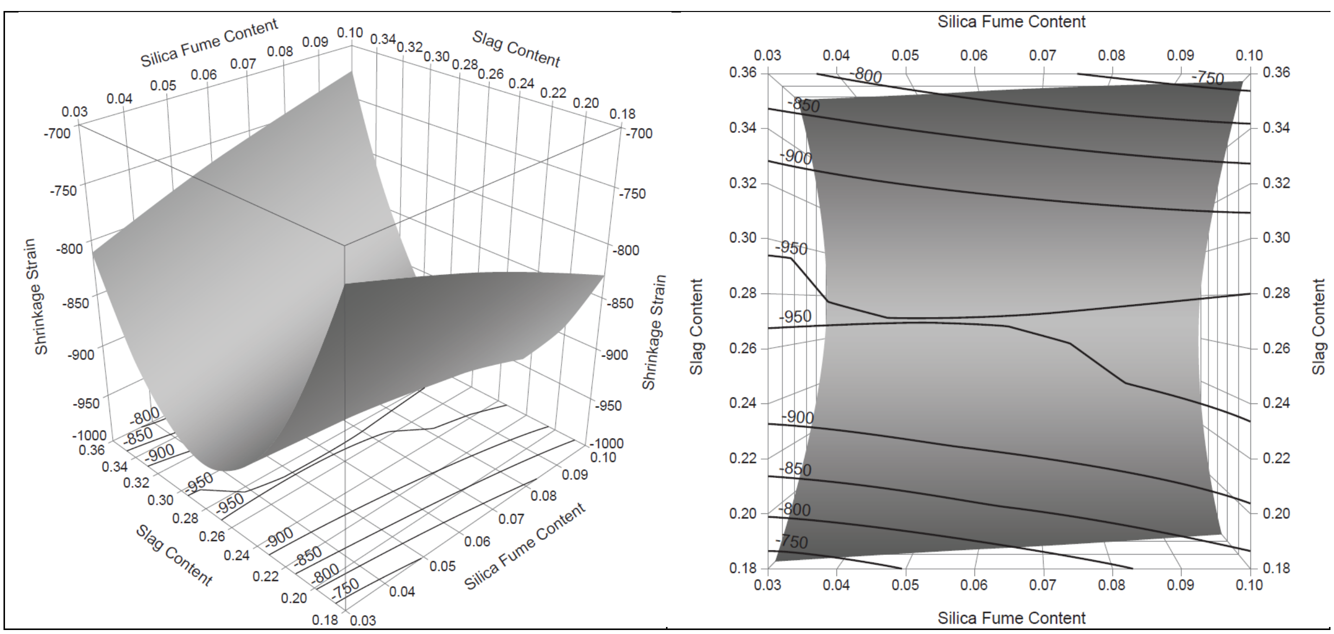

FIGURE 3 (a) response surface plot for series III (b) 2-D Contour plot

Table 4 shows the measured and the predicted 28-day drying shrinkage values for the verification mixtures. The same data is also plotted in Figure 4 together with the measured shrinkages and the predicted values derived from statistical models. Figure 4 implies a good shrinkage prediction by the models as the data are closely scattered around the line of equality except for a couple of outliers.

TABLE 4 28-day Drying Shrinkage Strain and Model Predicted Shrinkage Strain for Mortar Mixtures

ID	Measured shrinkage, μ-strain	Model predicted shrinkage, μ-strain	ID	Measured shrinkage, μ-strain	Model predicted shrinkage, μ-strain	ID	Measured shrinkage, μ-strain	Model predicted shrinkage, μ-strain
I-1	-1192	-1045	II-1	-902	-835	III-1	-798	-818
I-2	-990	-1045	II-2	-769	-835	III-2	-880	-818
I-3	-970	-1045	II-3	-929	-898	III-3	-763	-818
I-4	-1030	-1045	II-4	-867	-898	III-4	-853	-818
I-5	-932	-861	II-5	-807	-885	III-5	-813	-845
I-6	-957	-861	II-6	-978	-885	III-6	-865	-845
I-7	-742	-861	II-7	-700	-708	III-7	-705	-791
I-8	-798	-861	II-8	-802	-805	III-8	-823	-770
I-9	-987	-898	II-9	-920	-934	III-9	-887	-935
I-10	-920	-898	II-10	-937	-934	III-10	-911	-964
I-11	-920	-924	II-11	-898	-913	III-11	-914	-924
I-12	-898	-924	II-12	-925	-913	III-12	-993	-948
I-13	-968	-924	II-13	-762	-761	III-13	-983	-948
I-14	-841	-924	II-14	-708	-708	III-14	-933	-891
I-15	-653	-691	II-15	-712	-708	III-15	-936	-921
I-16	-720	-691	V-II-1	-812	-836	V-III-1	-889	-935
I-17	-938	-913	V-II-2	-960	-1006	V-III-2	-894	-936
I-18	-853	-913	V-II-3	-789	-628	V-III-3	-919	-964
I-19	-730	-736	V-II-4	-992	-1076	V-III-4	-786	-661
I-20	-758	-736						
V-I-1	-1072	-1053						
V-I-2	-926	-861						
V-I-3	-986	-1006						
V-I-4	-1013	-1071						

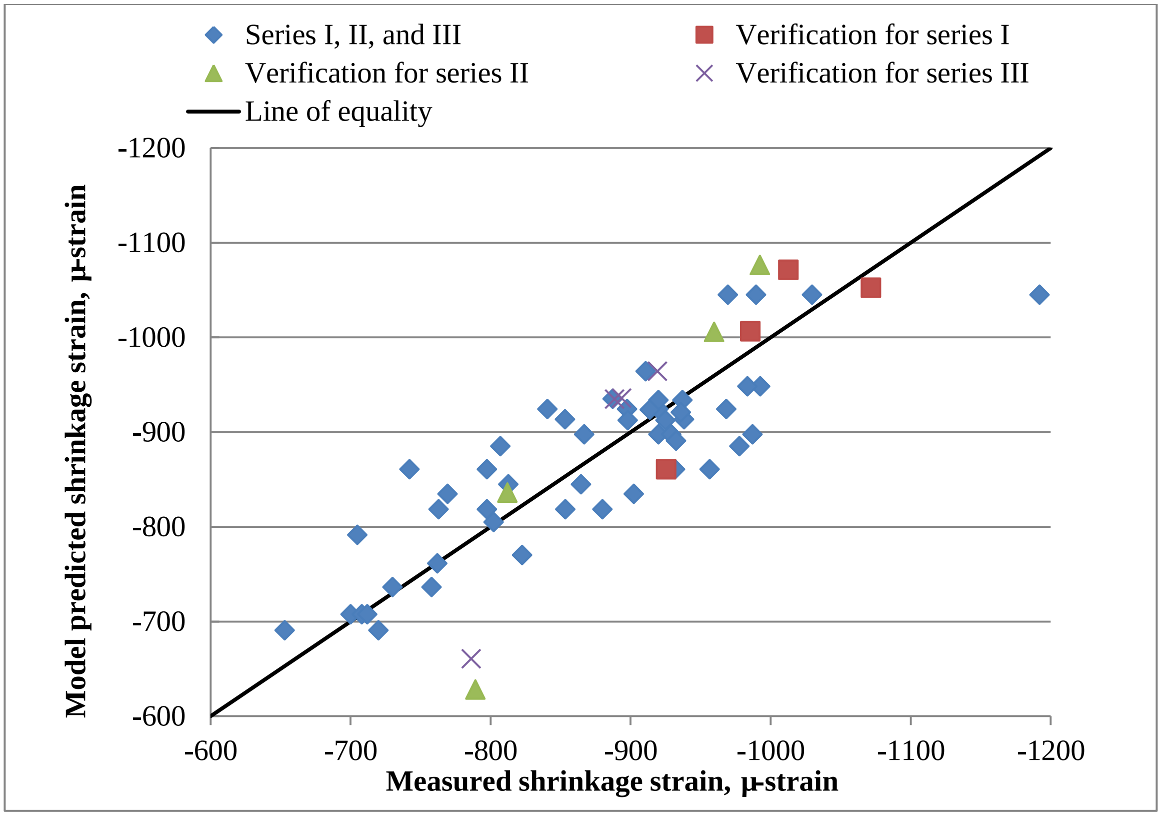

FIGURE 4 designed and verification mixtures shrinkage strain versus model predicted shrinkage strain

The shrinkage strain of plain portland cements mixtures, 100% Type I-1 and Type I-2, are 1178 and 1069 µ-strain, respectively. 49 out of 50 ternary mixtures in phase I and 15 out of 16 in phase II have lower shrinkage values than their corresponding 100% Type I PC.

Effect of CaO/SiO_2 Ratio and Alkali Content on Drying Shrinkage

CaO/SiO_2 ratio and equivalent alkali content (i.e., Na_2O + 0.66 K_2O) of the ternary mixtures were calculated and plotted against shrinkage.

Figure 5 shows the relationship between the 28-day shrinkage and CaO/SiO_2 of the ternary blends. Although the data yields a low R^2, the trend implies that ternary blends with higher CaO/SiO_2 ratio tend to exhibit a lower shrinkage strain. This behavior is consistent with literature: high CaO/SiO_2 ratio leads to reduced size and volume of gel pores in the calcium silicate hydrates (C-S-H). The removal of interlayer water from C-S-H is likely reduced therefore decreasing shrinkage risk [16].

The alkali content for all the mixtures tested is plotted against the 28-day shrinkage strain in Figure 6. Jawed and Skalny [17] have reported that the shrinkage increases with increased alkali content of cement.

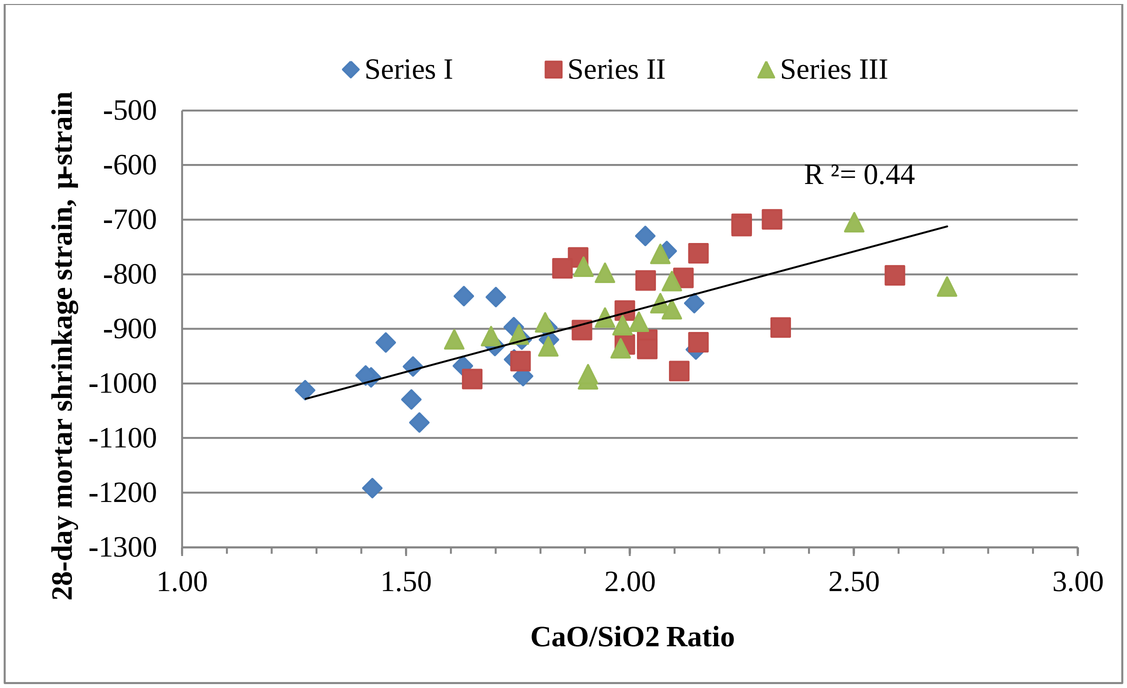

FIGURE 5 the relationship between Ca/Si ratio and 28-day shrinkage strain

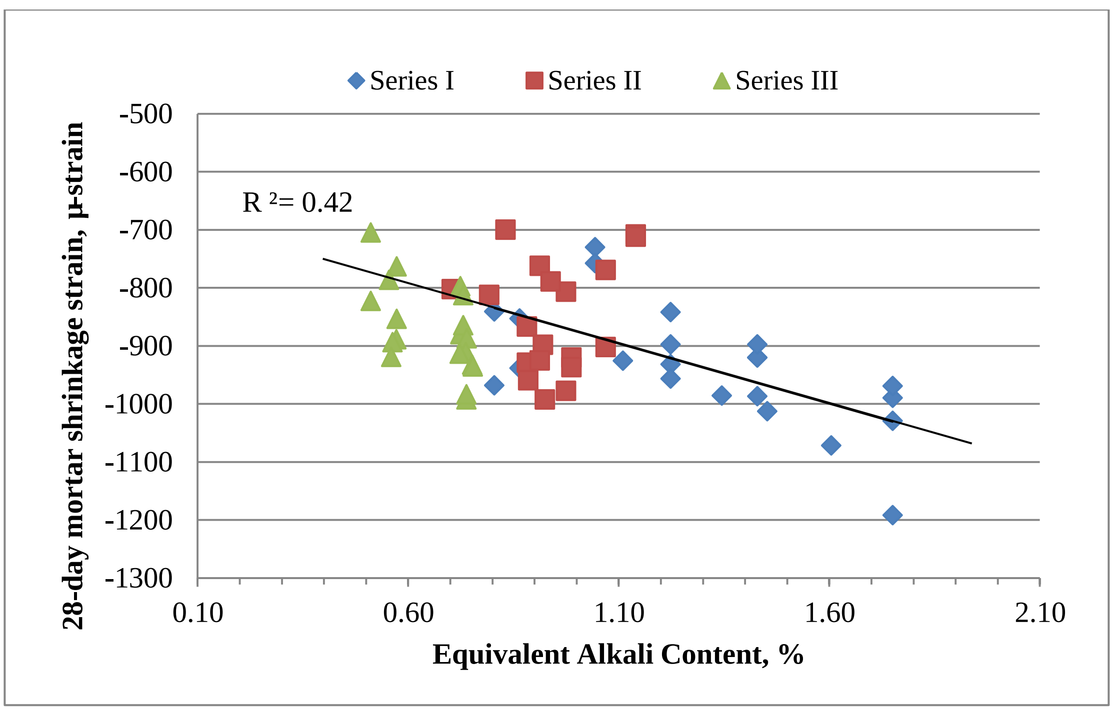

FIGURE 6 the relationship between alkali content and 28-day shrinkage strain

Shrinkage of a cementitious system at a constant water-to-cement ratio is affected by the degree of hydration which might be well affected by SCM reactivity that is determined by particle size distribution and chemistry. CaO/SiO_2 ratio of cementitious material could be an important factor that alters volume of gel pores in C-S-H, hence, drying shrinkage. Pastes containing SCMs might develop smaller sized capillaries (5 to 50 nm) compared to plain portland cement mixtures leading to greater strain for a given volume of water removed.

The composition of the paste is directly related to the chemistry of the ingredients, therefore it is not surprising that there is a trend associated with chemical composition of the mixture, although the model has been developed around types of SCMs that have similar compositions.

The mechanisms of shrinkage are complex and the governing mechanism might depend on numerous factors. This study did not intend to find answers for the mechanism but to approximate shrinkage performance of ternary cementitious blends developing statistical model based on experimental data.

CONCLUSIONS

Shrinkage prediction models were constructed for mortars prepared containing ternary blends of cementitious materials. The prediction models are valid for the combination of cementitious materials within the replacement ranges studied (i.e. slag: 15-35%, class F fly ash: 13-30%, class F fly ash: 15-30% and silica fume: 3-10%). The following conclusions can be drawn from the present study:

1. Slag shows a dominant effect on mortar shrinkage in all three series. An increase of class C fly ash is likely to decrease the mortar shrinkage. Silica fume slightly increases free shrinkage of the mortar in ternary blends selected. An increase of class F fly ash tends to increase the mortar shrinkage. However, this behavior is likely tenuous and class F fly ash shows the least effect on the shrinkage when used together with slag.

2. Although this study pertaining to cementitious material chemistry do not show strong correlation with shrinkage, high CaO/SiO_2 tends to give a higher shrinkage and high equivalent alkali gives a lower shrinkage strain.

3. The shrinkage measurements from a group of verification mortar mixtures have shown a good correlation between the measured shrinkage strain and the strain predicted from the shrinkage model developed from the response surface analysis. Although the mechanisms behind the interaction of cementitious materials in ternary blends are unknown, statistical models could be used to predict the performance.

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