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Residual Strength of Blended Cement Pastes and Mortar Exposed to Elevated Temperatures

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ABSTRACT: This paper presents the outcome of a series of tests to determine properties of blended cement pastes and mortar mixtures using Taguchi's design of experiment approach. Fly ash (FA), water to cementitious material ratio (w/c), superplasticiser (SP) and sand to cementitious material ratio (s/c) are the main parameters considered. Mixture samples were tested at varying temperatures up to 800°C. The ANOVA results and regression analysis show that an increase in FA content and w/c ratio lead to a decrease in the density of blended cement. For compressive strength development, an increase in FA content and s/c ratio decreased the compressive strength development. Within the tested levels of the parameters, the optimum mix to achieve the highest compressive strength of blended cement mixtures is 20% of FA content, 1.5 of s/c ratio, 0.35 of w/c ratio and 0.2% of SP. Increasing the content of FA and SP improved the overall residual strength, and the optimum mix design to achieve the highest residual strength is 20% of FA, 1.5 of s/c ratio, 0.35 of w/c ratio and 0.2 of SP. Based on all the test results, an empirical formula for predicting residual strength of blended cement paste and mortar is proposed.

Keywords: high temperature, cement, blended, design of experimental, anova

1 INTRODUCTION

Blended cement is a hydraulic cement that contains general purpose cement and pozzolan such as fly ash and ground granulated iron blast-furnace slag (GGBF) [1]. Blended cement has been used because of its low cost and improved properties. Fly ash is commonly used in blended cement and the main improvement to strength of blended cement is due to the reaction between fly ash and calcium hydroxide (CH), which is a by-product of hydration of ordinary Portland cement [2, 3]. When blended cement mixes with water, fly ash reacts with water and CH to form Calcium Silicate Hydrate (CSH) [4]. CSH is the main hydration product of OPC, existing around 50 to 70% of the volume of total hydration products, and governs fundamental properties of concrete such as strength [5]. The description of CSH is up to morphology and relationship between the microstructure and macroscopic properties.

Fly ash is a by-product material, there are two types of fly ashes, Class C and Class F, which are classified according to the total percentage of silica dioxide (SiO_2), Aluminium oxide (Al_2O_3) and Iron oxide (Fe_2O_3) [6]. Generally, class C fly ash has calcium oxide contents more than class F fly ash. Sometimes, class C fly ash and class F fly ash are calling 'High calcium fly ash' and 'Low-calcium fly ash', respectively. Nevil [5] noted that when 5 to 15% of fly ash is used by mass, there is a reduction in water demand of concrete for a consistent workability. The main reason for a decrease in water demand of concrete is 'ball-bearing' effect which is ascribed to fly ash due to its spherical shape. At the same time, another mechanism is involved that fine fly ash particles become adsorbed on the surface of the cement particles by electrical charges. Thus, the water demand for given workability is reduced because fine fly ash particles are covering the surface of the cement particles. However, the excess amount of fly ash would be no benefit on water demand. Approximately up to 20% of fly ash content is reported to reduce water demand in concrete.

According to Ramezanianpour [7], class C fly ash has self-hardening properties such as Belite (C_2S), Aluminate (C_3A), Calcium sulphate ($CaSO_4$), Magnesium oxide (MgO) and fee Calcium oxide (CaO). The hydration behaviour of C_2S and C_3A in class C fly ash is the same as that in OPC, but the hydration rate of CSH form is comparatively slow. Somehow, thus, class C fly ash when mixed with water produces hydration products in OPC as AFm ($Al_2O_3Fe_2O_3$-

mono), Aft ($C_3A{\cdot}3CaO{\cdot}SO_3H_{12}$) and CSH. Class C fly ash, however, has little or no self-hardening properties. Class F fly ash only hydrates when alkalis and CH are present. The hydration products such as CSH, C_2ASH_8 and C_4AH are formed and they are produced in the later stage of the hydration process. Generally, fly ash in concrete is to be incorporated because packing action of fly ash particles at the interface of coarse aggregate particles would result in reduced permeability. However, the chemical reactions of fly ash have an effect in improving the microstructure of hydrated cement pates which can lead to the strength development of concrete. Papadakis [2] studied the effect of class F fly ash on OPC system. They found higher strength in concrete when using class F fly ash to replace aggregates or cement. When class F fly ash reacts with $CaOH_2$, it gives higher water content and lower total porosity. Poon et al [8] reported that high strength of concrete with 45% of class F fly ash has a lower heat of hydration and chloride diffusivity than normal plain cement concrete. Class F fly ash in concrete with lower water to binder ratio has better strength contribution. Another research from Papadakis [3] reported that class C fly ash content in concrete directly led to the strength enhancement after the mixing. The reaction of class C fly ash in hydrating cement makes it lower porosity due to the high content of reactive calcium bearing phase in fly ash.

Concrete can be exposed to high temperature during a fire event tor when it is near to furnace and power reactions. During its exposure to high temperatures, the mechanical properties, such as strength and elastic modulus deteriorate and decrease significantly [9]. Physical deterioration process affects the durability of the concrete structure, therefore, is an important issue during and after high temperature exposure. The harmful effects of high temperature on concrete can be minimised by taking a preventive measure such as choosing the right material [10]. However, when OPC is exposed to high temperature, spalling of OPC binder concrete could occur, resulting in a rapid loss of concrete cover, layer by layer, potentially exposing the main steel reinforcement. Therefore, it is beneficial to improve the property of cement binder that carries good resistance to high temperature in term of spalling resistance and strength loss [11]. Chan et al. [12] studied high strength and normal strength concrete exposed to high temperatures in a range between 400 to 1200°C. They found that temperature between 400°C and 800°C was a critical range of a loss of concrete strength. The normal strength concrete loses around 10 to 25% and 80% of its original compressive strength at 400°C and 800°C, respectively. Several researches [13-15] reported that using pozzolan as partial cement replacement could lead to an improvement in temperature resistance properties. Xu et al. [13] studied the influence of high temperature on FA in which OPC was replaced with 0%, 25% and 55% of FA. The residual strength of concrete samples after exposing to 250°C to 800°C were determined and found that a dosage 55% of FA in concrete led to high residual strength than other dosages FA samples. Poon et al. [14] conducted research to investigate the effect of the residual strength and durability on normal and high pozzolanic concrete exposed to high temperatures. They concluded that pozzolanic concrete has better performance than OPC concrete after exposed to high temperatures. The mix containing 30% of FA replacement has maximum relative residual strength. Also, most of spalling occurred between 400°C and 600°C but no spalling was observed in FA concrete. Mendes et al. [15] used slag at 35%, 50% and 65% of replacement OPC and determined the residual strength after exposing to 100°C to 800°C. It was found that the samples with slag showed less damage under elevated temperatures.

Although most research has been conducted to study properties of blended cement, limited research has been studied relationship between parameters. In the present work, an experimental program of blended cement mixtures was designed according to Taguchi's Design of Experiment (DOE) approach, which is an efficient method for investigating optimum design parameters for the required performances. Also, analysis of variance (ANOVA) and regression were conducted to understand the better statistical relationship between properties on different parameters and influence of parameters that contribute to measured variation of properties.

2 EXPERIMENTAL PROGRAM

Taguchi's approach to parameters design provides the design engineer with a systematic and efficient method for investigating optimum design parameters according to the required performance and cost [16]. Taguchi's DOE method identifies the 'signal to noise (S/N)' factors. S/N factors are what causes a measureable product or process characteristic to deviate from its target value [16]. Target value might be:

$$S/N = -10 \times \log_{10}\left(\frac{1}{n}\sum_{i=1}^{n} Y_i^2\right) \quad (1)$$

Larger is better: select when the goal is to the maximum the response, which larger the characteristics gives the better performance. The S/N ratio can be determined as:

$$S/N = -10 \times \log_{10}\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{Y_i^2}\right) \quad (2)$$

Nominal is better: select when the goal is to target the response and it is required to base the S/N ratio on standard deviations only. The S/N ratio can be determined as:

$$S/N = -10 \times \log_{10}\left(\frac{1}{n}\sum_{i=1}^{n}(Y_i - Y_0)^2\right) \quad (3)$$

Y_i is the measured value of each response in Equations (1) to (3). When variability occurs, it is because the physically active in the design and environment that promotes change [17].

In this research, fly ash (FA), sand to cementitious material ratio (s/c), water to cementitious material ratio (w/c) and superplasticiser (SP) are considered in the mix proportions. Four parameters and three levels of test variables were selected as shown in **Error! Reference source not found.**. The standard L_9 (3^4) orthogonal array [17] according the selected parameters was used, as presented in **Error! Reference source not found.**.

Table 1. Variation parameters and levels.

Levels	FA	s/c ratio	w/c ratio	SP
1	0%	0	0.3	0 %
2	10%	1.5	0.35	0.1%
3	20%	2.5	0.4	0.2%

Table 2. Standard L_9 orthogonal array.

Mix	FA	s/c ratio	w/c ratio	SP
1	1	1	1	1
2	1	2	2	2
3	1	3	3	3
4	2	1	2	3
5	2	2	3	1
6	2	3	1	2
7	3	1	3	2
8	3	2	1	3
9	3	3	2	1

In this experimental work, general purpose (Type I) Portland cement, and class F (low calcium) fly ash from Collie in Australia were used to prepare specimens. **Error! Reference source not found.** shows the typical chemical compositions of OPC and fly ash. The Loss on Ignition (LOI) of fly ash for the experiment was 1.53% and the median particle size was 45µm. The sand (SSD: saturated surface dried condition) with a specific gravity of 2.6 was used as fine aggregate. The X-ray diffraction (XRD) patterns of OPC and fly ash are shown in Figure 1. The specimens were cast in 50mm cubic moulds. Blended cement mixtures were cured in ambient curing at 23°C ± 3 until testing. The compressive strength of the specimens were tested at a loading rate of 900N/s with a universal testing machine in accordance with ASTM C109 [18]. Also, the density of the specimen was measured at the age in accordance with ASTM C642 [19]. A naphthalene sulphonate superplasticiser was used to improve workability. The reported results are the average of five samples.

Table 3. Chemical composite of OPC (Type I) and low calcium fly ash (wt. %).

	SiO_2	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO	SO_3
OPC	21.1	4.7	63.6	2.7	-	2.6	2.5
FA	65.9	24.0	1.59	2.87	1.44	-	-

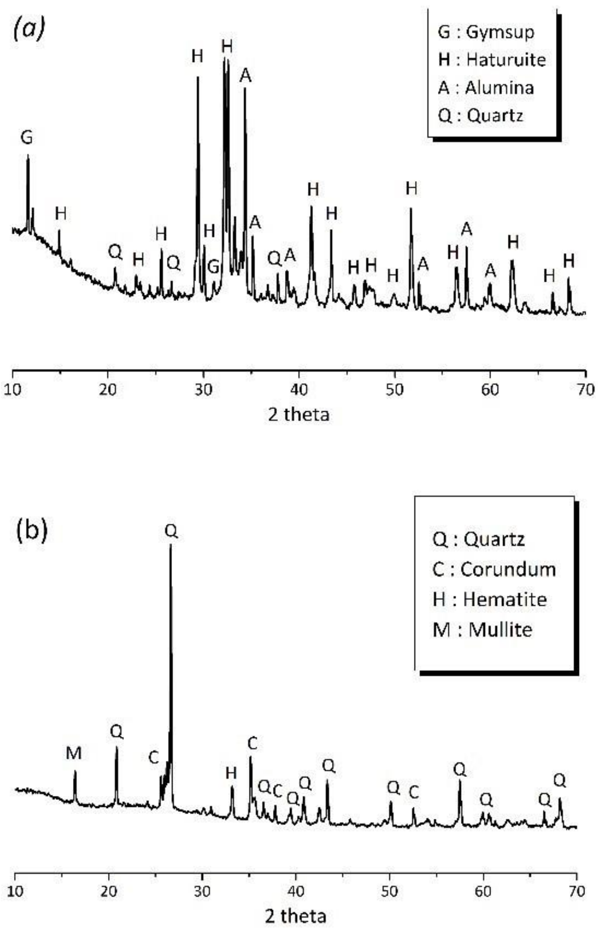


Figure 1. XRD pattern (a) OPC (Type I) (b) class F fly ash (low calcium).

3 RESULTS AND DISCUSSION

3.1 *Density*

Density is important in reducing the self-weight. Figure 2 and **Error! Reference source not found.**

show the average density results of the tested blended cement mixtures. The results indicate that most of the mixtures have fairly uniform density during the 7 to 28 days of curing ages. The paste samples with FA, Mixes 4 and 7, have the lowest density of 1929 kg/m^3 and 1776 kg/m^3, respectively. The mortar samples without FA, Mixes 2 and 3, have the highest density of 2164 kg/m^3 and 2145 kg/m^3, respectively. Overall, the density of all the mixtures slightly decreases after curing for 28 days, with an exception that the mortar samples, Mixes 8 and 9, which contained 20% of FA to cement ratio, have a slight increase in the density during 7 to 14 days of curing age. Based on these results, a statistical S/N ratio analysis was performed to determine the effect of these parameters on the density of the mixtures, as shown in Figure 3, which can be seen that s/c ratio has the most effect on the density at 28 days of curing age. ANOVA was performed on the results from the nine mixtures. ANOVA results indicate that s/c ratio contributed 54% to the density at 28 days of curing age as presented in **Error! Reference source not found.** and **Error! Reference source not found.**. It was observed that increasing FA contents resulted in decreasing the density. The contribution of FA on the density is determined as 24%. The contribution of increasing the w/c ratio on the density is 14%. SP has a minor influence on the density at 28 days of curing age; its contribution is obtained as 5%. As expected, the s/c ratio was the most important parameter on density.

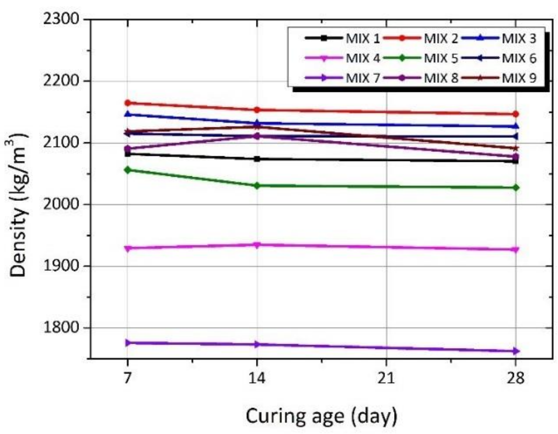


Figure 2. Density with curing ages

Table 4. Density with curing ages (kg/m^3).

Mix	7 days	14 days	28 days
1	2082	2073	2070
2	2164	2153	2146
3	2145	2132	2126
4	1929	1934	1927
5	2056	2030	2027
6	2115	2111	2110
7	1776	1773	1762
8	2090	2111	2077
9	2118	2126	2091

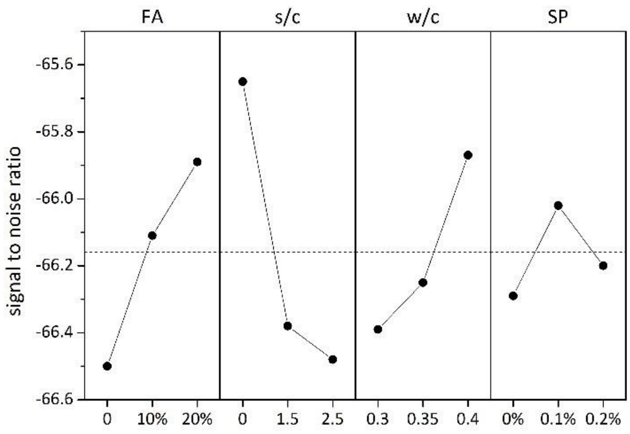


Figure 3. S/N ratio of density at 28 days.

Table 5. Analysis of variance on density.

	Source	DF[a]	SS[b]	MS[c]	Contribution
7 days	FA	2	29447	14724	23.84%
	s/c ratio	2	69882	34941	56.57%
	w/c ratio	2	17432	8716	14.11%
	SP	2	6773	3386	5.48%
14 days	FA	2	22893	11477	18.86%
	s/c ratio	2	68161	34081	56.14%
	w/c ratio	2	23721	11861	19.54%
	SP	2	6626	3313	5.46%
28 days	FA	2	29462	14731	24.82%
	s/c ratio	2	63513	31756	53.51%
	w/c ratio	2	20772	10386	17.50%
	SP	2	4954	2477	4.17%

[a]degree of freedom [b]sum of square [c]mean square

3.2 *Compressive strength*

The compressive strength results are presented in **Error! Reference source not found.** and **Error! Reference source not found.**(a). The average percentage increase in compressive strength at 7 days of curing age is 8% of 28 days compressive strength. The paste mixture Mix 1 has the minimum compressive strength 87.8 and 94.5 MPa at 7 days and 28 days of curing age, respectively. The mortar mixture Mix 5, with 10% of FA, 0.15 of s/c ratio, 0.4 of w/c ratio and no SP has the minimum compressive strength of 43.6 MPa and 54.5 MPa at 7 days and 28 days of curing age, respectively. **Error! Reference source not found.**(b) shows the compressive strength development between 7 days to 28 days of curing age. Mix 4 has the highest compressive strength gain between 7 to 28 days of curing age with 21.62 MPa. The compressive strength gain in Mix 6 between 7 to 28 days is the lowest, which is

4.31 MPa. Therefore, Mix 5 has minimum compressive strength but the highest compressive strength gains between 7 to 28 days of curing ages.

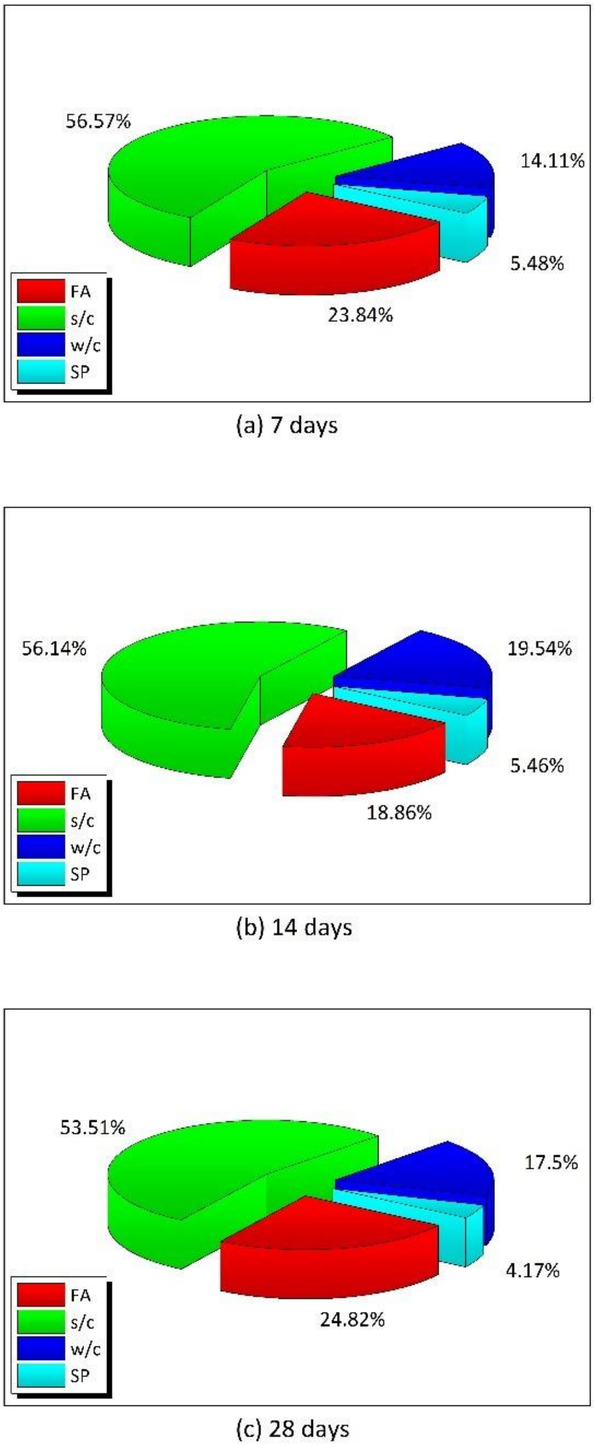


Figure 4. Contribution of experimental parameters on density

Table 6. The results of compressive strength (MPa).

Mix	7 days		14 days		28 days	
	$f_{ck,cubic}$	StD	$f_{ck,cubic}$	StD	$f_{ck,cubic}$	StD
1	87.8	2.4	91.3	1.7	94.5	2.2
2	63.5	1.5	67.4	3.5	72.8	4.3
3	53.2	1.6	58.7	2.6	62.6	2.8
4	59.5	5.6	73.3	4.6	81.2	3.4
5	43.6	3.4	51.6	2.4	54.5	2.7
6	52.0	1.3	55.2	2.2	56.3	1.8
7	51.3	3.1	58.6	2.6	58.7	3.0
8	54.7	2.9	65.0	4.1	67.1	6.9
9	47.6	2.0	58.4	2.1	59.5	3.5

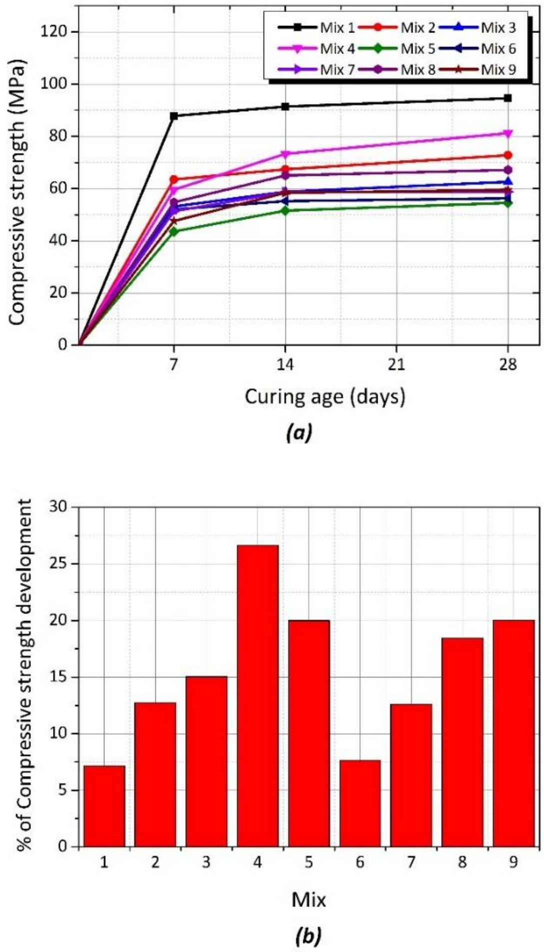


Figure 5. (a) Compressive strength development and (b) percentage of compressive strength development between 7 to 28 days.

The typical XRD pattern of OPC paste (Mix 1) is as presented in **Error! Reference source not found.**. Highly noticeable peaks are CSH, which is main hydration products of the cementing compound, CH and C_2S. Due to curing ages, XRD pattern indicates that CSH phase increase. Scanning Electron Microscope (SEM) also shows the growth of hydration products as fabric structures when curing proceeds as presented in **Error! Reference source not found.**.

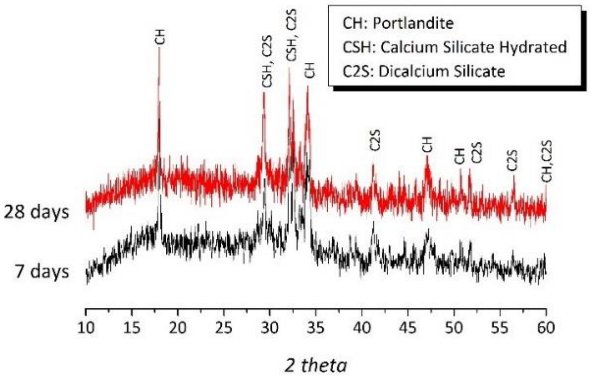


Figure 6. XRD pattern of OPC paste (Mix 1) 7 and 28 days.

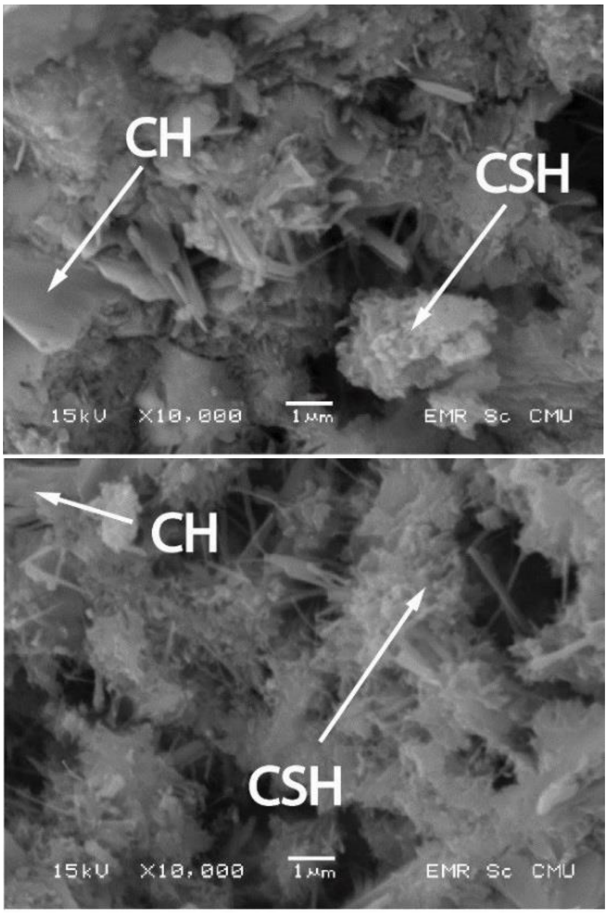


Figure 7. SEM image of OPC paste (Mix 1).

The S/N ratio graphs of parameter for compressive strength at 7 days and 28 days of curing age are shown in Figure 8. FA has the most significant effect on 7 days compressive strength. On 28 days compressive strength, however, s/c ratio is the most affecting parameter. ANOVA results show s/c ratio has 41.56% contribution on 7 days and 39.59% contribution on 28 days, as shown in **Error! Reference source not found.** and Figure 9. The effect of SP on 7 days and 28 days compressive strength is 2.32% and 7.64% contribution, respectively. The S/N ratio of FA and s/c at 7 days and 28 days of curing age results show similar patterns. The S/N ratio of w/c ratio shows that w/c ratio of 0.3 to 0.35 has not significant effect on 28 days compressive strength. ANOVA results of sample containing w/c ratio present mostly similar contributions of curing ages which are in range 25.32% and 27.49% between 7 days to 28 days. Therefore, an increase in FA, s/c and w/c ratio in blended cement mixtures lead to a decrease in the compressive strength.

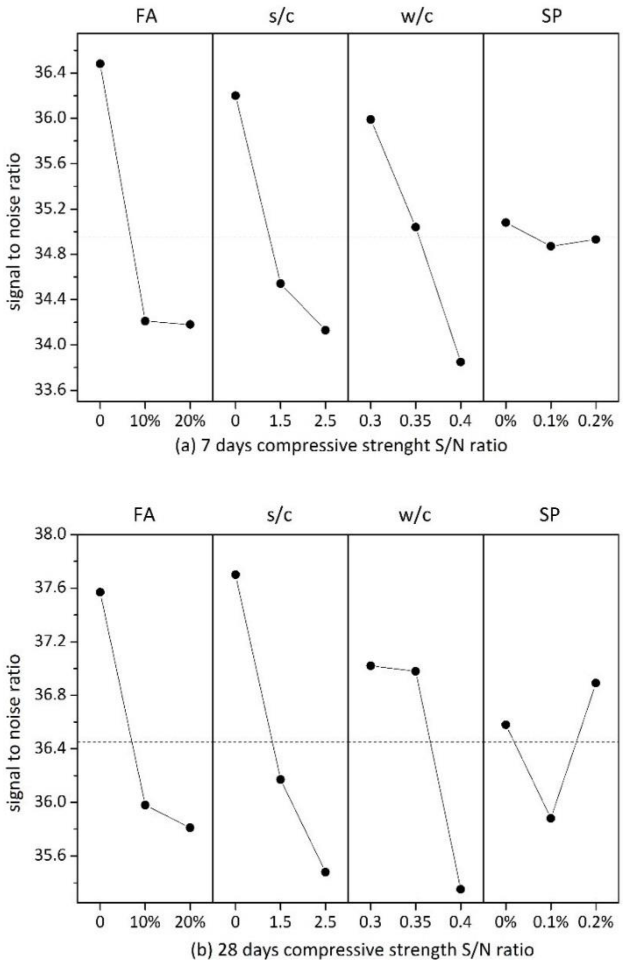
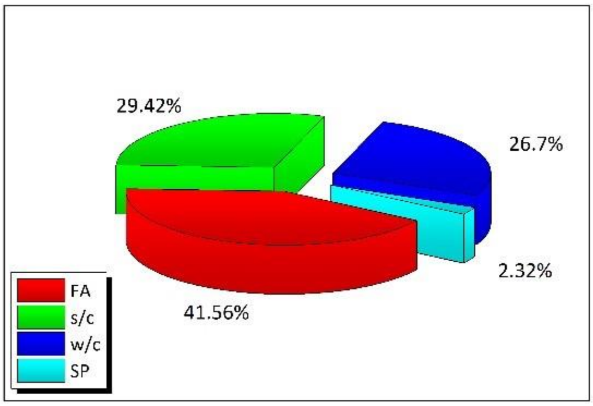


Figure 8. S/N ratio of compressive strength.

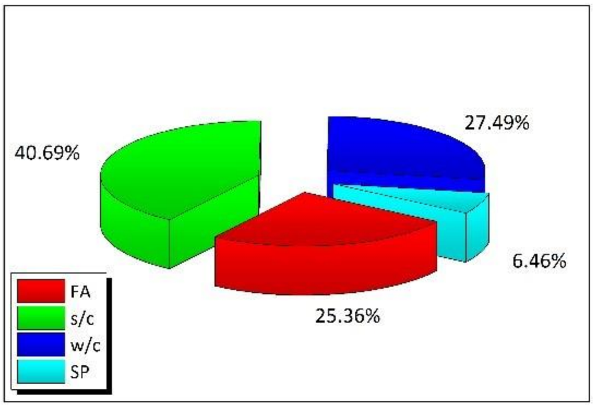
Table 7. Analysis of variance on compressive strength.

	Source	DF[a]	SS[b]	MS[c]	Contribution
7 days	FA	2	557.51	278.75	41.56%
	s/c ratio	2	394.68	197.34	29.42%
	w/c ratio	2	358.14	179.07	26.70%
	SP	2	31.18	15.59	2.32%
14 days	FA	2	295.47	474.02	25.36%
	s/c ratio	2	474.02	237.01	40.96%
	w/c ratio	2	320.24	160.12	27.49%
	SP	2	75.28	37.64	6.46%
28 days	FA	2	385.4	192.69	27.45%
	s/c ratio	2	555.9	277.95	39.59%
	w/c ratio	2	355.6	177.78	25.32%
	SP	2	107.3	53.67	7.64%

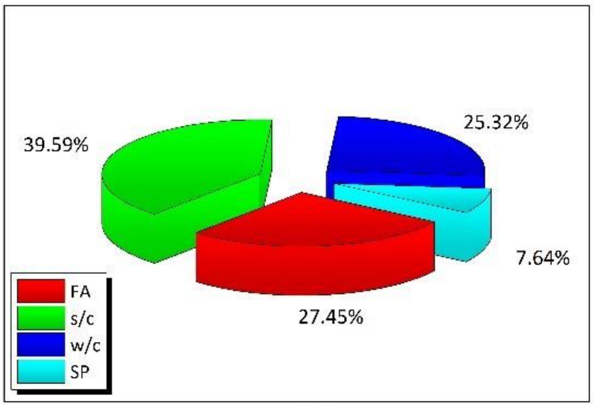
[a]degree of freedom [b]sum of square [c]mean square



(a) 7 days



(a) 14 days



(c) 28 days

Figure 9. Contribution of experimental parameters on compressive strength.

The results of S/N ratio on compressive strength gain between 7 days to 28 days of curing ages indicate that SP is the most significant parameter as shown in **Error! Reference source not found.**. The s/c ratio has minor effect on the compressive strength gain. ANOVA results reveal that the contribution of SP on compressive gain is 44.01% and s/c ratio is 9.19%, as shown in **Error! Reference source not found.**. The optimal mixing design for compressive strength gain on blended cement mixture is 20% of FA, 1.5 of s/c ratio, 0.35 of w/c ratio and 0.2% of SP.

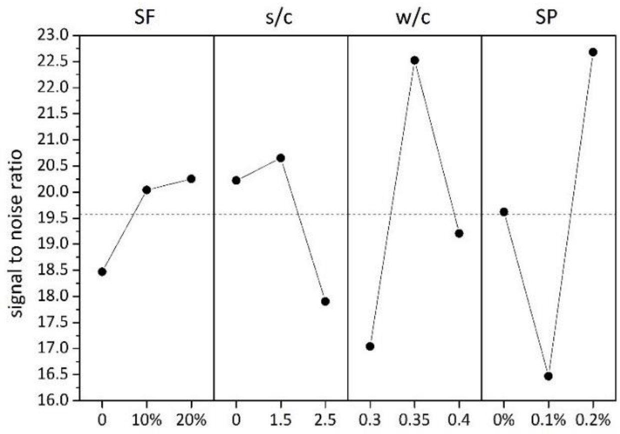


Figure 10. S/N ratio of compressive strength gain.

Table 8. Analysis of variance on compressive strength gain.

Source	DF[a]	SS[b]	MS[c]	Contribution
FA	2	21.7	10.85	11.19%
s/c ratio	2	17.83	8.915	9.19%
w/c ratio	2	69.09	34.546	35.61%
SP	2	85.37	42.687	44.01%

[a]degree of freedom [b]sum of square [c]mean square

3.3 *High temperature exposure*

High temperature exposure test was conducted on selected five specimens of each mixture as shown in **Error! Reference source not found.** and Figure 11. After 28 days curing the samples were dried in an oven at 105 ± 5°C for 24 hours then, the specimens were placed in an electric kiln to be heated up to maximum temperature of 200, 400, 600, or 800°C with 2 hours holding time. The heating rate was 10°C per minute. After that, the specimens were allowed to cool down gradually to room temperature before testing to determine the compressive strength. The test results show that each temperature range has a distinct pattern of strength gain or loss. It is observed that there is a significant increase in strength around 18.3 MPa when mixtures are exposed to 200°C. This increase could be due to the hydration of unhydrated particles which were activated as a result of temperature rise [9]. The maximum and minimum residual strength of Mix 1 and Mix 5 are 112.8 and 56.8 MPa, respectively, when exposed to 200°C. With Mix 1, a significant decrease in the strength is obtained when exposed to between 600 to 800°C.

Eurocode 4 Standard [20] provides residual strength reduction factors of normal weight concrete at elevated temperatures, as shown in Figure 12. The results of residual strength reduction factors illustrate that the obtained experimental results in the present work have high residual strength reduction factors compared to European standard. However, the overall trend of residual strength is similar in a range of 1.12 to 0.33 exposed to between 600 to 800°C.

The XRD pattern of OPC paste (Mix 1) is shown in Figure 13, where main peaks have been identified. Typical peaks are associated to CH, CSH and C_2S. After Mix 1 was exposed to high temperatures, some peaks disappeared or reduced in the intensity. It is clear that in samples exposed to 600°C compared to 23°C, the main hydration products such as CH and CSH disappeared. However, between 23°C to 400°C high temperatures, the intensity of main peaks in XRD patterns are not clear. The reduction and dehydration of CSH can explain that CSH hydration products reform to CaO but in less proportion [13, 21, 22]. The SEM images of Mix 1 show that there is a limited amount of fabric structures in the mixture after exposed to 600°C compared to the image of the mixture at 23°C, as shown in Figure 16.

Table 9. Residual strength (mean) of blended cement mixture (MPa).

Mix	ambient	200°C	400°C	600°C	800°C
1	94.5	112.8	97.5	57.0	-
2	72.8	81.3	66.2	44.2	25.7
3	62.6	66.7	49.9	38.7	21.3
4	81.2	86.3	76.1	48.1	31.7
5	54.5	56.8	42.4	28.7	16.4
6	56.3	61.9	49.2	36.2	19.9
7	58.7	67.2	64.4	35.7	24.8
8	67.1	84.9	71.8	51.1	29.6
9	59.5	66.0	52.7	35.4	20.3

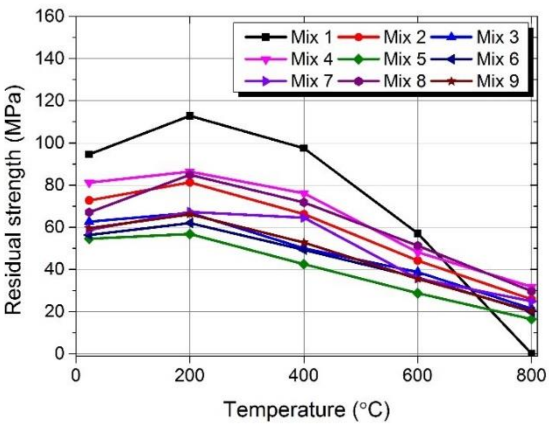


Figure 11. Residual strength of blended cement mixtures.

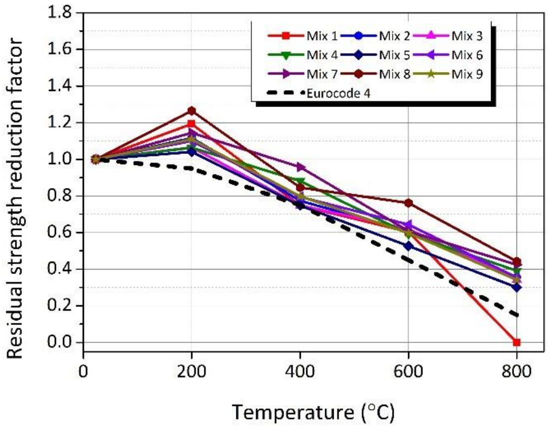


Figure 12. Residual strength reduction factors

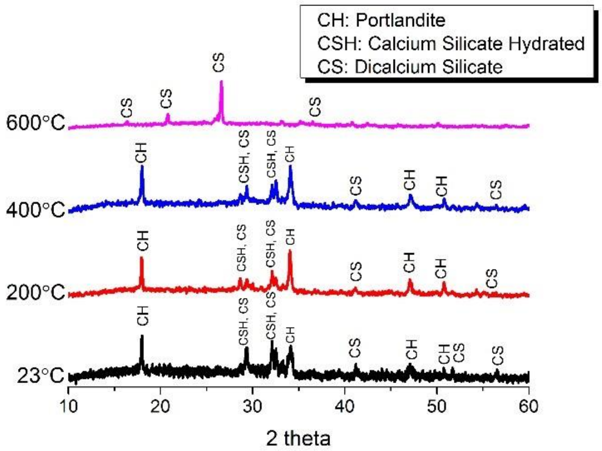
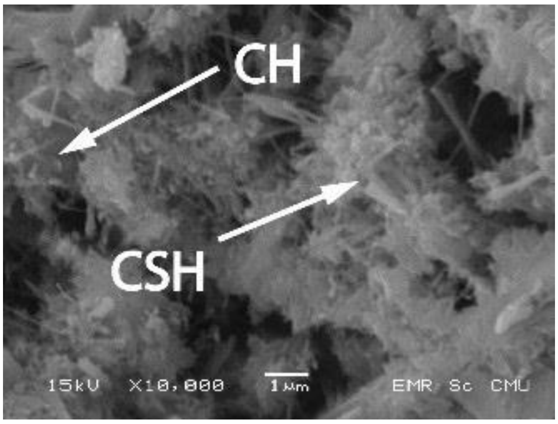


Figure 13. XRD pattern of OPC paste (Mix 1).



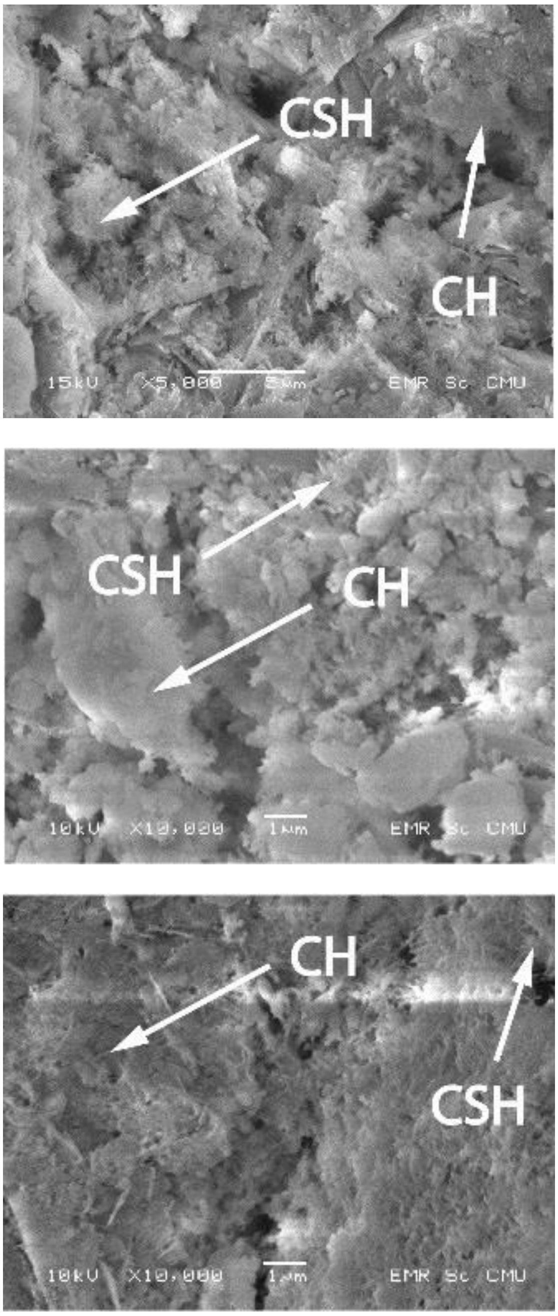


Figure 14. SEM image of OPC paste (Mix 1) after exposed to high temperatures; ambient, 200°C, 400°C, 600°C and 800°C from top to bottom.

The S/N ratio is analysed for residual strength at 200°C, 400°C, 600°C and 800°C and plotted in **Error! Reference source not found.**. It is observed that SP has the most effect on the residual strength of blended cement after high-temperature exposure. From ANOVA results, the contribution of SP on the residual strength is determined to be 54.90%, as shown in **Error! Reference source not found.**. The s/c ratio has the minor effect on residual strength with 5.79% contribution. FA has the second most significant influence on the residual strength with 20.07% contribution. The contribution of w/c ratio is determined as 19.24%. Generally, the increase of w/c decreases the residual strength. This is well known as the release of water vapour could influence the chemical composition of hydration products known as dehydration process [22]. However, it was found that reducing w/c ratio would reduce the overall residual strength, especially after exposed to 800°C high temperature. Between the exposed temperature ranges 23°C to 600°C, reducing w/c ratio would increase the residual strength but after exposed to 800°C would decrease the residual strength. Therefore, based on the S/N ratio results of the residual strength, the optimal mixing design of blended cement mixture is 20% of FA, 1.5 of s/c ratio, 0.35 of w/c ratio, and 0.2% of SP. The optimal mixing design can be determined with the predicted S/N ratio function as [23]:

$$\eta_{predic} = \overline{\eta} + \sum_{i=1}^{f} \overline{\eta}_i - \overline{\eta}_0 \quad (4)$$

where $\overline{\eta}$ is overall mean of S/N ratio, f is number of factor, and $\overline{\eta}_i$ is the mean of S/N ratio at the optimal level of each factor. The S/N ratio of optimal mix design and maximum and minimum parameter levels are determined as presented in **Error! Reference source not found.**. With the predicted S/N ratio, the residual strength is calculated by Equation **Error! Reference source not found.**, as shown in Figure 16. The residual strength of the minimum parameter levels (P2) has high residual strength between 23°C to 600°C. After exposed to 800°C, however, significant loss in residual strength is indicated.

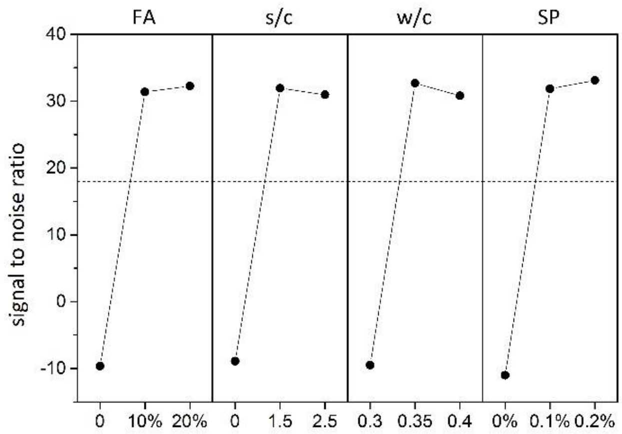


Figure 15. S/N ratio after exposed to high temperatures.

Table 10. Analysis of variance on high temperature exposure.

	Source	DF[a]	SS[b]	MS[c]	Contribution
200°C	FA	2	566.3	283.14	23.59%
	s/c ratio	2	871	435.49	36.29%
	w/c ratio	2	808	404.01	33.66%
	SP	2	155.1	77.54	6.46%
400°C	FA	2	153.09	76.55	9.71%
	s/c ratio	2	916.17	458.09	53.10%

	w/c ratio	2	418.82	209.41	26.56%
	SP	2	88.93	44.47	5.64%
600°C	FA	2	124.79	62.39	19.06%
	s/c ratio	2	155.13	77.57	23.70%
	w/c ratio	2	287.23	143.62	43.87%
	SP	2	87.54	43.77	13.37%
800°C	FA	2	139.01	69.50	20.07%
	s/c ratio	2	40.08	20.04	5.79%
	w/c ratio	2	133.29	66.64	19.24%
	SP	2	380.31	190.16	54.90%

[a]degree of freedom [b]sum of square [c]mean square

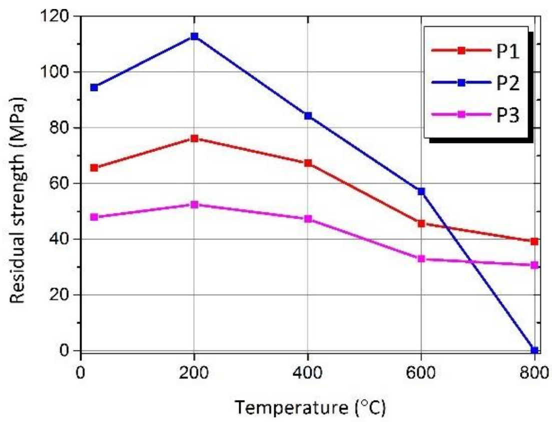


Figure 16. S/N ratio after exposed to high temperatures.

Table 11. Predicted S/N ratio.

Index		FA	s/c ratio	w/c ratio	SP	S/N
P1	Optimal	20%	1.5	0.35	0.2%	75.9
P2	Min level	0%	0	0.3	0%	-93.0
P3	Max level	20%	2.5	0.4	0.2%	73.1

3.4 *Regression analysis*

Regression analysis is a statistical tool for determining relationships between variables. One important reason to determine the regression model is to uncover causes by studying the relationship between variables. Sometimes, statistical relationship does not necessarily imply causal relationship but the presence of relationship can give a good starting point for research. If statistical confidence is indicated with the regression model, then values of the explanatory variables can be used to predict the output variables. Another reason for regression analysis is to examine the test hypotheses [24]. In this study, therefore, regression analysis was used as a tool for determining the relationship between variables and to predict values outside of parameters ranges of explanatory variables. Through linear regression analysis of the density and the compressive strength test results, the empirical relationship of blended cement mixtures can be obtained as:

$$\begin{aligned}\rho_{28} &= 1747 + 5478.9x_1 + 44.308x_2 + 1086.8x_3 \\ &- 43947x_4 + 209.85x_1x_2 - 18486x_1x_3 \\ &- 73.85x_2x_3\end{aligned} \quad (5)$$

$$\begin{aligned}f_{ck,cubic,28} &= 199.11 - 745.64x_1 - 49.188x_2 \\ &- 347.35x_3 + 9736.9x_4 + 79.579x_1x_2 \\ &+ 1716.3x_1x_3 + 106.24x_2x_3\end{aligned} \quad (6)$$

Similarly, the empirical relationship between the residual strength and parameters from high temperature exposure testing can be written as:

$$\begin{aligned}R_{(t)} &= C_0 + C_1x_1 + C_2x_2 + C_3x_3 + C_4x_4 + C_5x_1x_2 \\ &+ C_6x_1x_3 + C_7x_2x_3\end{aligned} \quad (7)$$

where x_1, x_2, x_3, x_4 denote FA, s/c ratio, w/c ratio and SP, respectively, and the corresponding coefficients, C_0, C_1, C_2, C_3, C_4, C_5, C_6 and C_7 are as shown in **Error! Reference source not found.**. $R_{(T)}$ is the residual compressive strength of a blended cement mix after exposed to temperature *T* in Celsius.

Table 12. Coefficients of empirical relationship in Equation (7).

Coefficient	Temperature (*T*)			
	200°C	400°C	600°C	800°C
C_0	325.51	329.07	165.98	-180.6
C_1	-475.71	-767.82	-153.39	270.16
C_2	-90.57	-85.59	-44.62	63.617
C_3	-706.61	-769.5	-361.94	603.37
C_4	4435.1	6791.2	4038.6	9894.7
C_5	-8.72	-15.832	-9.06	107.97
C_6	1420	2355.7	540.29	-1258.6
C_7	242.49	230.12	121.57	-217.66

Validation of predictive model, Equation (7), can be carried out with corrected coefficient [25]. The information about regression analysis is presented in **Error! Reference source not found.**. It can be seen that corrected coefficient values of density and compressive at 28 days are 0.980 (98.0%) and 0.983 (98.3%), respectively. The corrected coefficient values of residual compressive strength at 200, 400, 600 and 800°C are 0.957 (95.7%), 0.955 (95.5%), 0.952 (95.2%) and 0.952 (95.2%), respectively. These corrected coefficients indicate that empirical model states a good fit of the model being validated.

It is identified affecting parameters and optimal mix proportion of blended cement for high temperature exposure. The proposed optimal mix proportion when designed for high temperature exposure is: 20% of FA, 1.5 of s/c ratio, 0.35 of w/c ratio and 0.2% of SP. It should be noted that the proposed empirical model for predicting the residual compressive strength $R_{(T)}$ is based on cubic strengths, which are generally higher than uniaxial compressive strength [20]. The correct empirical cylindrical compressive strength can be determined approximately by:

$$f_{ck} = 0.4381 \cdot f_{ck,cubic}^{1.135} + 3.123 \quad (8)$$

where f_{ck} is a residual uniaxial cylindrical compressive strength, $f_{ck,cubic}$ is a cubic compressive strength.

4 CONCLUSION

Based on the results of the investigation conducted on blended cement mixtures using Taguchi's design of experiment approach, it was found that an increase in FA content and w/c ratio lead to a decrease in the density of blended cement. SP has a minor effect on the density of blended cement mixtures. The optimal mix design was obtained as: 20% of FA, no content of s/c ratio, 0.4 of w/c ratio, and 0.1% of SP. For compressive strength development, an increase in FA content and s/c ratio decreased the compressive strength development. The optimisation of the compressive strength of blended cement mixtures was found to be 20% of FA content, 1.5 of s/c ratio, 0.35 of w/c ratio and 0.2% of SP. XRD and SEM analysis confirms that the main hydration products of OPC paste is CSH phase, which increases with curing ages. SEM image also show growth of hydration products after curing processes. With respect to resistance to high temperature, SP was identified as the most significant parameter, and FA had the second most effect on the mixtures. Increase in FA and SP was found to improve the overall residual strength, and the optimisation of mix design was 20% of FA, 1.5 of s/c ratio, 0.35 of w/c ratio and 0.2 of SP. XRD and SEM analysis results also confirmed the dehydration process.

Taguchi's design of experiment approach was successfully applied as a useful tool in studying the influence of parameters in cementitious matrices. The results can be analysed using the ANOVA technique to examine the variation in the measured properties of blended cement paste and mortar. Based on the test data, and the statistical and regression analysis, an empirical model has been developed for predicting the ambient compressive strength and the residual compressive strength of blended cement mixtures after exposed to elevated temperature.

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