

Strength estimation model for high-strength concrete incorporating metakaolin and silica fume

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ABSTRACT

A mathematical model is presented for estimating compressive strength of high-strength concrete incorporating pozzolanic materials, based on the strength of a control OPC concrete made with similar mixture characteristics and curing history. In this study, metakaolin and silica fume were used as cement replacement materials at 5, 10 and 15% by mass. Water-cementitious materials ratios varied from 0.27 to 0.33, and strength testing was conducted up to an age of 180 days. It was found that the strength of a pozzolanic mixture could be related to the strength of its equivalent control by a linear function. Key parameters involved in the model are the pozzolanic and dilution factors, which can be correlated to the pozzolan content in the mixture. The study concludes that the accuracy of the model increases with concrete age. At ages 28 days and above, 97% of the estimated strengths are within $\pm 5\%$ of the actual value.

Keywords: Compressive strength (C); high-performance concrete (E); metakaolin (D); silica fume (D); strength estimation

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1. Introduction

Speed is one of the most crucial factors in determining the profitability of a construction project. Hence, more often than not, contractors are pressured to remove structural formwork in the shortest time possible; that is immediately after the concrete is assumed to have gained sufficient strength to safely support its self-weight and additional loads from construction machineries. However this practice can compromise safety and catastrophic construction failures resulting from inaccurate estimates of in-situ concrete strength are well documented. This gives rise to increased emphasis on the need for accurate concrete strength estimation.

Concrete gains strength gradually as a result of chemical reaction between cement and water; and for a specific concrete mixture, strength at any age is related to the degree of hydration. Since the rate of hydration is a function of temperature, the strength development of a given concrete depends on its time-temperature history, assuming that sufficient moisture is available for hydration. This is the basis of the maturity concept, which was developed in the early 1950s to assess the development of in-situ concrete strength during construction. According to this concept, strength of hardening concrete can be estimated at any age by computing the “maturity” based on the temperature-time history of the concrete [1]. The maturity rule proposed by Saul [2] states that specimens of concrete of the same mixture will have equal strengths if they have equal maturity values, even though their temperature histories may differ. By using a maturity function, the measured temperature history of the concrete is converted to a numerical index, which indicates the extent of strength development. Concrete strength is then estimated based upon the measured maturity index and the strength maturity relationship for that particular concrete mixture [3]. ASTM C 1074 [4] provides a standard practice for using maturity to estimate concrete strength.

In the case of high-performance concrete, however, strength development is more complex in nature due to the combined physiochemical effects of pozzolans in concrete. The physical influence is in the refinement of pore structure of the cement paste, while the chemical phase consists of the pozzolanic reaction, which replaces C-H crystals with cementitious C-S-H gel. However, partial replacement of cement in concrete by pozzolans can produce an immediate dilution effect, which will cause early concrete strength to reduce in approximate proportion to the degree of replacement [5]. In this paper, an investigation is carried out to relate the strength of concrete mixtures made with pozzolans to the strength of the OPC control mixture. The parameters involved in this model are the pozzolanic and dilution factors, which depend on the amount of pozzolanic material present in the mixture. The key feature of this model is in its simplicity; since other factors relating to water-cementitious materials ratio (w/cm), age, cement content and temperature can be disregarded because both the pozzolanic and control mixtures have similar material proportions and are assumed to have undergone the same curing history.

2. Experimental procedure

2.1 Materials and mixture proportions

Ordinary portland cement (ASTM Type I), commercial densified silica fume, and laboratory produced metakaolin was used. Metakaolin was obtained by calcination of raw Malaysian kaolin at 700°C for 7 hours, using a rotary electric furnace. Physical properties and chemical composition of the cementitious materials are given in Table 1 and Table 2 respectively. The coarse aggregate was crushed granite with 10-mm nominal maximum size, and the fine aggregate was a medium graded (BS 882: 1992) siliceous sand. Specific gravities for the coarse and fine aggregate were 2.57 and 2.65 respectively. A polycarboxylic ether

based superplasticizer with 20% solids and specific gravity of 1.05 was used. Mixing and curing water was taken directly from a tap supply at a temperature of approximately 28°C.

Twenty-one concrete mixtures were proportioned using the Sherbrooke mix design method [6]. The mixtures were divided into series A, B and C with free w/cm of 0.27, 0.30 and 0.33 respectively. The pozzolans were used to replace 5, 10, and 15 percent of the mass of cement at each w/cm . Total cementitious materials content for all mixtures was 500 kg/m³, while coarse aggregate content and sand-to-total aggregate ratio was 1050 kg/m³ and 0.4 respectively. Superplasticizer dosages for Series A, B and C were 1.8%, 0.8% and 0.5% by mass of cementitious material content respectively. Mixing water was adjusted to correct for aggregate absorption and for the additional water brought into mixture from superplasticizer. Mixture proportions are summarised in Table 3.

2.2 Mixing and curing

A pan mixer was used. Fine aggregate and cement were mixed first, followed by the addition of pozzolan and coarse aggregate. Materials were mixed dry for a period of 1½ minute. Three quarters of the mixing water was then added while the materials were being mixed, followed by superplasticizer and finally the remaining water. Wet mixing was continued for a total period of 5 minutes.

Cube specimens were moulded using 100 by 100-mm steel moulds and compacted in three uniform layers by means of vibrating tables. The amount of vibration required to ensure good compaction was adjusted based on the Vebe time of the fresh concrete. Forty cube specimens were prepared for each mixture. After casting, specimens were covered with wet burlap to prevent moisture loss, and were stored in the laboratory at ambient temperature of 28°C and 75% relative humidity. After 24 hours, specimens were demoulded and cured in a water tank, under room temperature until the day of testing.

2.3 Strength testing

Compressive strength tests (BS 1881: Part 103: 1983) were performed on 100-mm cube specimens at ages of 1, 3, 7, 28, 56, 90 and 180 days, using a 2000-kN compression-testing machine with a digital load display. Testing was conducted immediately after specimens were removed from the curing tank. Specimen dimensions and masses were measured to check for any gross fabrication error. While waiting to be tested, specimens were covered with wet burlap to maintain a wet condition. At least three specimens were tested at each age to compute the average strength. Additional specimens were tested if any individual strength result deviated substantially from the mean. A new average was computed based on the three closest strength results.

3. Test Results

3.1 Data analysis

The average coefficient of variation for all strength results was found to be approximately 1%. This low variation indicates reliability of the results, which is attributable to a good control of the materials used, and adhering to standard concreting and testing procedures. Fig. 1 shows the strength of the MK and SF mixtures plotted against the strength of the control mixture. It is observed that the strength of a mixture with pozzolan is almost a linear function of the strength of the control mixture. Subsequently, the best-fit linear equation for each case was determined using simple regression analysis based on least squares method. The coefficient of determination, r^2 , for each linear equation was found to be very close to unity, indicating that the linear model is a good description for the relationship between the two variables. The lowest r^2 value obtained from the regression analysis was 0.96. A total of 147 sets of data were used in the analysis.

The r^2 value provides an index of the degree to which a set of plotted points clusters about the regression line. The closer the points fall along the regression line, the larger the value of r^2 and the greater the proportion of the total sum of squares accounted for by the linear regression of Y on X [7]. However, to be reasonably confident that the two variables are, in fact related, further statistical analysis is required. A confidence test was performed to investigate whether or not a significant direct relationship exists between the strength of a pozzolanic mixture and its control, the results of which are shown in Table 4. Standard errors are estimates of uncertainties in the regression coefficients. The t-statistic tests the null hypothesis that the regression coefficient is zero, that is, the independent variable does not contribute to estimating the dependent variable while P value is the probability of falsely rejecting the null hypothesis. From the results, it is concluded that the relationship is significant at 95% confidence.

Another statistical tool to evaluate the suitability of the linear model is by observing residuals plotted as a function of the control strength. Residual plot is a standard tool used to diagnose non-constant variance, curvature and outliers [8]. If the relationship between X and Y is linear and if the various assumptions made in a regression analysis are true, then a plot of residuals against the values of X will show no apparent trend or pattern with changes in X [9]. Fig. 2 is a plot of standardised residuals against control strength for all mixtures. Standardised residual is the raw residual (the difference between the estimated and observed values) divided by the standard error of the estimate, which is a measure of the actual variability about the regression plane of the underlying population. If the residuals are normally distributed about the regression, 95% of the standardised residuals should lie between -2 and +2 and 99% between -2.5 and +2.5 [10]. From Fig. 2, almost 100% of the standardised residuals are between -2 and +2, indicating that there are no outliers or extreme residual values. The figure also shows a reasonably well scattered plot, although slightly larger

residuals are observed for control strength between 55 MPa and 75 MPa. This shows that the linear model is less accurate in estimating strength for that range, which corresponds to strengths of pozzolanic mixtures at ages 3 and 7 days of this study.

It is also observed from Table 4 that when more cement was replaced with a pozzolan, the slope of the regression line increased while the Y-intercept decreased. On one hand, the increase in slope with replacement level can be explained by the strength enhancement due to the pozzolanic effect; higher replacement levels provide more available pozzolan for pozzolanic reaction and thus contribute to enhancement in compressive strength. On the other hand, the decrease in Y-intercept can be related to the pozzolan dilution effect; a reduction in cement content will cause a loss in early strength of the blended mixture. Although the finely divided pozzolans may behave as microfillers and increase early strength by promoting efficient packing and a denser early-age transition zone [11], it seems that the increase in strength resulting from the filler effect did not negate the strength loss due to dilution, even at the lowest replacement level of 5%.

A two-way analysis of variance (ANOVA) was conducted to ascertain whether the pozzolan content or w/cm has any significant influence on both the slope, α and Y-intercept, β . The following null hypotheses were tested: (1) H_o : there is no difference in the slope (or Y-intercept) when different pozzolan replacement levels, P are used; and (2) H_o' : there is no difference in the slope (or Y-intercept) when different w/cm are used. The results of the analysis, shown in Table 5, found that at 95% confidence level, only the pozzolan content exerts a significant effect on α and β . The influence of w/cm was insignificant.

3.2 Strength estimation model

Based on the preceding discussion, it is proposed that the compressive strength of a mixture incorporating metakaolin (or silica fume) S_p with w/cm ranging from 0.27 to 0.33, can be related to the compressive strength of its control mixture S_c by using a linear equation:

$$S_p = \alpha S_c + \beta \quad \text{Equation 1}$$

Where α and β are factors related to the pozzolanic and dilution effects of the particular pozzolan. It was established from ANOVA that the factors α and β for each particular pozzolan are dependent on the pozzolan content. Hence, in order to apply the above equation for strength estimation, relationship between α , β and the pozzolan content P must be determined beforehand.

Fig. 3 shows the variation of α and β with the pozzolan content P . In comparing the α and β values obtained for mixtures with metakaolin and silica fume, it was found that the latter produced higher pozzolanic and dilution (absolute value) factors for all replacement levels. This indicates that at equal pozzolan content by mass, silica fume creates a greater strength enhancement from its pozzolanic reactivity but a higher early-age strength loss due to dilution, compared with metakaolin. It can also be deduced from the graph that the pozzolanic factor for SF is likely to increase at replacement levels greater than 15%; while for the case of MK, it is obvious that this factor has somewhat reached its optimum value. This observation suggests that silica fume is more effective than metakaolin in enhancing compressive strength on an equal mass basis.

A statistical computer programme was used to obtain a best-fit equation between the independent variables. The programme uses the Marquardt-Levenberg algorithm [12], which seeks parameter values that minimises the sum of the squared differences between the observed and estimated values of the dependent variable. The analysis found that the α - P

relationship could be represented by a three-parameter rectangular hyperbola equation, while the β - P relationship could be represented by a quadratic equation:

$$\alpha = \alpha_0 + \frac{P}{(a + bP)} \quad \text{Equation 2}$$

$$\beta = cP^2 + dP \quad \text{Equation 3}$$

Where α_0 , a , b , c and d are constants derived from the curve fitting process. The coefficients of determination r^2 for the α - P and β - P relationships were 0.93 and 0.88 for MK, and 0.97 and 0.97 for SF, respectively.

The computed best-fit equations for α and β were then used to estimate compressive strength of all the blended mixtures, the results of which are presented in Fig. 4. Overall, it is observed that 90% of the estimated values lie within $\pm 10\%$ of their actual value. The largest errors occurred in the estimation of early strength where the concrete age is less than 7 days. However, at age 7 days and above, 90% of the estimated values are within $\pm 5\%$ of the actual value. For mature concrete at 28 days and above, 97% of the estimated values are within $\pm 5\%$ of the actual value. This shows that the accuracy of the model increases as concrete matures.

3.3 Application

The strength of an OPC concrete can generally be estimated with good accuracy based on strength charts or by experience if the mixture proportions, age and curing conditions are known. However, when pozzolans are used, exact nature of the strength development becomes complicated as it now depends on the combined effects of cement hydration, pozzolanic reaction, filler and dilution. Different pozzolans show different characteristics depending on its physical and chemical properties. As such, the proposed model provides a quick solution for strength estimation of mixtures where cement is partially replaced with these pozzolans. With this model, strength of blended mixture at a particular age can be

estimated based on a prior knowledge of its equivalent control strength. The model will be of value in the design process of blended concrete mixtures where specific target strength needs to be achieved at certain ages.

3.4 Limitations

Similar to any other strength-estimation models, the proposed model has its flaws and therefore it is imperative that its limitations be discussed and highlighted. At present, the model was derived based on metakaolin and silica fume mixtures, hence the applicability of this model on mixtures blended with other pozzolans is uncertain. For less reactive pozzolan such as pulverised fly ash, it is likely that the f_c (blended concrete) versus f_c (control) curve would produce a different trend than the ones observed. The study also considered mixtures with only up to 15 percent replacement levels and a limited w/cm range of 0.27 to 0.33; hence its applicability to mixtures beyond these limits is unknown. However, bearing in mind that the model is proposed for high-strength concrete, the mixtures studied here can be considered as representative of conventional HSC used in practice. Mixtures with very high contents of MK or SF would not be practical because of economical reasons and also due to possibility of strength loss caused by self-desiccation.

The lower accuracy in estimating strength at early ages is an inherent weakness of the linear model. Whilst this can be corrected with a more complicated curvilinear equation, it does not mean that a curved regression line is always to be preferred to a simple linear one. By using a two- and three-parameter power function to describe the f_c (blended concrete) versus f_c (control) curve, it was found that although both functions gave a better fit, the resultant regression parameters have very high dependencies, indicating that the equation is too complicated or 'over-parameterised'. The parameters also have very high standard error, with coefficient of variation of a few hundred percent for certain cases, and naturally did not

pass the significance test. Although it is always desirable to find an accurate fit to the data it also important to describe the underlying relationship and interpret the meaning of the regression equation.

Another issue is whether the resultant errors are substantial or acceptable for a strength-estimation model of this type. Since the design of concrete structure is usually based on 28-day strength, the accuracy in estimating early age strength is not as critical as in the mature strength. Moreover, considering the fact that blended concrete takes a longer time to achieve full maturity compared to normal concrete, high-strength concrete should be given a longer curing period to ensure that sufficient strength has been achieved before the next construction phase is allowed to proceed. Finally, it should be cautioned here that the pozzolanic factor α and dilution factor β quoted in this study is merely the slope and Y-intercept values for the best-fit linear equation of the blended concrete strength against control strength; and they should not be interpreted as the actual measure of the pozzolanic reaction rate or the dilution effect.

4. Conclusions

It has been demonstrated from the test results examined in this paper that the strength of a concrete mixture made with metakaolin or silica fume can be related to the strength of an OPC control mixture. This relationship is described by using a linear equation in the form of $S_p = \alpha S_c + \beta$ where α and β are factors related to the pozzolanic and dilution effects of the particular pozzolan. The pozzolanic factor α and the dilution factor β can be represented by a three-parameter hyperbolic function and a quadratic function of the pozzolan content, respectively. These relationships are based on mixtures designed with up to 15% mass replacement of OPC with metakaolin and silica fume, and mixtures with w/cm from 0.27 to 0.33. The proposed model was found to have good accuracy in estimating the strength of

mature concrete at age 28 days and above, where 97% of the estimated values are within \pm 5% of the actual values.

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Table 1
Physical properties of cement, metakaolin and silica fume

	Cement	Metakaolin	Silica fume
Specific gravity	3.11	2.52	2.22
Average particle size, μm *	23	9.5	99.4
Specific surface area, m^2/kg			
Blaine	340	-	-
Nitrogen adsorption (BET)	4 200	9 500	21 300
Standard consistency, %	27.4	-	-
Setting time, min			
Initial	110	-	-
Final	300	-	-

* Average particle size was measured using laser particle size analysis. The reported value for silica fume represents the average agglomerate size.

Table 2
Chemical composition of cement, metakaolin and silica fume

%	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃	MgO	Na ₂ O	K ₂ O	P ₂ O ₅	TiO ₂	MnO	LOI
Cement	20.99	6.19	65.96	3.86	0.20	0.17	0.60	0.05	0.40	0.06	1.53
MK	57.40	35.26	0.02	0.94	0.18	<0.01	3.17	0.09	0.43	<0.01	2.52
SF	92.06	0.48	0.40	2.11	0.63	0.28	1.24	0.02	<0.01	0.23	2.54

Table 3
Mixture proportions

Mixture	Cement (kg/m ³)	MK (kg/m ³)	SF (kg/m ³)	Water (kg/m ³)	w/cm	Granite stone (kg/m ³)	Silica sand (kg/m ³)	SP (L/m ³)
Series A (w/cm = 0.27)								
C - 0.27	500	-	-	135	0.27	1050	720	43
MK 5 - 0.27	475	25	-	135	0.27	1050	720	43
MK 10 - 0.27	450	50	-	135	0.27	1050	715	43
MK 15 - 0.27	425	75	-	135	0.27	1050	710	43
SF 5 - 0.27	475	-	25	135	0.27	1050	725	43
SF 10 - 0.27	450	-	50	135	0.27	1050	715	43
SF 15 - 0.27	425	-	75	135	0.27	1050	715	43
Series B (w/cm = 0.30)								
C - 0.30	500	-	-	150	0.30	1050	695	19
MK 5 - 0.30	475	25	-	150	0.30	1050	690	19
MK 10 - 0.30	450	50	-	150	0.30	1050	685	19
MK 15 - 0.30	425	75	-	150	0.30	1050	680	19
SF 5 - 0.30	475	-	25	150	0.30	1050	685	19
SF 10 - 0.30	450	-	50	150	0.30	1050	680	19
SF 15 - 0.30	425	-	75	150	0.30	1050	680	19
Series C (w/cm = 0.33)								
C - 0.33	500	-	-	165	0.33	1050	700	12
MK 5 - 0.33	475	25	-	165	0.33	1050	695	12
MK 10 - 0.33	450	50	-	165	0.33	1050	690	12
MK 15 - 0.33	425	75	-	165	0.33	1050	685	12
SF 5 - 0.33	475	-	25	165	0.33	1050	690	12
SF 10 - 0.33	450	-	50	165	0.33	1050	685	12
SF 15 - 0.33	425	-	75	165	0.33	1050	680	12

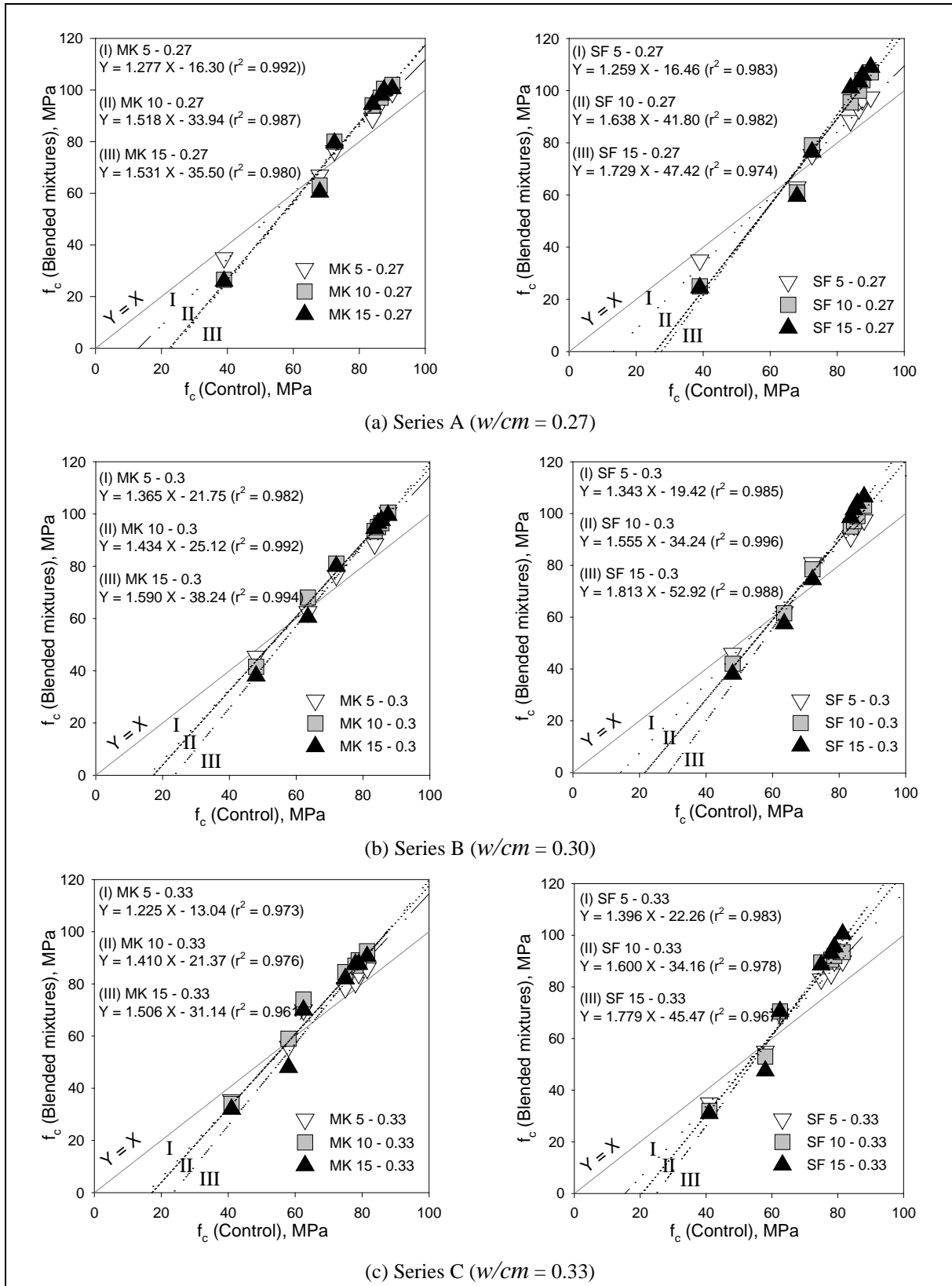


Fig. 1. Relationship between compressive strengths of blended and non-blended mixtures

Table 4
Standard error of estimate and t-statistic for the regression coefficients

Mixture	Slope, α				Y-intercept, β				r^2
	α	Std. Error	t	P	β	Std. Error	t	P	
Series A									
MK 5	1.277	0.0518	24.660	< 0.0001	-16.30	3.99	-4.078	0.0096	0.992
MK 10	1.518	0.0782	19.402	< 0.0001	-33.94	6.04	-5.622	0.0025	0.987
MK 15	1.531	0.0981	15.601	< 0.0001	-35.50	7.57	-4.687	0.0054	0.980
SF 5	1.259	0.0739	17.045	< 0.0001	-16.46	5.70	-2.887	0.0343	0.983
SF 10	1.638	0.0986	16.616	< 0.0001	-41.80	7.61	-5.494	0.0027	0.982
SF 15	1.729	0.1254	13.789	< 0.0001	-47.42	9.68	-4.901	0.0045	0.974
Series B									
MK 5	1.365	0.0701	19.462	< 0.0001	-21.75	5.34	-4.074	0.0096	0.987
MK 10	1.434	0.0565	25.374	< 0.0001	-25.12	4.30	-5.839	0.0021	0.992
MK 15	1.590	0.0571	27.841	< 0.0001	-38.24	4.35	-8.789	0.0003	0.994
SF 5	1.343	0.0735	18.279	< 0.0001	-19.42	5.60	-3.470	0.0179	0.985
SF 10	1.555	0.0465	33.451	< 0.0001	-34.24	3.54	-9.670	0.0002	0.996
SF 15	1.813	0.0901	20.114	< 0.0001	-52.92	6.87	-7.709	0.0006	0.988
Series C									
MK 5	1.225	0.0905	13.534	< 0.0001	-13.04	6.26	-2.081	0.0190	0.973
MK 10	1.410	0.0997	14.140	< 0.0001	-21.37	6.90	-3.0963	0.0270	0.976
MK 15	1.506	0.1360	11.077	< 0.0001	-31.14	9.41	-3.308	0.0213	0.961
SF 5	1.396	0.0812	17.195	< 0.0001	-22.26	5.62	-3.960	0.0107	0.983
SF 10	1.600	0.1068	14.980	< 0.0001	-34.16	7.40	-4.619	0.0057	0.978
SF 15	1.779	0.1467	12.122	< 0.0001	-45.47	10.16	-4.477	0.0065	0.967

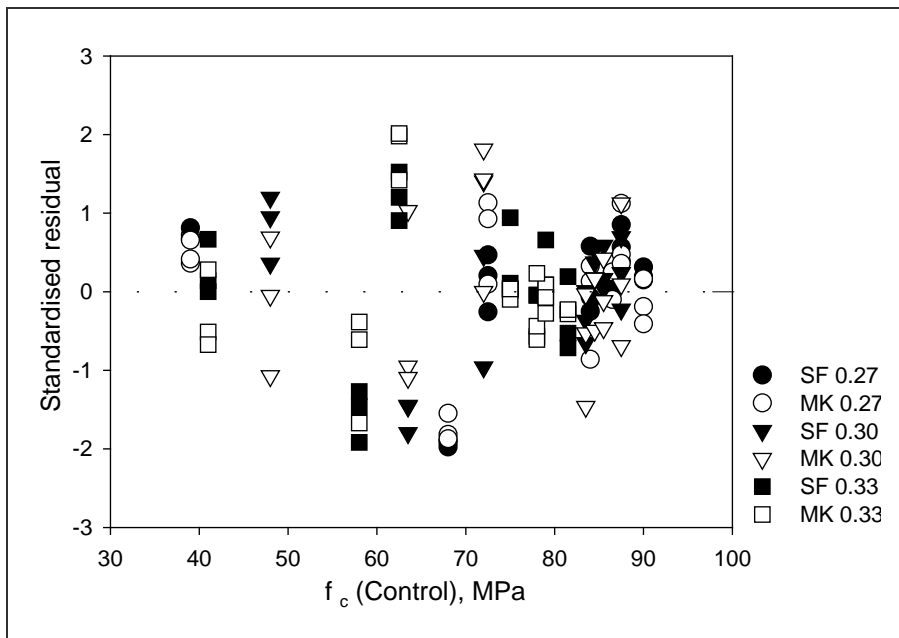


Fig. 2. Standardised residual

Table 5
Two-way analysis of variance (ANOVA)

Dependent variable	Source of variation	Degrees of freedom	Mean square	F	P	Significance
α (MK)	Replacement level, P	2	0.0497	21.787	0.007	Yes
	w/cm	2	0.00554	2.426	0.204	No
	Residual	4	0.00228			
α (SF)	Replacement level, P	2	0.148	45.872	0.002	Yes
	w/cm	2	0.00186	0.578	0.602	No
	Residual	4	0.00322			
β (MK)	Replacement level, P	2	241.763	16.163	0.012	Yes
	w/cm	2	43.923	2.936	0.164	No
	Residual	4	14.958			
β (SF)	Replacement level, P	2	648.070	31.975	0.003	Yes
	w/cm	2	2.065	0.102	0.905	No
	Residual	4	20.268			

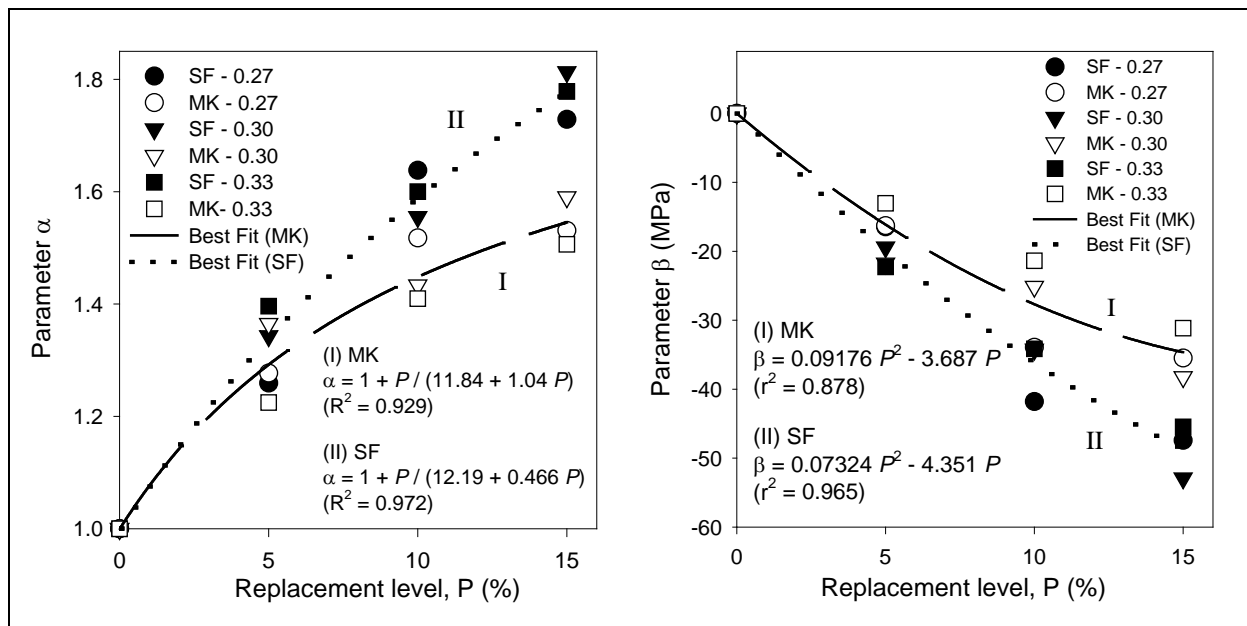


Fig. 3. Best-fit equations for parameters α and β

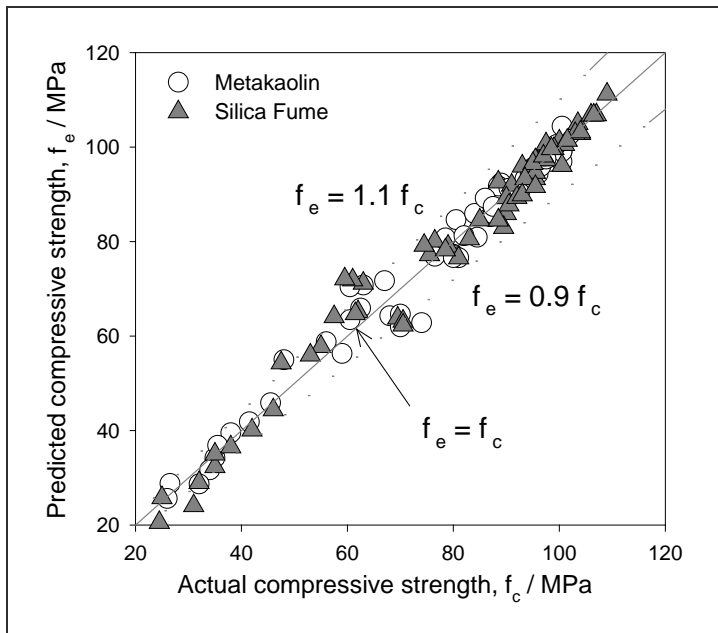


Fig. 4. Estimated strength using proposed method