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AN EXPERIMENTAL FACTORIAL DESIGN FOR ASR MITIGATION WITH FLY ASH

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Abstract: Use of fly ash by percent replacement of cement by weight is considered as one of the most economical and effective method for mitigating Alkali-silica reaction (ASR) related distress in the concrete. Fly ash reduces the pore solution alkalinity through increasing the alkali binding capacity of the cement hydrates and through pozzolanic reaction. However, Fly ash is proven to be somewhat variable in its effectiveness on inhibiting alkali-silica reactivity, principally because its composition depends on the coal properties from which it is derived. Typically Class C fly ashes are not as efficient as Class F ashes due to their higher calcium oxide content. Also, it is not established if the dosage of fly ash is more influential than type of fly ash and vice versa. Therefore, in the field, for a certain job mixture, the prediction of mitigation effect of a certain type and dosage of fly ash is difficult. This research aims to correctly predict the effectiveness of fly ash mitigation, to find out the most influential factor and interaction effects between factors. A statistical model, of two-level design with 3 factors, was developed based on three main factors: fly ash lime content, dosage and soak solution alkalinity. The statistical model was verified with additional experimental results with random fly ash-lime content and different dosages; which matched very well with the model predictions. Therefore, such model(s) could be applied in practice with the availability of larger database. Also, another finding of this research is that, the lime content of the fly ash is the most significant factor followed by the dosage level.

Keywords: *Fly ash, Concrete, Alkali Silica Reactivity, Lime, Factorial Design*

1.0 Introduction

The alkali-silica reaction (ASR) related distress is a matter of great concern to the concrete industry and regarded as second most deterioration issue after corrosion. Reactive silica in the presence of alkali in the pore solution inside the concrete creates a hydrophilic alkali-silica gel, often referred to as ASR gel. Formation of the ASR gel alone does not cause cracking, however when the gel absorbs water, it shows significant potential to swell. The resulting expansion often results in pressures greater than what the concrete can withstand, which in turn causes cracks in the concrete. ASR-induced expansion will occur only if the following three conditions are met: (1) the aggregates in the concrete mixture contain reactive forms of silica, (2) sufficient alkalies; alkali content in the cement greater than 0.60%; and (3) sufficient moisture is available in the hardened concrete (above 75% RH within the concrete). Preventing any of these three conditions from being a reality is sufficient to prevent deterioration and is often the emphasis of prevention strategies. Alkalies are present in the cement since cement production involves raw materials that contain alkalis in the range of 0.2 to 1.5 percent of Na_2O. This generates a pore fluid with high pH (12.5 to 13.5). ASTM C150/C150M-15 (2015) designates cements with more than 0.6 percent of Na_2O as high-alkali cements.

The combustion of pulverized coal at high temperatures and pressures in power stations produces different types of ash. The 'fine' ash fraction is carried upwards with the flue gases and captured before reaching the atmosphere by highly efficient electro static precipitators. This material is known as Pulverized Fuel Ash (PFA) or 'fly ash'. It is composed mainly of extremely fine, glassy spheres and looks similar to cement. Types of Fly ash:

Class F (Less than 6% Lime- Calcium Oxide, CaO)
Class C (more than 20% Lime- Calcium Oxide, CaO)
Intermediate Class (Lime content between 6% to 20%)

Certain mitigation measures are employed to reduce the ASR distress with reactive aggregates (Touma *et al*., 2001; Hudec and Banahene, 1993). Fly ash (by % replacement of cement by weight) is widely used in the industry as a mitigation measure. The fly-ash in concrete reduces the amount of non-durable calcium hydroxide (lime), and converts lime into calcium silicate hydrate (CSH) over time. Typically Class C fly ashes are not as efficient as Class F ashes due to their higher calcium oxide content. The increased binding capacity of the hydration products has been linked to the lower Ca/Si ratio of the hydrates compared to neat portlandcement pastes (Thomas *et al*., 1999). Fly ash is proven to be somewhat variable in its effectiveness on inhibiting alkali-silica reactivity, principally because its composition depends on the coal properties from which it is derived (Hudec and Banahene, 1993; Malvar *et al*., 2002).

A pozzolan is defined by ASTM C618 (2015)as a siliceous or siliceous and aluminous material which, in itself, possesses little or no cementitious value but which will, in finely divided form in the presence of moisture, react chemically with calcium hydroxide at ordinary temperature to form compounds possessing cementitious properties. Pozzolanic reaction stands a simple acid-base reaction between calcium hydroxide, also known as Portlandite, or ($Ca(OH)_2$), and silicic acid(H_4SiO_4, or $Si(OH)_4$). Simply, this reaction can be summarized in abbreviated notation of cement chemists:

$$CH + SH \rightarrow C\text{-}S\text{-}H \qquad (1)$$

Numerous test methods were developed to find the ASR potential and some methods can find the effectiveness of the mitigation measure. The Accelerated Mortar Bar Test (ASTM C1260, 2007) originally proposed by Oberholster and Davis in 1986 has been widely adopted for ASR. However, the results from this test method can be unreliable due to the aggressive conditions used in the test. On the other hand, the Concrete Prism Test (ASTM C1293, 2007) is recognized as the most reliable test procedure which requires two years for mitigation purpose. The long duration required in this test method renders this method impractical. In this research Miniature Concrete Prism Test -MCPT method (Latifee & Rangaraju, 2015) was used to find out the expansion of the specimens made with fly ashes, since MCPT proved to be one of the most reliable and results could be obtained within 56 days.

The mix design of concrete can also influence the ASR related damage in concrete. Therefore, in this research, as a screening test, three of the mix design parameters - water to cement ratio, amount of cement and cement alkalinity were investigated for ASR distress using a factorial design. It was found that cement alkalinity was the most dominant factor within these parameters.

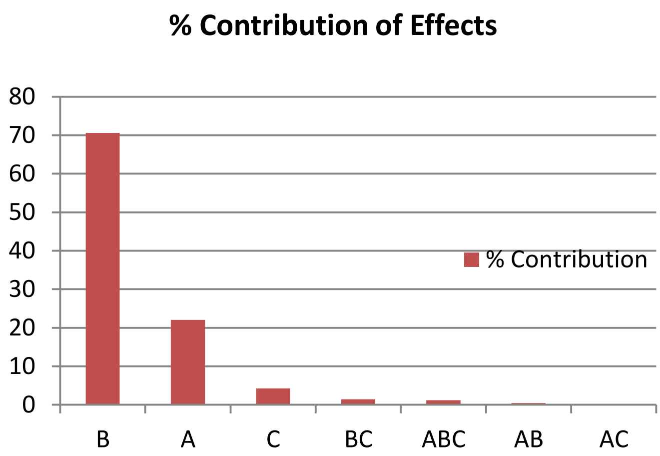


Figure 1: Pareto chart of % contribution of effects (*Note: A= w/c, B= Cement alkalinity, C = Amount of cement)*

The fly ash mitigation effectiveness model includes-fly ash amount (% replacement of cement), lime content (CaO %) in the fly ash and the soak solution alkalinity which includes cement alkalinity. The main effects and interaction effects were explored and a general ASR expansion prediction equation was developed. The equation was later verified by additional data of fly ash types, dosage etc. All the experiments were carried out using Miniature Concrete Prism Test (MCPT) method.

Fly ashes: The fly ash content of calcium oxide (or lime) has been considered as the main factor on the efficiency of the ash in mitigating ASR (Esteves *et al*., 2012; Folliard *et al*. 2006; Malvar *et al*., 2006; Malvar *et al*., 2001; Moser *et al*., 2010; Thomas, 1996). AASHTO T 303 requires minimum 15% Class F fly ash for ASR mitigation and CSA A23.2-27A recommends at least 25 to 30% low-lime fly ash, CALTRANS reports that Class F fly ash (and Class N pozzolan) are effective against ASR when replacing up to 30% of the portland cement (by mass); International Center for Aggregate Research (ICAR) at Austin ,TX recommends 25% Class F fly ash; or 35% Class C fly ash [Folliard *et al*., 2006]. In this research study, nine different fly ashes of varying chemical compositions were used.

2.0 Materials and Methods

2.1 Aggregate

A well-known representative reactive coarse aggregate Spratt limestone was selected with a known non-reactive fine aggregate. The reactive coarse aggregate is Siliceous Limestone from Spratt Quarry in Ontario, Canada and the non-reactive fine aggregate is Siliceous sand from Dixiana Plant in Pineridge, South Carolina.

Table 1: Properties of the Aggregates Used

Property	*Spratt Limestone (Coarse Aggregate)*	*Foster Dixiana (Fine Aggregate)*
SG_{OD}	2.69	2.63
SG_{SSD}	2.71	2.64
Absorption, %	0.46%	0.44%
Dry Rodded Unit Weight (kg/m^3)	1568	---

2.2 Cement

A high-alkali Type I cement from Lehigh Cement Company, from Evansville Plant in Pennsylvania and a low-alkali cement from ARGOS Cement Company from Harleyville, SC were used in this study. The autoclave expansion of both cements was well below 0.80 percent, at 0.03% for low-alkali cement and 0.018% for high-alkali cement.

Table 2: Chemical Composition of High-Alkali and Low-Alkali Cement

Oxides (%)	*High Alkali Cement*	*Low Alkali Cement*
SiO_2	19.78	20.6
Al_2O_3	4.98	5.1
Fe_2O_3	3.13	3.4
CaO	61.84	64.5
MgO	2.54	1
SO_3	4.15	3.1
Mn_2O_3	---	
Na_2O equivalent	0.82	0.49
Specific Gravity	3.15	3.15

2.3 Reagents

Reagent grade sodium hydroxide from Fisher Chemicals was used. Fly ashes of different types (low and high lime) at different levels (low and high dosage) with different cements (low and high alkali) were tested in “2^3 factorial design”. The three factors (continuous variable) are Lime content, CaO% of Fly Ash Fly Ash Dosage, %, (replacement % of cement) and Soak Solution Alkalinity (expressed as N NaOH). Each factor had two discrete levels as follows;

(a) CaO% (6.06% and 27.5%, Low and High lime fly ashes)
(b) Fly Ash Dosage, % (15% and 35% replacement levels of cement)
(c) Soak Solution Alkalinity (0.5 N and 1 N NaOH)

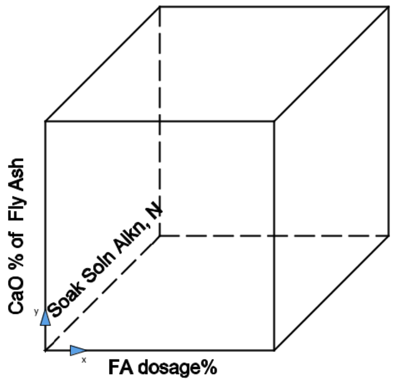


Figure 2: Graphic presentation of factorial design for FA dosage, CaO% of Fly Ash and Soak Solution Alkalinity effects on ASR mitigation.

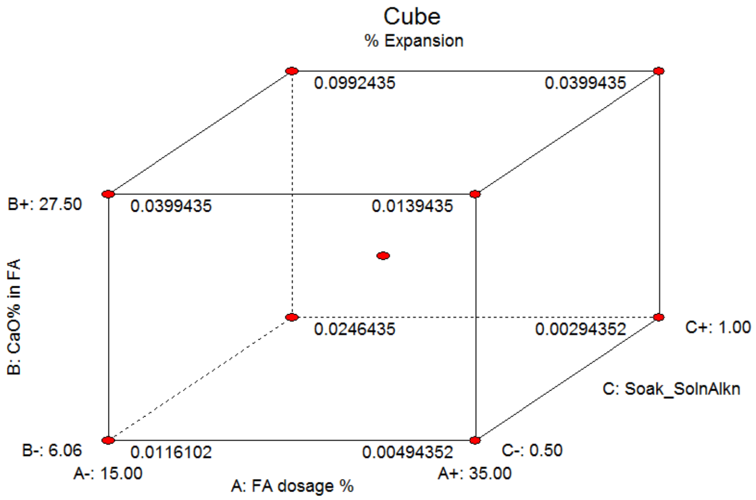


Figure 3: Cube plot of factorial design for FA dosage, CaO% of Fly Ash and Soak Solution Alkalinity effects with expansion values.

2.4 Description of the MCPT Method

In this method, concrete prisms of dimensions 50 mm x 50 mm x 285 mm (2 in. x 2 in. x 11.25 in.) are used for evaluating the reactivity of both coarse and fine aggregates. Mixture proportions of ingredients used in preparing the MCPT specimens are standardized as follows:

Table 3: MCPT Specimen Mixture Proportion

Item	*Mix Proportion*
Cement content of the mix	420 kg/m^3 (708 lb/yd^3)
Water-to-cement ratio	0.45
Coarse aggregate volume fraction (dry)	0.65
Maximum size of coarse aggregate	12.5 mm (1/2 in.)
Coarse aggregate gradation (% by weight of total coarse aggregate)	
12.5 mm – 9.5 mm	57.5%
9.5 mm – 4.75 mm	42.5%
Fine aggregate	Determined based on ACI 211 absolute volume method, i.e., subtracting the proportions of all the other ingredients from 1 m^3 of concrete

The proportions of aggregate in the 12.5 mm – 9.5 mm fraction and the 9.5 mm – 4.75 mm fraction were selected, based on the assumption of maintaining approximately constant surface area across each of the two aggregate size fractions. To ascertain the coarse aggregate reactivity, a non-reactive fine aggregate is used in the concrete mixture to isolate the effects of the reactive aggregate. Similarly, when the reactivity of a fine aggregate is to be ascertained, a non-reactive coarse aggregate is used. In this protocol, a cement having a high alkali content of 0.9 ± 0.1% Na_2O_{eq} is required to be used. The alkali content of the concrete is boosted to 1.25% Na_2O_{eq} by weight of cement similar to the procedure used in the standard ASTM C1293 test method. The test specimens are demolded 24 hours after casting and after taking the initial length reading the prisms are submerged in water at 60° Celsius for an additional 24 hours. At the end of 48 hours from the time of casting, the zero-day length change reading is taken, before the prisms are transferred to 1N NaOH soak solution that has already been pre-conditioned to 60° Celsius temperature. Subsequent length change readings are periodically taken at 3, 7, 10, 14, 21, 28, 42, 56, 70 and 84 days.

2.4.1 Modified MCPT Method

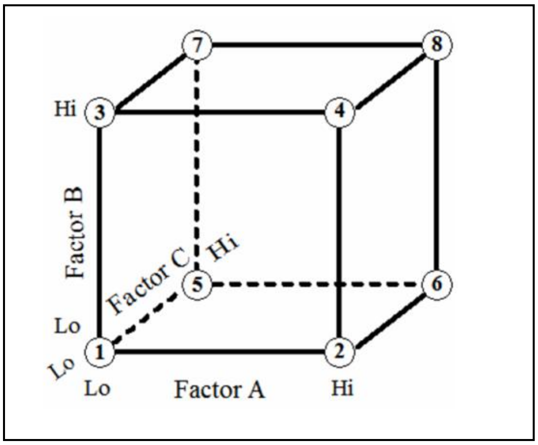
In this method, everything is same except the soak solution (instead of being 1N NaOH), matches the pore solution based on the predictive equation described below. The predicted alkalinity of the pore solution was calculated based on the equation developed by Stark *et al.* (1993) as follows:

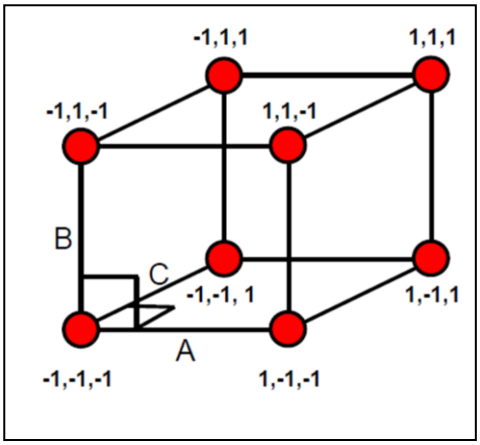
$$[OH-] = 0.339\ Na_2O\ \% / (w/c) + 0.022 +/- 0.06\ mol/L \qquad (2)$$

Also, 56-day expansion data in each case was taken as the specimen percent expansion taken into account.

2.5 Factorial Design

In this research, factorial design was chosen for the experimental data analysis which is a very powerful tool and widely used to analyze the data. A full factorial design is capable to analyze many factors simultaneously and it contains all possible combinations of a set of factors. It is also a test for an ‘interaction’ between treatments or factors– does a treatment or factor work even better in the presence of another. In other words, the factorial design can examine the interaction or joint effect of the independent variables on the dependent variable. We cannot get this information by running separate one-way analyses. A common experimental design is one with all input factors set at two levels each. These levels are called `high' and `low' or `+1' and `-1', respectively. A design with all possible high/low combinations of all the input factors is called a full factorial design in two levels. If there are k factors, each at 2 levels, a full factorial design has 2k runs or experiments. It is expressed as $level^{factor}$. For example, if 3 factors are investigated at 2 levels, this will need 2^3=8 experiments. In this research three factors (variables) are investigated at two levels (low and high), the detail of which is similar to the Figure 4.





(a) (b)

Figure 4 (a), (b) schematic diagram of 2^3 factorial design

3.0 Results and Discussion

A total set of nine MCPT mitigation tests were carried out, which includes 8 design corner points of the cube (using HL-1 and LL-1 fly ashes) and a center point(using IL-1 fly ash). The factorial design combination is given in Table 4. The cement alkalinity was boosted from 0.82% to 1.25% Na_2O_e for high alkali cement and from 0.49% to 0.55% Na_2O_e for low alkali cement. It was done to make sure that the pore solution in the MCPT specimens becomes same (calculated by empirical equations of C-342-Eq2.2.1 and Thomas *et al.*, 2011) as the soak solution of 1 N and 0.5N NaOH respectively.

Table 4: Factorial design data table with actual expansion of each combination

Exp. No.	*FA dosage%*	*CaO % of FA*	*Soak Soln. Alkn, N*	*56-Day, % Expansion*
1	15	27.5	1	0.1003
2	15	6.06	1	0.0257
3	35	27.5	0.5	0.015
4	25	16.78	0.75	0.0212
5	15	6.06	0.5	0.0127
6	35	6.06	1	0.004
7	35	27.5	1	0.041
8	15	27.5	0.5	0.041
9	35	6.06	0.5	0.006

The data from Table 4 were used in statistical analysis software to do the factorial design analysis. The main factor (variable) effects and the interaction effects are graphically shown in the Figure 5 (a-f). These were done in excel, using the average values of appropriate responses (Avg. values of 56 day % expansions for given combinations). For this purpose the Table 4 was modified according to Yates order in Table 5 and Table 6 is a sample table for interaction and main effects data. The interaction and main effect plots made in excel are cross checked with the software output.

Table 5: Yates order table for factorial design data with actual expansion

Standard Order	*FA %*	*CaO % of FA*	*Soak Soln. Alk.*	*56 day % Expansion*
1	-	-	-	0.01267
2	+	-	-	0.00600
3	-	+	-	0.04100
4	+	+	-	0.01500
5	-	-	+	0.02567
6	+	-	+	0.00400
7	-	+	+	0.10030
8	+	+	+	0.04100

Table 6: Sample table for interaction effect data

	FA dosage%	
	15% (-)	*35% (+)*
CaO % of FA, 6.06(-)	0.019	0.005
CaO % of FA, 27.5 (+)	0.071	0.028
	56-Day, % Expansion	

Table 7: Percent contribution of different factors

	% Contribution
B	39.75
A	23.15
C	16.63
BC	9.88
AB	5.81
AC	4.19
ABC	0.60
Sum=	100

Note: A= FA %, B= CaO % of FA, C- Soak Soln. Alkalinity

The findings are summarized as follows: (a) the Lime i.e., CaO% of Fly Ash increment increases the ASR expansion; (b) the Fly Ash (FA) dosage increase in the test decreases the ASR expansion, (c) the Soak Solution Alkalinity increase in the test increases the ASR expansion, (d) there is interaction between Fly Ash dosage and CaO% of Fly Ash (FA), (e) FA dosage % increase reduces the ASR expansion more rapidly in high lime FA than low lime FA, (f) there is interaction between Fly Ash dosage and soak solution alkalinity, (g) FA dosage % increase reduces the ASR expansion more rapidly in high soak solution alkalinity than low soak solution alkalinity, (h) there is interaction between lime content, CaO% of Fly Ash and soak solution alkalinity, (i) the lime content increase in FA increases the ASR expansion more rapidly in the high soak solution alkalinity than the low soak solution alkalinity.

3.1 Prediction Equation for 56-Day Expansion with Fly Ash

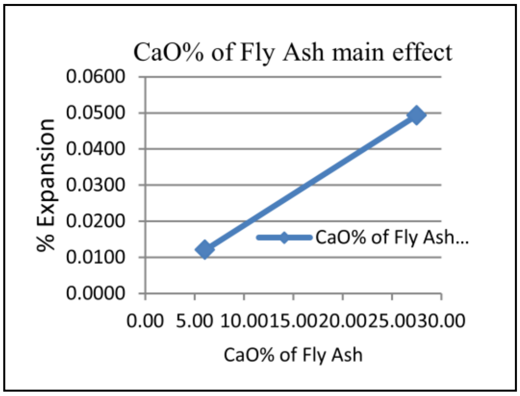
Based on the factorial design model a prediction equation is developed using the statistical software "Design Expert", which has a general form as the following, Prediction Equation (general form), Y = f (A, B, C);

$$Y= b_0+b_1*A+ b_2*B+b_3*C+b_4*(A*B) + b_5*(B*C) + b_6*(C*A) +b_7*(A*B*C) \quad (3)$$

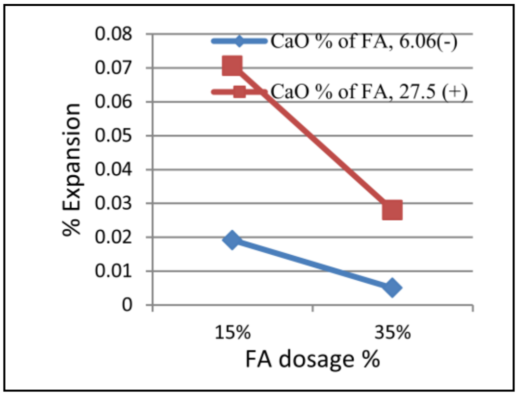
Let, *A=FA dosage %, B =CaO % of FA, and C= Soak Solution Alkalinity.*

Prediction Equation = 0.000230824+0.000257773*A+0.000192766*B+0.00966084*C-0.0000175713*A*B+0.00368378*B*C -0.000648737*C*A-0.0000561544*A*B*C

The prediction equation involved two different fly ashes (one low and one high lime). Then, the equation is verified with six other fly ashes that were not part of the model in Table 8. Figure 7 shows that the correlation (R^2=0.91) between experimental and predicted values of six different fly ashes at 56 days are in harmony.



(a)



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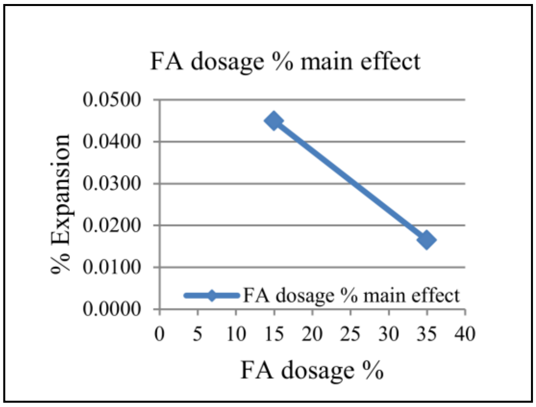

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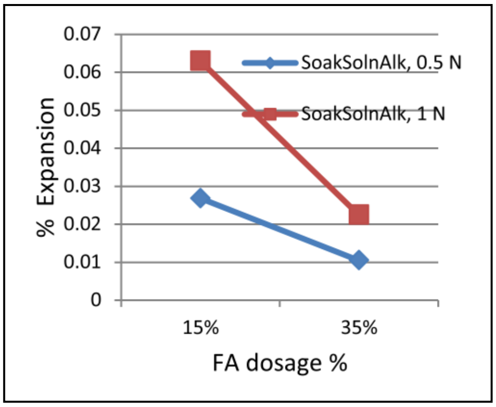

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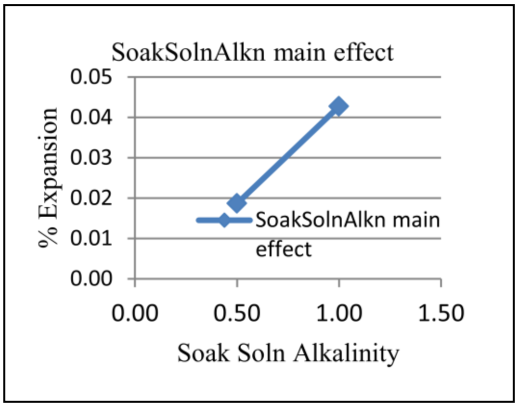

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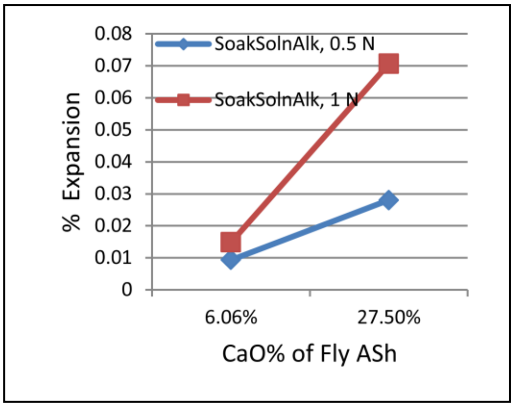

(f)

Figure 5: (a-f) Main factors and interaction effects in FA mitigation model

In figure 6 the percent contribution of different factors are shown. It is clear that CaO % of FA, factor B, has the most contributing effect towards ASR expansion followed by FA dosage %, factor A and soak solution alkalinity, factor C. Among the interaction effects the BC, (B =CaO % of FA, and C= Soak Solution Alkalinity) is the most contributing.

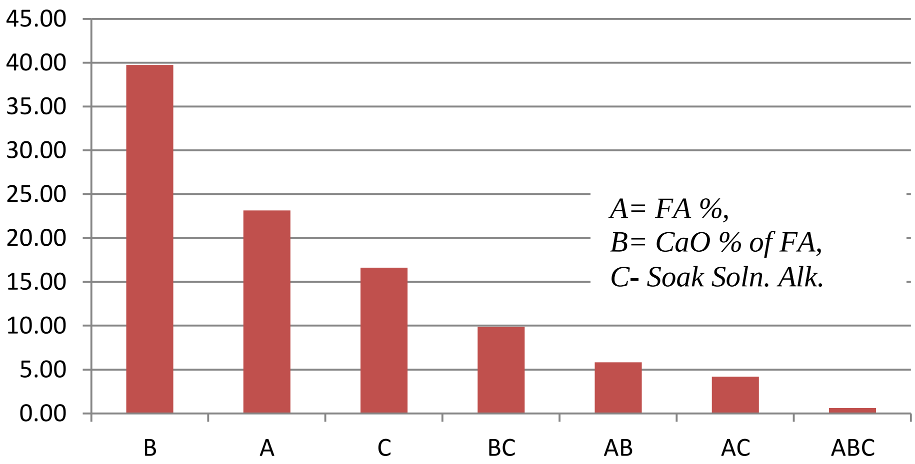

Figure 6: Pareto chart of % contribution of A=FA %, B =CaO % of FA, and C= Soak Solution Alkalinity

Table 8: Six different fly ashes experimental and predicted values at 56 days

	Experimental % Expansion	*Predicted % Expansion*
GeraldGentleman 25%FA, 26.6% CaO, 1 N	0.0453	0.0542
Comanche 25%FA, 29.85%CaO, 1 N	0.0600	0.0608
SanJuan 25%FA, 6.06% CaO, 1 N	0.0110	0.0124
CoalCreek 25%FA, 15.63% CaO, 1 N	0.0212	0.0319
Apache 25%FA, 10.33% CaO, 1N	0.0177	0.0211
ColetoCreek 25%FA, 18.94% CaO, 1 N	0.0237	0.0386

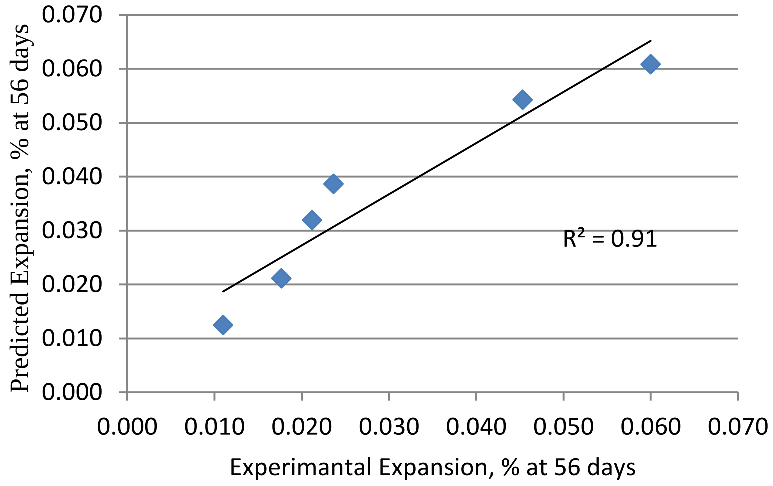

Figure 7: Correlation between experimental and predicted values of six different fly ashes at 56 days.

4.0 Conclusions

The following conclusions are drawn from this research:

1. The lime content, CaO% of the fly ash is the single most influential factor for fly ash mitigation of ASR, rather than the dosage. The lower the lime content the more effective it will be regarding ASR mitigation. Therefore, using higher replacement of cement by high lime fly ash will not bring the same effect as low lime fly ash.

2. The dosage of fly ash, as percent replacement of cement by weight, is the second most significant factor followed by the cement alkali level.

3. There is interaction or joint effect, between Fly Ash dosage and CaO% of Fly Ash (FA), i.e., high dosage is more effective in case of high lime fly ash.

4. Also, interaction between Fly Ash dosage and soak solution alkalinity: higher dosage is more effective in high alkaline environment

5. Interaction between lime content (CaO%) of Fly Ash and soak solution alkalinity: soak solution alkalinity representing the pore alkalinity within concrete, effect is more in case of high lime content of fly ash.

6. The prediction equation can be handy as a screening test or first trial for fly ash mitigation with reactive aggregates for certain job mixture.

7. The factorial model can also be used to optimize the fly ash dosage, lime content of FA and concrete pore solution (soak solution) alkalinity by setting a target expansion using any statistical software such as Minitab, Jmp etc.

5.0 Acknowledgement

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