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Prediction of Property –Composition Relation of a Polymer Modified Mortar (PMM): Box-Wilson Composite Design

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Abstract: A Box-Wilson composite design scheme has been applied to study the property-composition relation of a polymer-modified concrete. The ultimate goal was to develop a regression equation, which would predict the compressive strength development of a composition with the curing period. A two-level design was constructed with three variables, mainly sand: cement, water: cement and polymer: cement. The homogeneity of the data was tested with Cochran ratio; the significance of the coefficients of the regression equation is tested by t-criterion. Finally the adequacy test was performed by Fishers' criterion. The regression equation, so formed, describes the property-composition relation satisfactorily. This equation may be used in making repairing as well as structural material making compromise between the price and strength.

Keywords: Polymer Modified Mortar (PMM), Epoxy resin, Binding material, Compressive Strength, Box-Wilson composite design.

I. INTRODUCTION

This Cement is the mostly used binding material which is still using from long years ago. Cement mortar is the mixture of the cement, water and fine aggregate. The property of a cement mortar depends mainly in the composition of cement, sand and water. Depend on the area of application different types of mortar with different property are also manufactured. Polymer as a binding material in complete or partial replacement of cement in construction industry has been being used for the last decades. These materials have improved mechanical strength and better adhesion to old structures that the conventional cement-based compositions have. The replacement of cement by polymer, however, is directly related to the composition. By only using intuition skilled personnel can tell about required composition for a definite property. But this is not a good way, there must need a scientific way to calculate the property of a mortar for different materials composition.

In modern construction and repair works, application of polymer is increasing day by day by the replacement of conventional mortar or concrete to polymer modified mortar or concrete, in order to overcome the shortcomings of conventional mortar or concrete like poor flexural strength, poor tensile strength, high porosity, freeze thaw deterioration, destruction by corrosive chemicals etc. [1].

Epoxy resin has been applied to reduce the seepage of water into concrete structures. Epoxy has the properties that give it excellent adhesion to the surface of concrete as well as the ability to repel water and many chemicals which come in contact [2].

Few investigations have been reported in the literature on different aspects of the epoxy based mortars are discussed in the following paragraphs. Aggarwal et al. prepared the PCM based on epoxy resin and acrylic emulsion, and found that both the PCMs have higher compressive and flexural strength, and better resistance to the penetration of chloride ion and carbon dioxide than the conventional Portland cement mortar (CM) [3]. Among the epoxy resin and acrylic emulsion based PCMs under investigation, the epoxy-based mortars showed superior quality. Hassan et al. reported that the epoxy resin-based PM exhibited lower porosity, lower water permeability and lower shrinkage than the CM [4].

Although, outer world is far ahead in polymer modified mortar, but to the author's knowledge, this is first time attempts have been made to develop a regression equation based on Box-Wilson composite design experiment for epoxy based polymer mortar in Bangladesh, which relates the compressive strength of mortar with the composition.

II. EXPERIMENTAL

A. Material:

The components of the mortar are Portland cement, epoxy resin, hardener (for the resin), and sand (as fine aggregate).The cement used in this study was a commercial ASTM type I ordinary Portland cement. The epoxy resin and the hardener for the resin were collected from the local market.

Characteristics of the fine aggregate

We performed the sieve analysis of fine aggregate (sand) for 500gm dry sample and Fig. 1 shows the size distribution of the sand used for polymer mortar. Most of the sand size was in the range of 0.6-1.18mm and least sample size was above 4.75mm and less than 0.15mm.

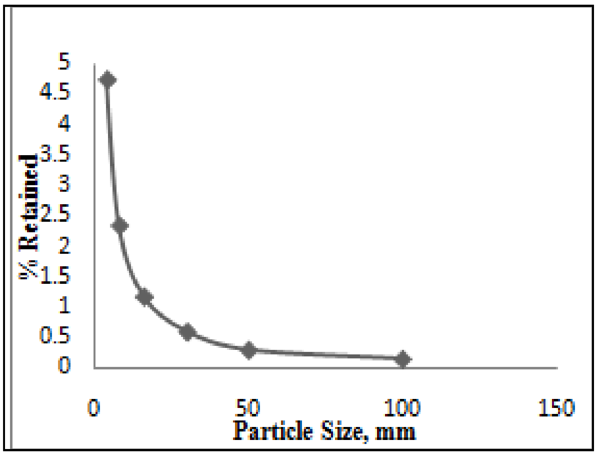

Fig1. Particle size distribution curve

Conventional binder (Portland cement)

The chemical composition and some other characteristics (in weight percent) of the Portland cement as per document are as follows: CaO-63.58, SiO2 -20.44, Al_2O_3 -5.34, Fe_2O_3 -4.0, Loss on ignition-1.10, Insoluble residue-0.07 and Moisture content-0.5. The characteristic test-properties of the used Portland cement are presented in TABLE I.

TABLE I**:** Properties of Portland cement used in the experiment

Test performed	result
1. Fineness test	0.068%
2. Setting time	
Initial	2 hours
Final	3 hours
3. Compressive strength	
7 day	15 MPa
28 day	19 MPa

The setting time of Portland cement was determined by Vicat Niddle following ASTM standard method (C 191-82). The compressive strength and the fineness of Portland cement were determined by ASTM standard method C109-80 and C 184-83 respectively.

Polymer binder system (epoxy resin and the hardener)

The epoxy resin was collected from the local market and was used without further treatment/purification. As a commercial product of technical grade, the exact chemical composition of the resin remains unknown, but as is being presented as epoxy resin and is serving the purpose, it is presumed that the main component of the technical grade resin contains epoxy-functional group. The product is in liquid form, hydrophobic in nature and almost insoluble in water. In contact with water, it forms heterogeneous dispersion. On laboratory test, the following data was obtained for the resin at 30^0C: viscosity - 3600cps, specific gravity-1.15 and pH= 7.70. The hardener belongs to the group of poly-functional amine compounds and was used as a hardener of epoxy resin in the form as was available in the local market. It is in liquid phase at ambient temperature and is soluble in both epoxy resin and water. It has a specific gravity of 0.98gm/cm^3.

B. Mixing, Casting and Curing:

A concrete mixer machine was used for mixing. This machine had a moving bowl with a steel wall and mixing rod inside the bowl. The component ratio of cement, sand, polymer and water was taken as the design equation. The amount of hardener was 10 wt% of the added epoxy resin. As epoxy resin is insoluble in water, the following mixing-procedures were attempted with a view to ensure homogeneity of the composition and effective bonding in the structure.

The mixing was done as per the following sequence/order of operation:

- Cement and sand aggregate were put in the mixer.
- Calculated amount of water was added to the composition and homogenized for 2 min.
- Calculated amount of hardener was dissolved in the epoxy resin
- The resin with the hardener dissolved in it was added to the mixture of Portland cement, aggregate and water and homogenized for 1 min.
- The fresh concretes were cast into 50.8mm cube molds for compression testing, and were left for 24 hours and specimens were separated out from the mold and were kept in water for 7-28 days for curing

C. Experimental design of a given parameter

The total number of observation in the matrix of a composite design for k factors is given by

$N = 2^k + 2k + n_0$ for k<5

n_0 is the number of runs at the centre point, for our experiment total number of observation, N=15.

Second-Order orthogonal Design

Composite designs can be transformed into orthogonal ones by an appropriate selection of the star arm +α. The value of +α can be calculated by the following equation.

$\alpha^4+2^k\ \alpha^2 - 2^{k-1}(k+o.5n_0)=0$ for k<5

$\alpha^4+2^{k-1}\ \alpha^2 - 2^{k-1}(k+o.5n_0)=0$ for k≥5

TABLE II gives the Coded value and Decoded value for the composition design of the using materials of our experiment.

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TABLE II: Composition of material according to design: - coded value and decoded value

Coded value	De coded value		
	R_1(sand : cement)	R_2(water :cement)	R_3(polymer : cement)
+1	6	0.50	0.56
-1	1	o.25	0.02
0	3.5	0.375	0.29
+1.215(+α)	6.5375	0.526875	0.61805
-1.215(-α)	0.4625	0.223125	0.07805

For total number of observation 15, TABLE III gives the design matrix and observation results, where $\bar{Y}$ is the compressive strength of the tested polymer mortar.

TABLE III: Design matrix and observation results

Design points N	X_0	X_1	X_2	X_3	X_1X_2	X_1X_3	X_2X_3	X_1^2	X_2^2	X_3^2	$\bar{Y}$	S_i^2
1	+1	+1	+1	+1	+1	+1	+1	0.27	0.27	0.27	$\bar{Y}_1$	S_1^2
2	+1	+1	+1	-1	+1	-1	-1	0.27	0.27	0.27	$\bar{Y}_2$	S_2^2
3	+1	+1	-1	+1	-1	+1	-1	0.27	0.27	0.27	$\bar{Y}_3$	S_3^2
4	+1	+1	-1	-1	-1	-1	+1	0.27	0.27	0.27	$\bar{Y}_4$	S_4^2
5	+1	-1	+1	+1	-1	-1	+1	0.27	0.27	0.27	$\bar{Y}_5$	S_5^2
6	+1	-1	+1	-1	-1	+1	-1	0.27	0.27	0.27	$\bar{Y}_6$	S_6^2
7	+1	-1	-1	+1	+1	-1	-1	0.27	0.27	0.27	$\bar{Y}_7$	S_7^2
8	+1	-1	-1	-1	+1	+1	+1	0.27	0.27	0.27	$\bar{Y}_8$	S_8^2
9	+1	+1.215	0	0	0	0	0	0.747	-0.73	-0.73	$\bar{Y}_9$	S_9^2
10	+1	-1.215	0	0	0	0	0	0.747	-0.73	-0.73	$\bar{Y}_{10}$	S_{10}^2
11	+1	0	+1.215	0	0	0	0	-0.73	0.747	-0.73	$\bar{Y}_{10}$	S_{11}^2
12	+1	0	-1.215	0	0	0	0	-0.73	0.747	-0.73	$\bar{Y}_{11}$	S_{12}^2
13	+1	0	0	+1.215	0	0	0	-0.73	-0.73	0.747	$\bar{Y}_{12}$	S_{13}^2
14	+1	0	0	-1.215	0	0	0	-0.73	-0.73	0.747	$\bar{Y}_{13}$	S_{14}^2
15	+1	0	0	0	0	0	0	-0.73	-0.73	-0.73	$\bar{Y}_{14}$	S_{15}^2

Since design matrix is orthogonal and all regression coefficients are determined independently of one another using the following formula:

$$bj = \frac{\sum_{i=1}^{15} XjiYi}{\sum_{i=1}^{15} Xji^2}$$

As a result the following estimated regression equation is derived from the matrix with transferred columns for quadratic terms:

$$\hat{Y} = b_0 + b_1X_1 + b_2X_2 + \cdots + b_kX_k + b_1X_1X_2 + \ldots + b_{k-1,k}X_{k-1}X_k + b_{11}X_1^2 + \ldots + b_{kk}X_2^2$$

Where b_0, b_1, b_2 and b_k are linear coefficients; b_{12}, … $b_{k-1,k}$ are cross-product coefficients and b_{11}, … b_{kk} are quadratic coefficients, X_1 refers to the coded value of sand and X_2 refers to the coded value of polymer and X_3 refers to the coded value of water.

D. Data treatment method:

Data have been treated by the following procedure:

Test of homogeneity of Experimental value

For each raw the design matrix the mean of m replicate observation is found.

$$\bar{Y}_i = \frac{1}{2}Y_{iu} \quad , i = 1, 2 \ldots 15$$

And the sample variance is determined.

$S_i^2 = \frac{1}{2-1}\sum_{u=1}^{m}(Y_{iu} - \bar{Y}_i)^2$ Here, i=1, 2, 3…….. 15; u= 1, 2.

The sample variances are tested for homogeneity on the basis of Cochran's equation, to this end the ratio is formed:

$$G = \frac{S^2max}{\sum_{i=1}^{15} Si^2}$$

Here S^2_{max}= maximum value of the sample variance.

$\sum_{i=1}^{N} Si^2$ = Sum of all variance.

The ratio G is compared with tabulated value of Cochran's criterion, $G < G_{\alpha-1}(\nu_1, \nu_2)$

Here α = significance level and ν_1 = m-1 and ν_2 = 15 and m=2 and N=15.

Test of Significance of the Coefficient

The error mean square, $s_e^2 = \frac{1}{N}\sum_{i=1}^{15} Si^2$

The error mean square has a number of degrees $\nu$of freedom ν is given by,

$$\nu = N(m-1)$$

Since the variances of $\bar{Y}$ obtained from a sample of size m is m times less than the variances of sample observation.

$$s_{\bar{Y}}^2 = \frac{s_e^2}{m}$$

The sample variances $s_{b_j}^2$ is the jth component is given by

$$s_{b_j}^2 = \frac{s_e^2}{Nm}$$

The coefficient of the regression equations are tested for significance of the basis of the Student t-test. The t-ratio is formed

$$t_j = \frac{|b_j|}{s_{b_j}}$$

Here b_j = j estimated coefficient of the regression equation and standard deviation of the jth coefficient.

The value of t_j is compared to the tabulated value $t_\alpha(v)$ for the selected significance level α and the number of degree of freedom ν = N (m-1).

If $t_j < t_\alpha(v)$, then the hypothesis is that, the linear term of regression equation,β_j, equals zero is accepted and the respective sample coefficient b_j is deleted from the estimated regression equation since it is not significant. Owing to the fact that the design matrix is orthogonal, the remaining coefficients do not require calculations.

Adequacy Test

The experimenter finds whether or not the estimated regression equation fits the experimental data using **Fisher's test,** a procedure analogous to that employed in handling the data of passive experiment. In the design matrix, each observation is repeated m times. For the goodness of fit (adequacy) test, one forms Fisher's variance ratio.

$$F = \frac{s_g^2}{s_e^2}$$

Where,s_g^2 is the adequacy mean square and that is given by the following equation;

$$s_g^2 = \frac{m\sum_{i=1}^{15}(\bar{Y}_i - \hat{Y}_i)^2}{N-l}$$

Here l is number of significant coefficient in the regression equation.

The equation is an adequate fit to the experiment if the F-ratio is smaller than the tabulated value of $F_{1-\alpha}(v_1, v_2)$.

$$F < F_{1-\alpha}(v_1, v_2)$$

Where α is the significance level v_1 is the number of degrees of freedom for the adequate variance.

$$v_1 = N - l$$

And v_2 is the number of degree of freedom of the replication mean square.

$$v_2 = N(m-1)$$

If once has

$$F > F_{1-\alpha}(v_1, v_2)$$

Then the adequately approximating model is obtained by increasing the order of the approximating poly-mial.

III.RESULT AND DISCUSSION

a. Design for 14 days regression equation:

Value of the coefficients for the 14days lab test result:

$b_0 = 20.571$ $b_1 = 0.580685$
$b_2 = -0.16658$ $b_3 = 2.905452$
$b_{12} = 1.688125$ $b_{13} = 10.44438$
$b_{23} = 2.964375$ $b_{11} = 0.281139$
$b_{22} = -0.73391$ $b_{33} = -5.05127$

Substituting the coefficient b_j with the numerical value, the regression equation is:

$\hat{Y} = 20.57 + 0.58X_1 - 0.167X_2 + 2.91X_3 + 1.69X_1X_2 + 10.45X_1X_3 + 2.96X_2X_3 + 0.28X_1^2 - 0.73X_2^2 - 5.05X_3^2$

The sample variances are tested for homogeneity on the basis of Cochrans's equation, to this end the ratio is formed, G = 0.343

The ratio G is compared with the tabulated value of Cochran's criterion,G<$G_{\alpha-1}(v_1, v_2)$

From the tabulated value $G_{\alpha-1}(v_1, v_2)$ = 0.3346.

So, the sample variances are homogeneous.

The coefficients of the regression equation are tested for significance of the basis of the Student t-testand we found the coefficient b_1, b_2, b_{11}, b_{22} are insignificant.

Thus the 14 days regression equation is:

$\hat{Y} = 20.57 + 2.91X_3 + 1.69X_1X_2 + 10.45X_1X_3 + 2.96X_2X_3 - 5.05X_3^2$

The estimated regression equation is tested to see how it feet the observation, using Fisher's Testand the variance ratio,F =8.068242 and $F < F_{1-\alpha}(v_1, v_2)$

The adequacy test did not satisfy.

b. Design for 28 days equation

Value of the coefficients for the 14days lab test result:

$b_0 = 26.15933$ $b_1 = -4.70315$
$b_2 = -1.842513$ $b_3 = 6.897096$
$b_{12} = 4.71875$ $b_{13} = 6.36625$
$b_{23} = 0.04375$ $b_{11} = 2.985902$
$b_{22} = -1.1149$ $b_{33} = -4.64053$

Substituting the coefficient b_j with the numerical value, the regression equation is:

$\hat{Y} = 26.16 - 4.70X_1 - 1.84X_2 + 6.89X_3 + 4.71875X_1X_2 + 6.36625X_1X_3 + 0.04375X_2X_3 + 2.985902X_1^2 - 1.1149X_2^2 - 4.64053X_3^2$

The sample variances are tested for homogeneity on the basis of Cochran's equation, to this end, the ratio is formed: G =0.351184

The ratio G is compared with the tabulated value of Cochran's criterion, G<$G_{\alpha-1}(v_1, v_2)$

From the tabulated value $G_{\alpha-1}(v_1, v_2)$ = 0.4709.So, the sample variances are homogeneous.

The significance of the coefficient can be tested; using student t-test and found that the coefficient b_{23} is insignificant. Thus the regression equation is:

$\hat{Y} = 26.16 - 4.70X_1 - 1.84X_2 + 6.89X_3 + 4.71875X_1X_2 + 6.36625X_1X_3 + 2.985902X_1^2 - 1.1149X_2^2 - 4.64053X_3^2$

Now the estimated regression equation is tested to see how it feet the observation, using Fisher's Test, and the variance ratio, F= 43.45906 and $F < F_{1-\alpha}(\nu_1, \nu_2)$
The Adequacy test did not satisfy.

IV. CONCLUSION

This study shows that second order orthogonal regression equation which predicts compressive strength by model equation for polymer modified mortar (PMM).The regression equation doesn't satisfy the adequacy test, but it satisfies the homogeneity test of the experimental value and test of significance of the coefficient. As the values are not discrete, it can be said that number of observations or more complex mix ratio can be satisfied the adequacy test. In near future, this model equation can help optimum use of material and will be helpful for economical in construction work. The failure pattern of polymer modified mortar is appealing as it does not demolish immediately and totally spearing sometime to escape from probable disaster.

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