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Statistical Model for Compressive Strength of Metakaolin Concrete

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Abstract: In the present research work, investigations were carried out to improve the performance of concrete in terms of strength by incorporating metakaolin (MK) as mineral admixture in concrete. Parametric study was carried out by considering w/c ratio, various percentage of MK and age of concrete as parameters to understand the effect of each parameter. The study was conducted for different w/c ratios of 0.32, 0.35, 0.4 and 0.5. The MK proportion was varied from 0 to 15% with an increment of 5% and age of concrete from 3 to 90 days were considered and experiments performed accordingly. The effects of above said parameters on the various properties of concrete such as workability, compressive strength and pH of concrete were investigated, and the results of metakaolin concrete were compared with the conventional concrete. From the results, it was observed that MK concrete showed greater strength for higher water cement ratios (0.4 & 0.5). A Multiple non-linear regression analysis was used to develop a statistical model to predict the strength and found to have good correlation between the observed and predicted values. It was concluded that the concrete developed in this study have significant potential for use on real time projects.
Keywords: Metakaolin, w/c ratio, compressive strength, pH.

I. INTRODUCTION

Reinforced concrete has come to stay as the most accepted construction material in world wide. It is a composite consisting of steel and concrete, which, in itself, is another heterogeneous material consisting of cement and aggregates with varied shapes, sizes, configurations and orientations. During service, the reinforcement almost invariably undergoes rusting and hence deterioration has been observed in concrete structures well within their service life especially in some aggressive environments, leading to premature failure [1]. The economic loss due to premature failure and the cost of rebuilding runs up to several percentage of national income. In this context it is of paramount importance to produce durable concrete structures with longer life and less damage due to unforeseen factors. One of the main causes for damages is due to corrosion of reinforcing steel and therefore of late, there has been major interest in developing technology for corrosion control by adding mineral admixtures in concrete. Mineral admixtures can be used to enhance the corrosion-control potential of concrete by reducing permeability. The pozzolanic reaction in mineral admixtures converts the CH into more of the CSH thus leading to reduced permeability. With the use of mineral admixtures, the ingress of moisture, oxygen, chlorides, and aggressive chemicals are slowed significantly - thus improving durability and serviceability[2].These mineral admixtures, have been incorporated in concrete, either in addition to the cement or through the replacement of a portion of cement, to enhance the properties of concrete both in plastic and hardened states [3-6]. As a result, the use of these SCM's in concrete industry helps in two ways: proper utilization of by products and enhancement of concrete properties. Many studies have been carried out on the use of admixtures, however search for efficient alternative admixture is still continuing. Age of concrete and w/c also plays a major role in producing durable concrete [7-9]. Hence in the present work it is proposed to study the influence of MK, w/c ratio and various curing period on strength characteristics of concrete. Further an attempt is made to establish a correlation between various factors such as water-to-cement ratio, amount of admixtures and age of concrete, which influence the strength. The experimental program was planned to study the effect of various parameters like, mineral admixture, w/c ratio, age of concrete on the mechanical properties. Further, an attempt has been made to develop correlation between various factors, such as w/c, amount of metakaolin and age of concrete, which influence strength of concrete.

A Multi variate non-linear regression model was established to predict the strength of metakaolin concrete. A non-linear regression analysis was used to develop a statistical relationship between compressive strength, w/c ratio, MK percentage and age of concrete. In a multivariate experiment, there are several important problems with the conventional approach of changing only one or two variables in a run, because the variables often interact with each other. For example, the amount of admixture optimized for one w/c ratio, may not be applicable for other w/c ratios. These limitations of a classical method can be eliminated by considering the relative importance of all the influencing variables simultaneously by statistical methods. The functional relationships between the independent process variables such as water-to-cement ratio (w/c), amount of admixture, age of concrete and the dependent variable strength of the concrete were investigated. The analysis was done by response surface methodology (RSM) with coded values of the variables. Common statistical tools, such as analysis of variance, F-test, the student's t-test, and lack of fit were used to define the most important process variables affecting the strength.

II. EXPERIMENTAL PROGRAM

Four series of concrete mixes were prepared at the w/c ratios of 0.32, 0.35, 0.4 and 0.5.In each series, cement was partially replaced by MK with different proportions and a control mix without any admixture to assess their influence on strength of concrete for various curing periods. The percentage of MK varies from 5 to 15% by weight of cement with an increment of 5%. Detailed experimental investigations for various properties of concrete such as workability, compressive strength and *pH of* Concrete have been done for various mixes. The compressive strength was determined at various ages (3,7,14,28 & 90 days) . The results were compared to the same properties of a control concrete mixture. pH of each concrete mixture was measured to know the alkalinity of concrete. In total, 240 cubes of 100 mm size were cast for control and MK concrete for testing the compressive strength.

Materials Used

Ordinary Portland Cement (OPC) of 53 grade of Indian Standard was used in this study and MK was obtained from 20 microns Ltd. The chemical composition and physical properties of the cement determined as per IS: 12269-1987, Indian Standard Specifications for 53 Grade Ordinary Portland Cement are shown in Table 1. Table 2 presents the physical and chemical properties of metakaolin.

Aggregates

Locally available river sand lesser than 4.75 mm sieve size conforming to grading Zone III of IS 383 – 1970 was used as fine aggregate and crushed granite stones of 12.5 mm maximum size were used as coarse aggregates. The physical properties of fine and coarse aggregates were determined as per IS: 2386- Indian Standard Code of Practice for Methods of Test for Aggregate and are listed in Table 3.

Super plasticizer

Super plasticizer was used to obtain sufficient workability for the mixes of low w/c ratios. The super plasticizer used was CERAPLAST-300supplied by CeraChem Ltd. It was a brown colour solution of sulphonatenaphthalene formaldehyde type of super plasticizer. The specific gravity is about1.20.

Water: Locally available potable water was used for mixing and curing of concrete.

Mix Proportions

The mix proportions were arrived based on IS recommendations (IS 10262 – 1982). The mix designation and the mix ratios for the control mix are given in Table 4 and 5. The slump values were maintained between 50 and 100 for all the mixtures by adding suitable dosage of super plasticizer (Ceraplast-300).

Tests on Fresh and Hardened Concrete

Compressive strength

Slump test was conducted for measuring the workability of concrete and the results are tabulated in Table 6. The compressive strength of the plain and admixed concrete were determined by conducting compression test as per IS specification [10]. 100 mm concrete cubes were cast as per mix proportion for different water cement ratios and for the various percentage of replacement of cement with the mineral admixture (MK). These specimens were cast in steel moulds and compacted on a table vibrator. After 24 hours of casting, the cubes were demoulded and cured in water for 3, 7, 14, 28& 90 days. Three samples each were cast for various water cement ratio, percentage of replacement level and period of curing respectively. After the required curing period, the cubes were tested in the computerised compression-testing machine of capacity 3000 kN. The strength variations are shown in figures 1 and 2.

pH of Concrete

Various concrete samples were crushed mechanically and powered samples passed through 75microns sieve were collected for both control specimen and MK admixed concrete. The powdered sample thus collected was mixed with the distilled water in the ratio of 1:10 [11]. After 2 hours, the solution was filtrated and its pH was measured using a portable pH meter (Elico Ltd -Model 120, type: 003). The pH values are shown in fig 04.

Table 1. Physical and Chemical properties of Cement

Physical properties			Chemical composition		
01	Consistency	37%	01	Lime(CaO)	62.72 %
02	Specific gravity	3.2	02	Silica(SiO_2)	31.38 %
03	Initial setting time	55 min	03	Fe_2O_3andAl_2O_3	6.89%
04	Final setting time	355 min	04	Magnesia (MgO)	2.02%
05	Fineness by sieving	1% residue	05	SulphurTrioxide (SO_3)	3.14%

06	Soundness	1.5 mm	06	Loss on Ignition	2.76%
			07	Insoluble residue	1.25%

Table 2. Physical and Chemical Properties of MK

Physical properties			Chemical composition		
01	Colour	Off white	01	Lime(CaO)	0.39%
02	Specific gravity	2.5	02	Silica(SiO_2)	96.88%
03	Bulk density	800kg/m^3	03	Fe_2O_3andAl_2O_3	0.28%
04	Average particle size	1.5 mm	04	Magnesia(MgO)	0.08%
05	Fineness	1500m^2/kg	05	Sulphur Trioxide(SO_3)	-
			06	Losson Ignition	0.68
			07	Insoluble residue	1.69%

Table 3. Physical Properties of Aggregates

S.No	Physical properties	Fine aggregate	Coarse aggregate
01	Fineness modulus	2.60	7.93
02	Specific gravity	2.65	2.78
03	Bulk density	1.66g/cc	1.61g/cc
04	Void ratio	37.59%	45.61%
05	Bulking of sand	58%	-
06	Water absorption	1.01%	0.84%

Table 4. Mix Designation

S.No	Composition of mix	Mix designation
	w/cm = 0.32	
01	100%OPC	C_1
02	95% OPC+5% MK	M_1
03	90% OPC+10%MK	M_2
04	85% OPC+15%MK	M_3
	w/cm = 0.35	
05	100%OPC	C_2
06	95% OPC+5% MK	M_4
07	90% OPC+10%MK	M_5
08	85% OPC+15%MK	M_6
	w/cm = 0.40	
09	100%OPC	C_3
10	95% OPC+5% MK	M_7
11	90% OPC+10%MK	M_8
12	85% OPC+15%MK	M_9
	w/cm = 0.50	
13	100%OPC	C_4
14	95% OPC+5% MK	M10
15	90% OPC+10%MK	M11
16	85% OPC+15%MK	M12

Table 5. Mix Ratio for Different w/cm Ratio

S.No	Mix	Slump (mm)	Mix Ratio
01	C_1	65	1:0.69:1.61
02	C_2	70	1:0.90:2.10
03	C_3	70	1:0.99:2.31
04	C_4	90	1:1.20:2.80

Table 6. Slump Values

S.No	Mix Designation	Super plasticizer in % by weight of cement-metakaolin				Slump in mm			
		w/cm = 0.32	w/cm= 0.35	w/cm= 0.40	w/cm= 0.50	w/cm= 0.32	w/cm= 0.35	w/cm= 0.40	w/cm= 0.50
01	C	1	1	0.8	-	65	70	70	90
02	MK–5	1.1	1.1	0.6	0.1	75	62	75	52
03	MK–10	1.2	1.2	0.9	0.3	72	86	53	55
04	MK–15	1.4	1.4	1.1	0.7	57	53	75	65

Table 7. Important Findings

Ad mixture	Optimum % of admixture	Influence of	
		w/c m ratio	Curing period
MK	15%MK - 0.32 w/cm10% MK – all other w/cm	Effect of MK is more pronounced at higher w/cm	Increase in compressive strength is more pronounced at early ages

Table 8. Multiple Regression Coefficients, T and P Values for Strength of MK Concrete –Full Model

Term	Coef	SE Coef	T	P
Constant	58.074	1.166	49.822	0
w/cm	-5.111	0.527	-9.700	0
MK	2.985	0.546	5.471	0
Time	9.881	0.496	19.931	0
w/cm*w/cm	-4.564	0.841	-5.427	0
MK*MK	-5.988	0.777	-7.705	0
Time*Time	-9.747	1.135	-8.587	0
w/cm*MK	-1.120	0.610	-1.834	0.071
w/cm*Time	0.304	0.619	0.490	0.625
MK*Time	-3.264	0.631	-5.174	0.000

R^2 = 91.99%, SE Coef – Standard Error Coefficient

III. RESULTS AND DISCUSSION

Workability

From the results of slump test, shown in Table 6, it has been observed that the MK concrete gives improved workability than the control mix at lower w/c ratios. This is because of the fact that, fineness and glassy surface of the pozzolanic material which offer better lubricating effect resulting the better workability. However, there was a substantial reduction in workability when the MK replacement level was increased from 10% to 15% and is attributed to its high chemical activity and high specific surface leads to increased water requirement. From the literature it is evident that, further adjustments in the fineness results in retaining cohesiveness and reducing the loss in workability [12].

Compressive strength

From the test results shown in Figures 1& 2, it has been observed that there is a general trend of increasing strength with age for all the concrete mixtures with and without addition of MK and MK was found to be effective in improving the compressive strength. For all the mixes the highest rate of strength development over control concrete was observed up to 14 days (25% to 102%) and this is because the pozzolanic reaction starts at very early stages of hydration, and hence the contribution of MK towards strength is more significant at early ages. This finding is similar to studies by Tiwari. A.K, et al, Khatib, J.M et al [13, 14]. The replacement of 10% cement by MK was found to be more beneficial for all w/c ratios except for 0.32 and increase of MK beyond 10% tend to reduce the strength. The use of 10% MK increases the compressive strength around 54 % till 14 days and less than 10% after 14 days than control concrete at 0.35 w/c.

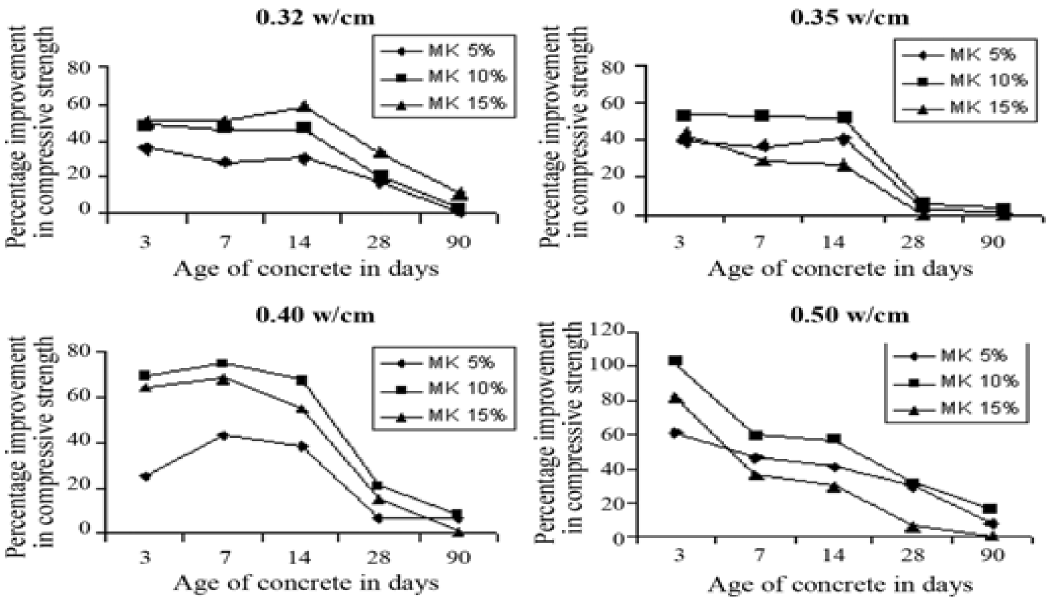

Fig.1. Improvement of Compressive Strength Compared to Control Specimen with Age for Different MK Proportions & w/cm Ratios

For the same replacement level, at 0.4 w/c, it behaves in a similar fashion up to 14 days with slightly higher increase and considerable increase was observed at 28 days also. The relative strength enhancement over control concrete at 7, 14 and 28 days were approximately 75%, 67% and 21% respectively. At 0.5w/c ratio, addition of 10% MK increases the relative compressive strength around 100% at 3 days and approximately by 60% to 32% from 7 days to 28 days. It is inferred from the above results that, the highest improvement over control concrete is achieved at higher w/c and hence the role of metakaolin is predominant at higher w/c ratio. When w/c ratio becomes 0.32, it can be seen from fig1, M15 showed better results than M10 concrete, and its behaviour was similar to 10 % MK with 0.35 w/c. The maximum compressive strength of 59.25 N/mm^2 was observed at 0.4 water cement ratio with 10% replacement of cement by MK. The important findings are given in Table 7, and among all w/c ratios, the optimum w/c ratio recommended is 0.4, with 10% MK.

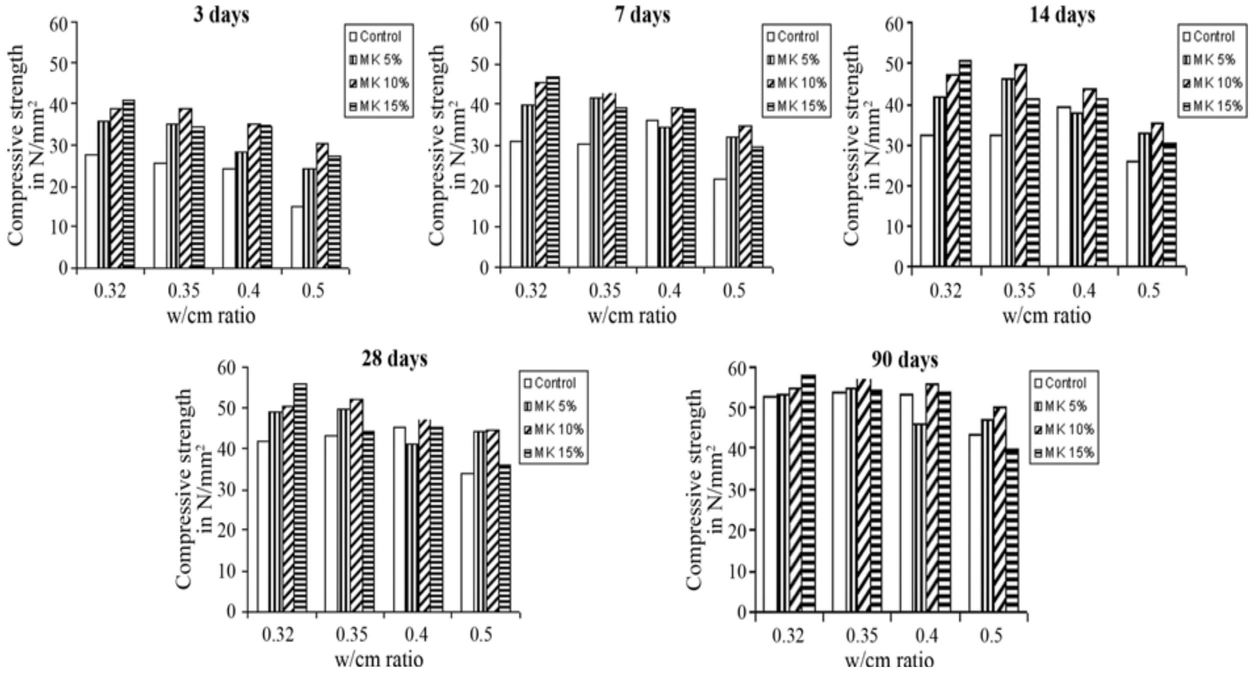

Fig.2. Effect of w/cm Ratio on Compressive Strength for Different Periods of Curing

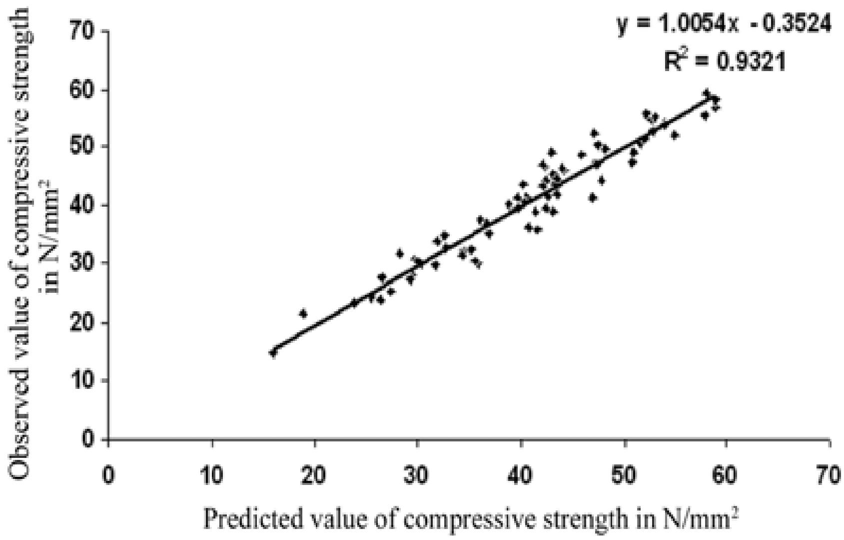

Fig.3. Comparison of Experimental Data with the Predicted Values from the Model

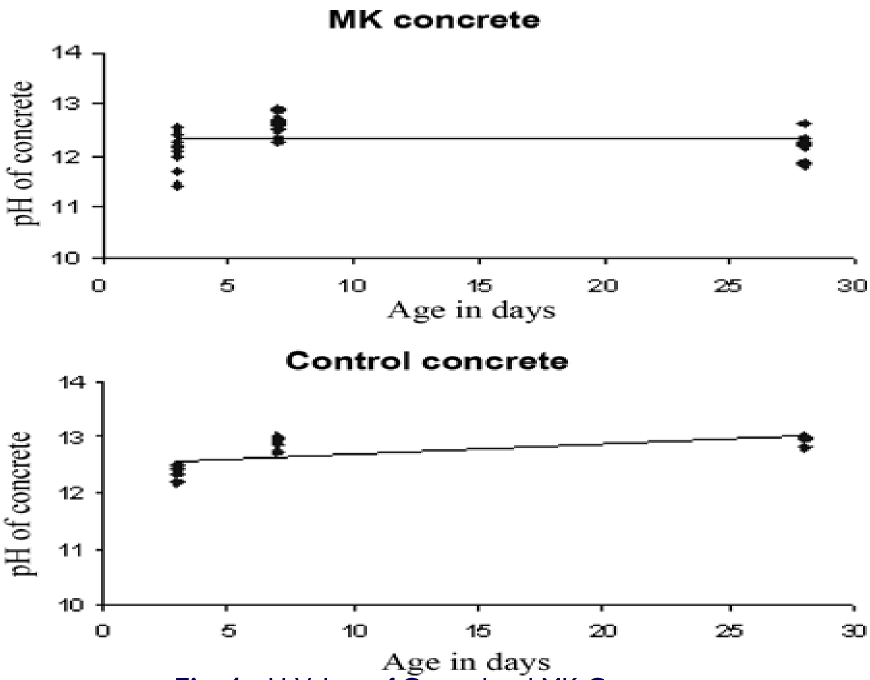

Fig. 4. pH Values of Control and MK Concrete

Non-linear regression model

The following quadratic equation was used to describe the behaviour of the system

$$c = \beta_o + \Sigma \beta_i X_i + \Sigma \beta_{ii} X_i^2 + \Sigma \beta_{ij} X_i X_j \text{ -----(1.0)}$$

Where fc = Predicted response, β_o = constant, X_i = Independent variables, β_i = linear coefficients, β_{ii} **=** Quadratic coefficients β_{ij} = Cross product coefficients. The multiple regression coefficients and the associated standard errors were determined by using statistical software MINITAB 14. Water-to-cement ratio (w/c) (X_1), amount of admixture (X_2), age of concrete (X_3) were chosen as predictor variables and the strength of the concrete fc as response variable. The student t-test was used to determine the significance of the regression coefficients of the parameters and the P values <0.05 indicated the significance of the model terms. A quadratic regression model was developed by using codal units from the experimental data and is shown in Table 8. The sum of the squares used to estimate the factors and F ratios are given.

The full quadratic model equation for MK concrete is fc

$$= 58.074 - 5.111X_1 + 2.985X_2 + 9.881X_3 - 4.564X_1^2 - 5.988X_2^2 - 9.747X_3^2 - 1.12X_1X_2 + 0.304X_1X_3 - 3.264X_2X_3 \text{------(1.0)}$$

It was observed that, the linear and quadratic effects of all the variables are found to be highly significant whereas the interaction effects between the variables w/c and Time, w/c and MK were considered to be insignificant in comparison to the linear and quadratic effects. Therefore these non-significant values (i.e. X_1X_2, and X_1X_3) are eliminated from model and regression was repeated for the reduced model. Values of the statistical parameters t, p and F confirmed the statistical significance of the factors in the reduced model.

Residuals plots were drawn to verify the adequacy of the model and are shown in figure 3. Run number 39 whose standardized residual is -2.69, is found to be outlier and therefore it was eliminated from the model. All other points were found to fall in the range of +2 to -2.Regression was repeated once again and uniform distribution of the residuals in the normal plot and a bell shaped histogram confirmed the adequacy of the model. Value of R^2 is found to be 0.9242, indicating that 92.42% of the total variation could be explained by the regression model. The predicted value were compared with experimental results**,** indicating predicted data were close to experimental data

Therefore Reduced model equation for MK concrete was fc

$$= 58.531 - 5.321X_1 + 3.4 X_2 + 9.876X_3 - 4.651X_1^2 - 5.74X_2^2 - 10.378X_3^2 - 3.267X_2X_3 \text{ ------ (2.0)}$$

It is understood that, the interactive effects between MK and time is more significant than other interactive effects. Thus relatively lower curing period and lower percentage of MK played an important role in increasing the strength. High R^2 value indicates a strong association between the pairs of variables. Therefore the model explains the variations in the predictor variables satisfactorily. Hence this model (shown in equation 2) can be used to predict the strength of MK concrete within the experimental values under similar conditions. These readily available models will be very much useful to the practicing engineers and researchers with simple input data.

pH of concrete

The pH value is very important for maintaining the passivity of steel. The results of pH of control and admixed concrete are shown in fig 4. The pH value for control concrete varies from 12.2 to 13, with slight increase as the age of the concrete increases. This is due to the fact that during hydration, more calcium hydroxide is liberated, which increases the pH. The pH values of MK were measured for various replacement levels and these ranges between 11.42 to 12.9 from 3 to 28 days and these values are marginally lower than that of control mix, but the reduction is insignificant. Since the reduction in pH is not significant, it is inferred that addition of MK up to 15% does not adversely affect the alkalinity of concrete.

MK concrete showed slightly lesser values at 3 days than 7 and 28 days and this reduction can be attributed to the fact that since the rate of pozzolanic reaction will be higher at early ages, the absorption of calcium hydroxide by MK was more, which reduces the pH at early ages.

IV. CONCLUSION

The results of the present study clearly show that MK is effective in improving the strength of concrete. Based on the results, the following conclusions were made.

- MK concrete gives improved workability than the control mix at lower w/c ratios. However the workability is significantly reduced at 15% MK.
- The increase in compressive strength for MK concrete was greater especially at higher water cement ratios (i.e. for 0.4 and 0.5) and hence more suitable for higher w/c ratios. From the studies an optimum percentage of MK was found to be 10% for all w/c ratios except for 0.32 and for 0.32 it was 15%.. MK concrete showed higher increase in strength at early ages and beyond 28 days it was found to be less than 10%. The maximum compressive strength of 59.25 N/mm^2 was observed at 0.4 w/c with 10% MK.
- The strength of concrete predicted from the multiple non-linear regression model was found to be in close agreement with R^2 value of 0.924.It is a good technique for studying the influence of various process variables on response by reducing the number of experiments and therefore saving time, energy and money.
- Addition of MK reduced the pH values, but the reduction is insignificant, since the pH values are still above 11.5, which will be helpful for maintaining the steel in a passive state itself.

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