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Development of high strength high performance concrete using Indian metakaolin

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ABSTRACT

A study had been undertaken to study the effect of incorporating local metakaolin (MK) on the properties of high strength concrete designed for a constant water/binder ratio of 0.3. Metakaolin (MK) mixtures with partial cement replacement of 5, 10 and 15 % were designed for target strength of 90 MPa. From the results of the investigation, it was observed that 10% replacement level was the optimum level in terms of compressive strength. Beyond 10% replacement levels, the strength was decreased but remained higher than the control mixture. Compressive strength of nearly 106 MPa was achieved at 10% replacement. Splitting tensile strength and elastic modulus values have also followed similar trend. In durability performance tests MK concretes have exhibited high resistance compared to control concrete and the resistance increases as the metakaolin percentage increases.

Keywords. metakaolin, strength, elastic modulus, permeability, absorption

1. INTRODUCTION

The necessity for the development of high strength and high performance concretes is increasing gradually because of the demands imposed by the construction industry in recent times. In the last three decades, supplementary cementitious materials such as fly ash, silica fume and ground granulated blast furnace slag have been judiciously utilized as cement replacement materials as these can significantly enhance the strength and durability characteristics of concrete in comparison with ordinary Portland cement (OPC) alone, provided there is adequate curing (Neville, 1997). Hence, it is common to produce high-performance concrete at lower w/b ratios by incorporating these supplementary materials. Fly ash addition proves most economical among these choices. However, when high strength is desired, use of silica fume is more useful (Basu, 2003). Silica fume gives a very good particle packing and, combined with its strong pozzolanic property increases the resistance of the concrete to aggressive environments also (Abdul and Wong, 2005). Though, silica fume initially considered as industrial waste, has become a top-of-the-range product for which demand is constantly increasing in the construction industry. However, this product is rather expensive. In India, good quality silica fume is mostly imported and the cost is 9 to 10 times the cost of ordinary Portland cement (OPC).

Metakaolin or calcined kaolin, another type of pozzolan, produced by calcination has the potential to replace silica fume as an alternative material. In India metakaolin can be produced in large quantities, as it is a processed product of kaolin mineral which has wide spread proven reserves available in the country (Basu *et al.,* 2000; Tiwari and Bandyopadhyay, 2003). Presently the market price of metakaolin in the country is about 3 to 4 times that of cement. Hence the use of metakaoiln proves economical over that of silica fume. Previously, researchers have shown a lot of interest in metakaolin as it has been found to possess pozzolanic and microfiller characteristics (Poon *et al.*,2001; Wild and Khatib, 1997; Wild and Khatib, 1996). However, very limited test data are available regarding the performance of the commercially available metakaolin and Indian cements in the case of high strength concrete in the country (Basu, 2003; Basu *et al.*,; 2000, Pal *et al.*,2001). The objective of this study was to investigate the effect of using local calcined kaolin or metakaolin obtained commercially as pozzolan on the development of high strength and permeability/durability characteristics of concrete for a very low w/b ratio of 0.3. In addition, the optimum replacement levels in terms of strength and durability properties were determined by varying the amount of metakaolin as partial cement replacement.

2. EXPERIMENTAL INVESTIGATION

An experimental program was designed to produce a high strength concrete by adding several combinations of metakaolin. The materials used and the experimental procedures are described in the following sections.

2.1 Materials

The cement used in all mixture was normal Ordinary Portland cement (53 grade) conforming to IS: 12269 (BIS, 1987). Commercially available metakaolin was used as mineral additive. Their chemical composition is specified in Table 1.

Table 1. Characteristics of cement and metakaolin

Chemical composition	Cement (%)	Metakaolin (%)
Silica (SiO_2)	34	54.3
Alumina (Al_2O_3)	5.5	38.3
Ferric Oxide (Fe_2O_3)	4.4	4.28
Calcium oxide (CaO)	63	0.39
Magnesium oxide (MgO)	1.26	0.08
Sodium oxide (Na_2O)	0.1	0.12
Potassium oxide (K_2O)	0.48	0.50
Sulphuric anhydride (SO_3)	1.92	0.22
Loss on ignition (LOI)	1.3	0.68
Blaine (m^2 / kg)	360	15000*
Specific gravity	3.15	2.5

* B.E.T. Surface Area

Good quality aggregates have been procured for this investigation. Crushed granite with nominal grain size of 20 mm and well-graded river sand of maximum size 4.75 mm were used as coarse and fine aggregates, respectively. The specific gravities of aggregates were determined experimentally. The coarse aggregates with 20, 12.5 mm fractions had specific gravities of 2.91 and 2.80, whereas the fine aggregate had specific gravity of 2.73, respectively. Commercially available poly carboxylate ether (PCE) - based super-plasticizer (SP) was used in all the concrete mixtures.

2.2 Mixture proportions

Trials mixtures were prepared to obtain target strength of more than 90 MPa for the control mixture at 28 days and the water/binder ratio for all the mixtures was kept constant at 0.30. The details of the mixtures for the study are presented in Table 2. Four different mixtures (MK0, MK5, MK10 and MK15) were employed to examine the influence of low water to binder ratio on concretes containing MK on the mechanical and durability properties. The control mixture (MK0) did not include metakaolin. In mixtures MK5, MK10 and MK15, cement content was partially replaced with 5%, 10%, and 15% MK (by mass) respectively. Trial mixtures were conducted to determine the optimum dosage of superplasticiser for each of the mixtures in order to achieve the target slump of 100 ± 25 mm.

Table 2. Details of the mix proportions in kg/m^3

Constituent	MK0	MK5	MK10	MK15
Cement	533.33	506.67	480.0	453.33
Water	160	160	160	160
Fine Aggregate	677	666	655	645
20mm	602	593	583	574
12.5mm	599	589	580	570
Metakaolin	0	26.67	53.33	80.0
S.P	2.13	2.93	3.47	4.26
Slump (mm)	130	110	110	100
Plastic density	2520	2477	2446	2421

2.3. Specimens and curing

All the specimens were cast on mechanical vibration table. After casting, all the specimens were covered with plastic sheets and water saturated burlap, and left at room temperature for 24 hours. The specimens were demolded after 24 hours of casting and were then cured in water at approximately 27^0C until the testing day.

2.5. Experimental procedures

The workability of the fresh concrete is measured by using the standard slump test apparatus. The unconfined compressive strength was obtained, at a loading rate of 2.5 kN/s at the age of, 3, 7, 28 and 90 days on 3000kN machine. The average compressive strength of three specimens was considered for each age. The split tensile strength was also tested on the same machine at the age of 28 days. The elastic modulus was determined at the age of 28 days according to ASTM C 469 (ASTM, 2002). All the specimens were tested on saturated surface dry condition. The permeability characteristics of the concretes were assessed at 28 days using Germann Water permeability Test (GWT meter, Denmark). Also the water penetration depths under pressure were performed on 150mm cubes as per EN 12390-8 (BS, 2000) at 28 days. The absorption test was carried out on two 100 mm cubes as per ASTM C 642 (ASTM, 2002) at 28 days of water curing. The rapid chloride penetrability test was conducted in accordance with ASTM C 1202 (ASTM, 2002). These were also determined at 28 days.

3. RESULTS AND DISCUSSIONS

3.1. Fresh Properties

3.1.1. Plastic Density

The results of the plastic densities with respect to the corresponding metakaolin percentages are given in Table 2. From this it can be seen that the plastic densities varied between 2421 - 2520 kg/m^3. The slight reduction in the densities of metakolin concretes is due to the lower specific gravity of metakaolin compared to cement alone.

3.1.2. Superplasticizer demand

In this study, different dosages of superplasticiser were added to the different mixtures in order to maintain the consistency or workability in terms of target slump of 100±25 mm. It can be seen from Table 2, the superplasticiser demand increased with increase in the metakaoiln replacements.

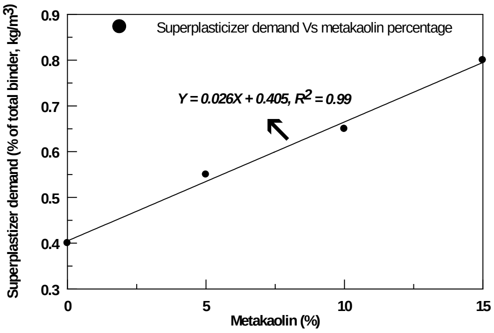

Figure 1 Superplasticizer demand Vs metakaolin percentage

For example, the 15, 10 and 5% metakaolin mixtures require 100, 62.5 and 37.5 % more superplasticiser dosage in comparison with that of the control mixture. This was mainly due to the higher specific surface area of metakaolin in comparison with the cement alone. Another reason, suggested by Nehdi *et al*. (1998), is that the Van der Waals and electrostatic attraction between cement and pozzolan grains are dominant due to the increase of wettable surface area. Consequently, at higher replacement levels, the tendency to flocculate is greater. In the presence of a surfactant such as superplasticiser on the surface of cement grains, particles repulse each other and remain separate thus giving the required workability (Nehdi *et al.,* 1998). The relationship between superplasticiser dosage based on the percentage of total dry weight of binder content is shown in Fig. 1. It can be seen from the figure, that the equation is linear in the form y = mx+c, where the coefficients of m and c are strictly governed by metakaolin content and w/b ratio. It should be noted that this equation only apply to cement content in the range 453 to 533 kg/m^3 for a slump within 100 ± 25 mm and chosen constituent materials and for w/b ratio of 0.30. As far as the workability is concerned, in fact all the concretes the control and the metakaolin mixtures have obtained their design slumps as shown in Table 2. According to these results, concretes made had a high slump values, highly cohesive and can be easily pumpable. No wide variations in the slump values for the mixtures containing increased amounts of metakaolin were observed.

3.2. Mechanical properties

The compressive strength results of samples presented in Table 3, shows that all the concretes made in this study are high strength, as even the seven day compressive strength varied between 78 and 80 MPa. The 28-day strength varied between 91 and about 99 MPa, and the 90-day strength varied between 101 and 106 MPa.

Table 3 Mechanical properties of the concretes investigated

Name	Compressive strength Age (days) (MPa)				Splitting ten. str. (MPa)	Elastic modulus (GPa)	f_{sp}/f_{ck}
	3 day	7 day	28 day	90 day	28 day	28 day	(%)
MK0	56.4	78.23	91.87	101.00	4.76	45.43	5.18
MK5	59.45	78.74	95.60	102.50	4.78	46.57	5.00
MK10	53.96	77.85	98.81	106.13	5.19	47.16	5.25
MK15	48.93	79.88	91.04	102.96	4.69	45.42	5.15

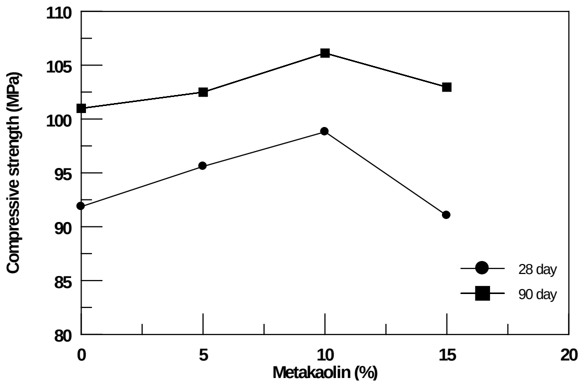

Figure 2 Variation of compressive strength with respect to metakaolin percentage

The 15% replacement metakaolin mixture had exhibited lower strengths comparatively than the other metakaolin percentages, but comparable strengths at all the ages to that of control concrete. All the concretes including the control had achieved their target strength of 90 MPa at 28 day and at 90 days all the concretes achieved strengths of more than 100 MPa. Fig. 2 presents the relation between compressive strength and metakaolin percentages at 28 and 90 days. It can be seen that the compressive strength was the highest for the MK10 mixtures achieving strengths of 102.5 and 106 MPa at 28 and 90 days. This indicates that the replacement level of 10% was the optimum level from the compressive strength point of view. This is slightly less than the replacement level of 15% reported in a previous study for the same water/binder ratio of 0.30 (Khatib, 2008). The reduction in compressive strength for MK15 compared to MK10 is explained as the result of a clinker dilution effect. The dilution effect is a consequence of replacing a part of cement by the same quantity of metakaolin. In MK concrete, the filler effect, pozzolanic reaction of MK with calcium hydroxide and compounding effect (synergetic effect of mineral admixture) react opposite of the dilution effects (Parande *et al.*, 2008; Ding and Zhang, 1999). For this reason, there is an optimum MK replacement for MK concrete.

The tensile strength results of MK concretes with varying amounts of metakaolin are shown in Table 3. The average value of the 28-day tensile strength for the concretes made was about 4.85 MPa, which corresponds to 5.15% of the compressive strength for the same concretes. Table 3 shows that the average ratio between the tensile strength (f_{sp}) to cube compressive strength (f_{ck}) of concrete at 28 days was lower than the range (of about 9-10%) for medium strength concrete reported previously (Neville, 1997; Rasiah, 1983; Haque and Kayali, 1998).

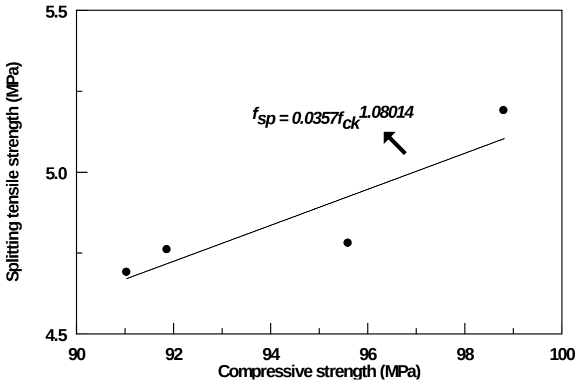

Figure 3 Variation of compressive strength with respect to splitting tensile strength

This indicates that the higher the compressive strength of concrete the lower the ratio, which is consistent with data published by other investigators (Rasiah, 1983; Haque and Kayali, 1998; Yogendran *et al.,* 1987). From the results it can be seen that similar to compressive strength the splitting tensile strength also exhibited the highest strength at MK 10 mixture. Fig. 3 presents the relation between compressive strength and splitting tensile strength of all the mixtures at 28 days. It can be observed that as the compressive strength increases, the tensile strength also increases. The relationship between compressive strength (f_{ck})and split tensile strength (f_{sp}) can be expressed as $f_{sp} = 0.0357(f_{ck})^{1.08014}$ $R^2 = 0.94$.

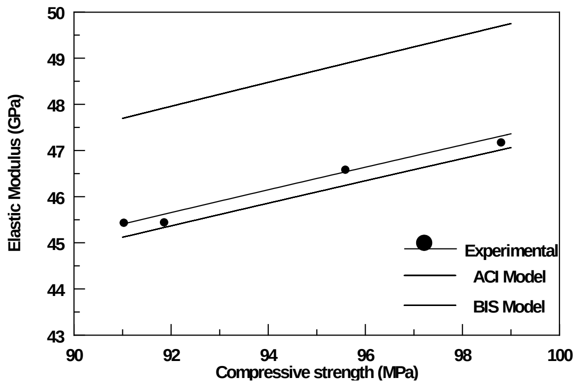

Fig. 4 Variation of compressive strength with respect to elastic modulus

The modulus of elasticity is related to the compressive strength of concrete. However, due to the presence of non-linear relationship between them (Neville, 1997; Mehta and Monteiro, 1999), the increase in the modulus of elasticity is not proportional to the increase in compressive strength as noted in Table 3. The modulus values presented in Table 3 indicate that the rate of increase in the modulus is lower than the rate of increase in the compressive strength. The elastic modulus (E) values with respect to the metakaolin contents are presented in Table 3. The trend is similar to that obtained for compressive strength; here the optimum metakaolin percentage that gives maximum E is at 10%. The strength (f_{ck}) is correlated with E as shown in Fig. 4. A direct linear, power and an exponential relationship were attempted and it was found that the power relationship in the form, $E = 4.76\sqrt{f_{ck}}$, R^2 =

0.98 fits the data points. In addition, the predicted values according to the American Concrete Institute (ACI) model ($E = 4.73\sqrt{f_{ck}}$) and BIS model ($E = 5\sqrt{f_{ck}}$) are also plotted in the same Fig. 4. The figure shows that the data points of MK mixtures lie slightly above the predicted modulus of ACI model but the BIS model overestimates the values obtained by actual testing.

3.3. Durability studies

A comprehensive summary of the results of the durability characteristics of all the concretes are presented in Table 4.

Table 4 Durability properties of the concretes investigated

Name	Permeability ($x10^{-12}$ m/sec)	Water penetration depth (mm)	Absorption (%)		Chloride permeability (Coulombs)
			30 min	Total	
MK0	0.918	17.08	1.198	2.81	1162
MK5	0.226	11.2	1.197	2.53	305
MK10	0.203	8.0	1.09	2.15	218
MK15	0.198	6.83	0.828	2.05	148

The volume of water penetrating with time for the different concretes was measured for evaluating the permeability values of these concretes. All the concretes including the control concrete were having permeability values less than $1x10^{-12}$ m/sec. As per CEB guide line (CEB-FIP, 1989), the control as well the metakaolin concretes were in the range of ‘‘Good’’ concrete quality. It can also be seen that the control concrete was showing a slightly higher permeability compared to the metakolin concretes though in the range of good quality, indicating that the permeability was decreasing with increasing percentage replacement of metakolin as shown in Table 4.

This is because the pore sizes are decreased with time either by refining the voids and/or by segmenting the interconnected voids with hydration products or metakaolin particles. In the present study, it was found that 15% replacement levels exhibited the lowest coefficient of permeability. This could be due to the fact that the pores were filled by hydration products, which would refine the overall size of the pores in the concrete system resulting in pore refinement, which leads to improved performance of the concrete (Zain *et al.*,2000). Also the adsorption of superplasticiser on cement particles may affect the diffusion of gel or the capillary pores in some way. In the current study, different dosages of superplasticiser for different levels of metakaolin replacements may influence the absortivity of concrete as suggested by Dhir and Yap (1984). In another study, according to Bai et al. (2002), the decrease in sorptivity (indirect measure of permeability of concrete) is due to the influence of particle packing on the initial capillary pore structure that develops, in that a wide distribution of binder particle sizes exists in blend mixtures with metakaolin and fly ash content. This will result in a denser packing than mixtures with cement only, thus reducing the sorptivity. In their study, it was also reported that the relative sorptivity values clearly reflected the strength values whereby the lowest sorptivity values had the highest strength except when the replacement level was 40% (Bai *et al.*, 2002). This contradicts the results obtained in the present study in which the lowest value of permeability of MK15 did not exhibit the highest compressive strength for the w/b ratio studied. As stated before, the dilution effect, which is caused by the high cement replacement level, will limit strength gain. The water penetration depths results also followed a similar trend as shown in Table 4.

The durability of concrete primarily depends upon its permeability, which defines the resistance to the ingress of aggressive ions. The absorption characteristics indirectly represent the porosity, through an understanding of the permeable pore volume and its connectivity. A limit on the initial (30 min) absorption for assessing the concrete quality was defined by CEB earlier (CEB-FIP, 1989). The absorption in 30 min (initial surface absorption) as well as the absorption after 72 h (final absorption) for all the concretes is shown in Table 4. From these results, it can be seen that the initial surface absorption of control as well as the metakaolin concretes show values much less than 3%, the limit specified for ‘‘good’’ concrete by CEB (CEB-FIP, 1989). The total absorption at the end of 72 h for these concretes also followed a similar trend, which was also similar to that of the permeability results. From the results it can be seen that the water absorption of the metakaolin mixtures was lower than that of the control mixture and the difference between control and metakaolin mixtures became greater for mixtures with higher replacement levels. Similar to the permeability results the water absorption was reduced as the metakaolin replacement levels increased. It is apparent that the pozzolanic reaction and filler effect contributed to reducing the porosity of the concrete. Sabir et al. (2001) pointed out that there is strong evidence that metakaolin greatly influences the pore structure in pastes and mortars and produces substantial pore refinement. This leads to significant modifications to the water transport properties. Guneyisi et al. (2008) explained that the beneficial effect of metakaolin in reducing the water absorption was noticeable due to the filling effect of ultrafine metakaolin as well as its pozzolanic reaction.

Accelerated chloride permeability test was conducted on all the concretes and the total charge passing in 6 h as a measure of the chloride permeability was presented in Table 4. It can be seen that, all the metakaolin concretes show very low chloride permeabilities in the range of 148 – 305 C, primarily due the metakaolin in these mixtures. In contrast, the control concrete (MK0) shows a low chloride permeability of about 1162 C, indicating clearly that the mixtures containing metakaolin were behaving significantly better. It was also observed earlier that the resistance to chloride ion penetration reduced significantly as the proportion of metakaolin increased (Kim *et al.,* 2007; Boddy *et al.,* 2001). Also Gruber et al. (2001) indicated that high reactivity metakaolin substantially reduced chloride ion penetration in concrete and such reductions can be expected to have a substantial impact on the service life of reinforced concrete in chloride environments. Abbas et al. (2010) explained that the addition of metakaolin increased the system’s capacity to bind chloride ions, thereby lowering the free ion content. This reduced steel reinforcement corrosion, as free chloride ions are generally acknowledged to be the only species in the pore water that effects reinforcement corrosion. The presence of C_3A in hydrated cement reacts with chloride ions and forms inert products like Friedels salt ($3CaO\ Al_2O_3\ CaCl_2\ .10H_2O$). If the part of cement is replaced with pozzolanic material like metakaolin, the alumina content in that further favors the binding of chloride ion (Schiessl, 1988).

4. CONCLUSION

(a) The results shows that by utilizing local metakaolin and cement designed for a low water/binder ratio of 0.3, high strength and high performance concretes can be developed and compressive strengths of more than 100 MPa can be realized.

(b) The optimum replacement level of OPC by metakaolin was 10%, which gave the highest compressive strength in comparison to that of other replacement levels; this was due to the dilution effect of partial cement replacement. These concretes also exhibited a 28-day splitting tensile strength of the order of 5.15% of their compressive strength and showed relatively high values of modulus of elasticity. Splitting tensile strengths and elastic modulus results have also followed the same trend to that of compressive strength results showing the highest values at 10% replacement.

(c) For the durability properties, local metakaolin was found to reduce water permeability, absorption, and chloride permeability as the replacement percentage increases. This may be due to the filler effect of metakaolin particles which has greatly reduced the permeability/porosity of the concrete.

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