

EXPERIMENTAL INVESTIGATION OF CONCRETE USING METAKAOLIN INCORPORATION

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Abstract - The infrastructure needs of our country is increasing day by day and with concrete is a main constituent of construction material in a significant portion of this infrastructural system. It is necessary to enhance its characteristics by means of strength and durability. An experimental study proposing changes to the conventional concrete to gain high strength and high performance by partial replacement of Metakaolin with OPC 53.

Metakaolin (MK) or calcined kaolin, other type of Pozzolanic, produced by calcination has the capability as an alternative material. In India MK 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. At present the market price of MK in the country is about 3–4 times that of cement. Therefore the use of metakaolin proves economical. Previously, researchers have shown a lot of interest in MK as it has been found to possess both Pozzolanic and micro filler characteristics. It has also been used successfully for the development of high strength self-compacting concrete using mathematical modelling. However, limited test data are available regarding the performance of the commercially available MK and Indian cements in the case of high strength concrete in the country.

The objective of this study was to investigate the effect of using local calcined kaolin or MK obtained commercially as pozzolan on the development of high strength and characteristics of concrete designed for a very low w/b ratio of 0.3. In addition, the optimum replacements with respect to strength were determined by varying the amount of MK as partial cement replacement by MK0% MK 5%, MK10% and MK15%.

Key Words: Metakaolin, Pozzolanic, High strength, OPC-53, Economical, Kaolin, Calcination

1. INTRODUCTION

The quest for the development of high strength and high performance concretes has increased considerably in recent

times because of the demands from the construction industry. 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, high-performance concretes can be produced at lower w/b ratios by incorporating these supplementary materials. Fly ash addition proves most economical among these choices, even though addition of fly ash may lead to slower concrete hardening. However, when high strength is desired, use of silica fume is more useful (Basu 2003). When designed at very low water/binder ratio, the presence of silica fume explains the mechanical performance of high strength concrete. Silica fume provides a very good particle packing and, because of its strong Pozzolanic property increases the resistance of the concrete to aggressive environments also (Abdul and Wong 2005). Silica fume, though initially considered as an industrial waste, has now become a world class product for which there is a constant demand in the construction industry. However, this product is rather expensive. In India, most of the good quality silica fume is imported and the cost is 9–10 times the cost of OPC. Metakaolin (MK) or calcined kaolin, other type of Pozzolanic, produced by calcination has the capability to replace silica fume as an alternative material. In India MK 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). At present the market price of MK in the country is about 3–4 times that of cement. Therefore the use of metakaolin proves economical over that of silica fume. Previously, researchers have shown a lot of interest in MK as it has been found to possess both Pozzolanic and micro filler characteristics (Poon et al. 2001; Wild and Khatib 1997; Wild et al. 1996). It has also been used successfully for the development of high strength self-compacting concrete using mathematical modelling (Dvorkin et al. 2012). However, limited test data are available

regarding the performance of the commercially available MK and Indian cements in the case of high strength concrete in the country (Basu 2003; Basu et al. 2000, Pal et al. 2001, Patil and Kumbhar 2012). The objective of this study was to investigate the effect of using local calcined kaolin or MK obtained commercially as pozzolan on the development of high strength and permeability/durability characteristics of concrete designed for a very low w/b ratio of 0.3. In addition, the optimum replacements with respect to strength and durability were determined by varying the amount of MK as partial cement replacement.

1.1 MATERIAL AND PROPERTIES

- Material Specification

The following materials were employed:

1.1.1 Cement:

OPC of 53 Grade confirming to IS: 12269-1987 was used in the investigation. The specific gravity of cement was 3.15.

Table 1 Physical properties of cement

No.	Properties	IS; 269-1976
1	Normal Consistency	31.25 %
2	Initial setting time	Minimum of 72 min
3	Final setting time	Maximum of 180 min
4	Specific gravity	3.15



Figure 1 Cement Bag 53 Grade

Table 2 Properties of Cement

Chemical composition	Cement %
Silica (SiO_2)	34
Alumina (Al_2O_3)	5.5
Calcium Oxide (CaO)	63
Ferric Oxide (Fe_2O_3)	4.4
Magnesium Oxide (MgO)	1.26
Potassium Oxide (K_2O)	0.48
Sulphuric anhydride (SO_4)	1.92
Blaine (m^2/Kg)	360

Loss in Ignition (LOI)	1.3
Specific gravity	3.15
Physical form	Fine powder
Color	Grey

1.1.2 Coarse Aggregate:

Crushed stone metal with a maximum size of 20 mm from a local source having the specific gravity of 2.7 conforming IS 383-1970 was used.

Table 3 Properties of coarse aggregate

Basic Property	Condition	Sylhet sand
Unit weight	Oven dry rodded unit weight	1584.9 kg/m ³
Specific Gravity	Oven-dry bulk Sp. Gr.	2.885
Absorption capacity		0.504%
Fineness Modulus		7.386



Figure 2 Coarse aggregate

1.1.3 Fine Aggregate:

Locally available river sand passing through 4.75mm IS sieve conforming to grading zone II of IS383-1970 was used. The specific gravity of fine aggregate was 2.54.

Table 4 Fine aggregate properties according to (ASTM C29/29M, 1991)-97 and (ASTM C 136, 2001)

Basic Property	Condition	Sylhet sand
Unit weight	Oven dry rodded unit weight	1162 kg/m ³
Specific Gravity	Oven-dry bulk Sp. Gr.	2.75
Absorption capacity		1.538 %
Fineness Modulus		3.338



Figure 3 Fine Aggregate

1.1.4 Metakaolin:

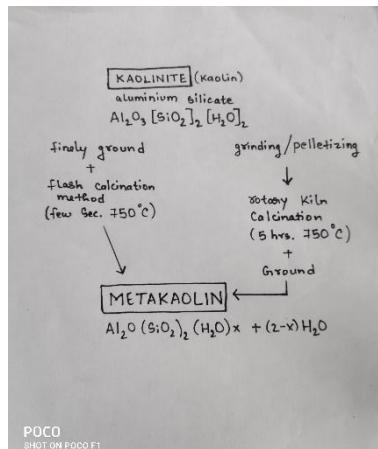


Figure 4 Formation of Metakaolin



Figure 5 Metakaolin

Metakaolin is not a by-product. It is obtained by the calcinations of pure or refined Kaolinite clay at a temperature between 600°C and 800°C , followed by grinding to achieve a finesse of $700\text{-}900 \text{ m}^2/\text{kg}$. It is a high quality Pozzolanic material, which is blended with cement in order to improve the durability of concrete. When used in concrete it will fill the void space between cement particles resulting in a more impermeable concrete. Metakaolin, is a

relatively new material in the concrete industry, is effective in increasing strength, reducing sulphate attack and improving air-void network. Pozzolanic reactions change the microstructure of concrete and chemistry of hydration products by consuming the released calcium hydroxide (CH) and production of additional calcium silicate hydrate (C-S-H), resulting in an increased strength and reduced porosity and therefore improved durability. The formation and properties of Meta kaolin are shown in below. The specimen kept immerse in water for 7 and 28 days. The chemical content of metakaolin presented in Table 5.

Table 5 Properties of Metakaolin

Chemical composition	Metakaolin %
Silica (SiO_2)	54.3
Alumina (Al_2O_3)	38.3
Calcium Oxide (CaO)	0.39
Ferric Oxide (Fe_2O_3)	4.28
Magnesium Oxide (MgO)	0.08
Potassium Oxide (K_2O)	0.50
Sulphuric anhydride (SO_4)	0.22
Blaine (m^2/Kg)	15000
Loss in Ignition (LOI)	0.68
Specific gravity	2.5
Physical form	Powder
Color	Off-White

1.1.5 Super Plasticizer:

Poly-carboxylate Ether Super plasticizer obtained from CHEMCON Tecsys was used. It conforms to IS 9103 – 1999 and its specific gravity of 1.2.

1.1.6 Water:

Fresh portable water is free from concentration of acid and organic substance is used for mixing the concrete and curing.

2.0 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 were 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 MK. In mixtures MK5, MK10 and MK15, cement content was partially replaced with 5, 10, and 15 % MK (by mass) respectively. The binder consists of cement and MK. Trial mixtures were

Table 6 Details of Mix proportions (Kg/m³)

Constituent	MK 0	MK 5	MK 10	MK 15
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

Conducted to determine the optimum dosage of SP for each of the mixtures in order to achieve the target slump of 100 ± 25 mm. The dosage of SP for each mixture was carefully selected as over dosage may induce bleeding and strength retardation. Table: 6 presents the mixture proportions for all of the mixture series with different dosage of SP for a target slump of 100 ± 25 mm. With respect to the aggregate grading, in the present investigation a combined aggregate grading as recommended by the DIN 1045 standards was utilized. The aggregates 20, 12.5 and 4.75 mm were combined in such a way, so that it meets nearly the combined grading specification of DIN 'A' curve. The percentage fractions of aggregates used for 20, 12.5 and 4.75 mm are 31, 32 and 37 of the total aggregate content. Blending aggregates in this fashion and designing concretes at very low water binder ratios will yield high strength concretes, because of the good packing density (Dinakar 2012).

3.0 Mixing and Casting Details:

All the materials were mixed using a pan mixer with a maximum capacity of 80 liters. The materials were fed into the mixer in the order of coarse aggregate, cement, MK and sand. The materials were mixed dry for 1.5 min. Subsequently three-quarters of the water was added, followed by the SP and the remaining water while mixing continued for a further 5 min in order to obtain a homogenous mixture. Upon discharging from the mixer, the slump test was conducted on the fresh properties for each mixture. The fresh concrete was placed into the steel cube molds and compacted on a vibrating table. Finally, surface finishing was done carefully to obtain a uniform smooth surface.

**Figure 6** Mixing of concrete

4.0 Specimens and Curing

The following specimens were cast from each mixture:

- $100 \times 100 \times 100$ mm cubes were cast in iron molds.

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 h. The specimens were demolded after 24 h of casting and were then cured in water at approximately 27 °C until the testing day.





Figure 7 Casted Cubes

5.0 Test Results

5.1 Fresh Properties

5.1.1 Plastic Density

The results of the plastic densities with respect to the corresponding MK percentages are given in Table 6. From this it can be seen that the plastic densities varied between 2,421 and 2,520 kg/m³. The slight reduction in the densities of MK concretes was due to the lower specific gravity of MK compared to cement alone.

5.1.2 SP Demand

In this study, different SP dosages were added to the different mixtures in order to obtain the consistency or workability in terms of target slump of 100 ± 25 mm. It can be seen from Table 6, the SP demand increased with increase in the metakaolin replacements. For example, the 15%, 10% and 5% MK mixtures require 100, 62.5 and 37.5 % more SP dosage in comparison with that of the control mixture. This was mainly because of the higher specific surface area of MK in comparison with the cement alone. Another reason, cited by Nehdi et al. (1998), is that the Van der Waals, which are the main causes for cement particles agglomeration and electrostatic attraction between cement and pozzolan particles becomes dominant due to the increase in the wet able surface area. Therefore, as the percentage replacement increases flocculation becomes more predominant. In the presence of a dispersant such as SP on the surface of cement grains, particles repulse each other because of the dispersion of agglomerated cement particles and remain separate thus delivering the required workability (Nehdi et al. 1998). The

relationship between SP dosage with respect to the percentage of total dry weight of binder content is shown in Fig. 5. 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 MK content and w/b ratio. It should be noted that this equation only apply to cement content in the range 453–533 kg/m³ for a slump within 100 ± 25 mm and chosen constituent materials and for w/b ratio of 0.30. The proposed best fit linear equation is as follows:

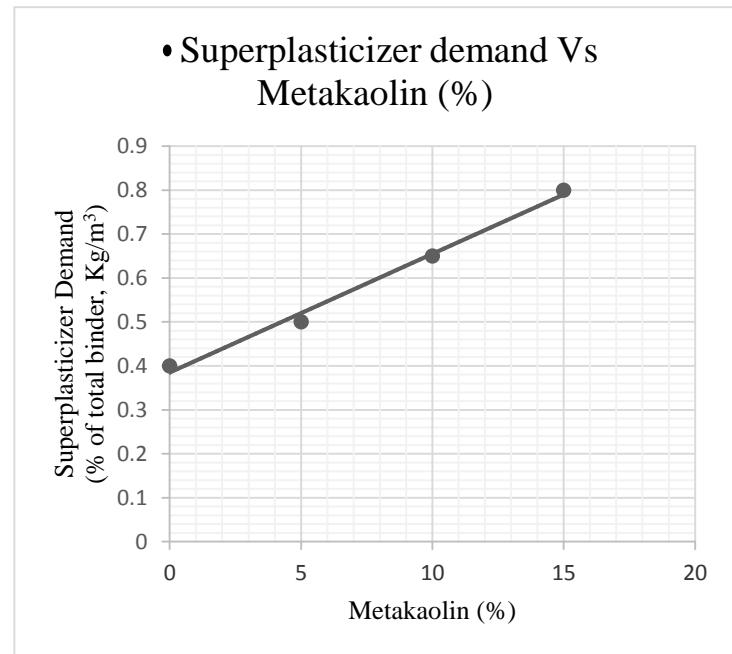


Figure 8 Superplasticizer demand Vs Metakaolin percentage

$$SP (\%) = 0.405 + 0.026 (MK \%), R^2 = 0.99 \dots \dots \dots (1)$$

As far as the workability is concerned, in fact all the concretes the control and the MK mixtures have obtained their design slumps as shown in Table 6. According to these results, concretes obtained had high slump values, highly cohesive and can be easily pump-able. No wide variations in the slump values for the mixtures containing increased amounts of MK were observed.

Table 7 Mechanical properties of concrete

Name	Compressive Strength age (days) MPa				Splitting tensile strength (MPa)	Elastic modulus (GPa)	Flexural Strength (MPa)
	3 days	7 days	28 days	90 days			
MK 0	56.4	78.2	91.8	101.0	4.76	45.43	6.3
MK 5	59.4	78.7	95.6	102.5	4.78	46.57	6.6
MK 10	53.9	77.8	98.8	106.1	5.19	47.16	7.0
MK 15	48.9	79.8	91.0	102.9	4.69	45.42	6.8

5.2 Mechanical Properties

5.2.1 Slump Cone Test:

Sampling of Materials for Slump Test:

A concrete mix (M30) by weight with suitable water/cement ratio is prepared in the laboratory similar to that explained and required for casting 6 cubes after conducting Slump test.

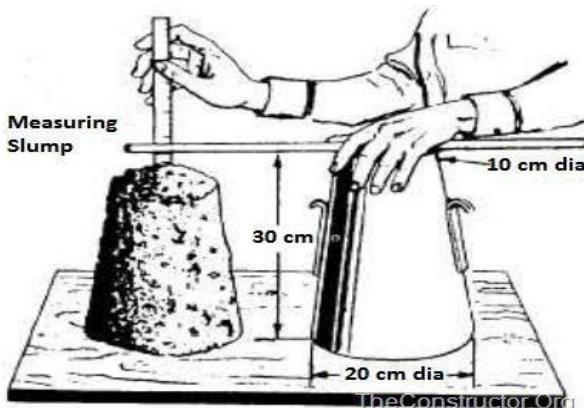


Figure 9 Measuring Slump of Concrete

Procedure for Concrete Slump Cone Test:

Clean the internal surface of the mould and apply oil.

Place the mould on a smooth horizontal non-porous base plate.

Fill the mould with the prepared concrete mix in 4 approximately equal layers.

Tamp each layer with 25 strokes of the rounded end of the tamping rod in a uniform manner over the cross section of the mould. For the subsequent layers, the tamping should penetrate into the underlying layer.

Remove the excess concrete and level the surface with a trowel.

Clean away the mortar or water leaked out between the mould and the base plate.

Raise the mould from the concrete immediately and slowly in vertical direction.

Measure the slump as the difference between the height of the mould and that of height point of the specimen being tested.

Slump Value Observation:

The slump (Vertical settlement) measured shall be recorded in terms of millimetres of subsidence of the specimen during the test.

Results of Slump Test on Concrete

Slump for the given sample is mentioned below.

When the slump test is carried out, following are the shape of the concrete slump that can be observed:

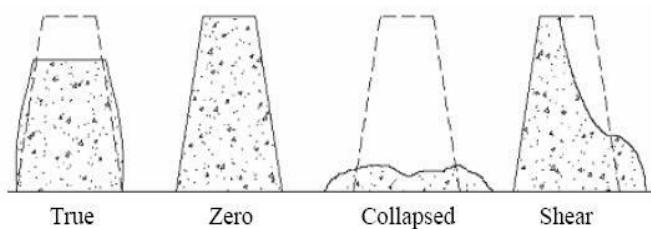


Figure 10 Types of Concrete Slump Test Results

- **True Slump** – True slump is the only slump that can be measured in the test. The measurement is taken between the top of the cone and the top of the concrete after the cone has been removed as shown in figure-1.
- **Zero Slump** – Zero slump is the indication of very low water-cement ratio, which results in dry mixes. These type of concrete is generally used for road construction.
- **Collapsed Slump** – This is an indication that the water-cement ratio is too high, i.e. concrete mix is too wet or it is a high workability mix, for which a slump test is not appropriate.
- **Shear Slump** – The shear slump indicates that the result is incomplete, and concrete to be retested.

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 3,000 kN 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.

5.2.2 Compressive Strength :

The compressive strength results of samples presented in Table 7, 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. The 15 % replacement MK mixture had exhibited lower strengths comparatively than the other MK 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. Figure 11 presents the relation between compressive strength and MK 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 clearly shows that the replacement level of 10 % was the optimum as far as the compressive strength is concerned. 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 result of replacing a part of cement by the equivalent quantity of MK. 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 et al. 1999). For this very reason, there was an optimum MK replacement for MK concrete. With time, the compressive strength differences between the MK mixtures and OPC concrete becomes smaller. This might be due to the fact that all cementitious materials reactions were close to completion, or had stopped; mainly because the reactions between MK and OPC mixtures were slowed down with time (Wild and Khatib 1997).

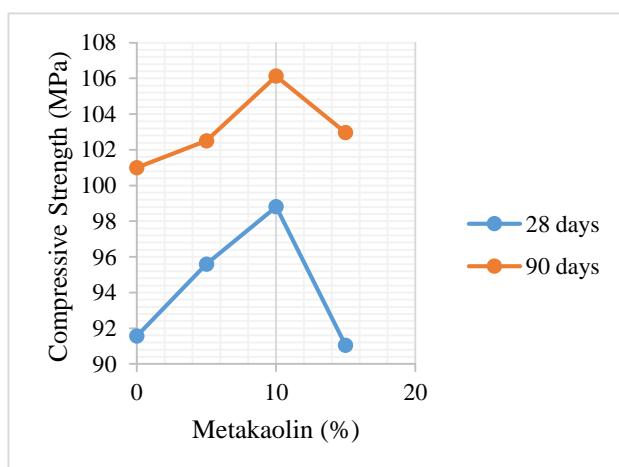


Figure 11 Variation of compressive strength with respect to metakaolin percentage

5.2.3 Splitting Tensile Strength :

The tensile strength results of MK concretes with varying amounts of MK are shown in Table 7. 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 7 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 earlier (Neville 1997; Rasiah 1983; Haque and Kayali 1998). This indicates that as the compressive strength increases lower would be the ratio, which is consistent with the results published by other investigators earlier (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. Figure 12 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 below (from Fig. 12).

$$f_{sp} = 0.0357(f_{ck})^{1.08014} \quad R^2 = 0.94 \dots\dots (2)$$

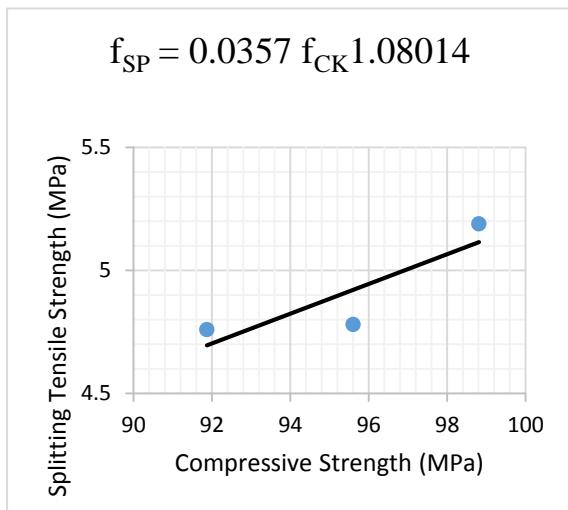


Figure 12 Variation of compressive strength with respect to splitting tensile strength

5.2.4 Elastic Modulus

The modulus of elasticity is mainly related to the compressive strength of concrete. However, due to the existence of non-linear relationship between them (Neville 1997; Mehta and Monteiro 1999), the increase in the modulus of elasticity is not in proportion to the increase in compressive strength as noted in Table 7. The modulus values presented in Table 7 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 MK contents are presented in Table 7. The trend is similar to that obtained for compressive strength; here the optimum MK percentage that gives maximum E is at 10 %. The strength (f_{ck}) is correlated with E as shown in Fig. 13. A direct linear, power and an exponential relationship were attempted and it was found that the power relationship in the form given below fitted the data best

$$E = 4.76 \sqrt{f_{ck}} R^2 = 0.98 \dots\dots\dots (3)$$

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. 13. 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

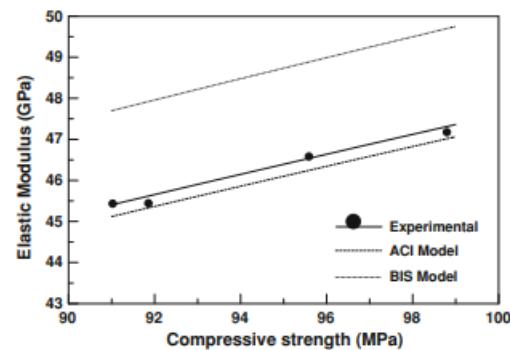


Figure 13 Variation of compressive strength with respect to elastic modulus

5.2.5 Flexural Strength

The Flexural strength compared to control specimen with various percentages of Metakaolin. When compared to control specimen the Flexural strength for MK5% increases 4.76%. The Flexural strength for MK 10%, 15% increases respectively and decreases with higher concentration of Metakaolin. The Flexural strength test values are presents in Table 7.



Figure 14 Flexural Strength

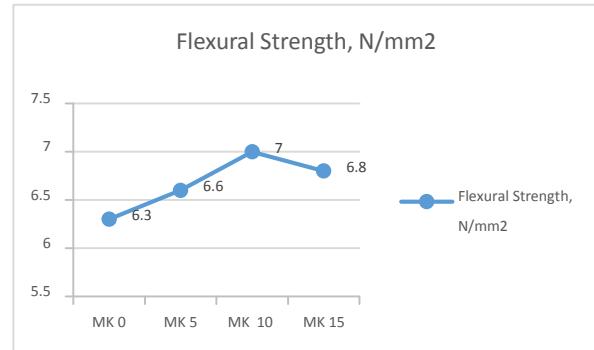


Figure 15 Variation of Flexural Strength

6. CONCLUSIONS

The following conclusion can be drawn from the current study.

(a) Using MK as a partial replacement for cement decreased the plastic density of the mixtures.
(b) The results shows that by utilizing local MK 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.
(c) The optimum replacement level of OPC by MK 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.

ACKNOWLEDGEMENT

We are glad to express our gratitude towards civil engineering department, Bhagwan Mahavir College of engineering and technology, Surat for gave us an opportunity to develop the project. Then our humble thanks to all professors and all staff members of our college for the co-operation and keep interest extended by them throughout our B.E course.

We are thankful to our project guide, Dr. Kamal Padhiar (Head of Civil Department), who inspires us to undertake this Project. His guidance and continuous interest kept us going in spite of all the difficulties throughout the project.

We deeply owe our gratitude to our faculties for their kind help, support and encouragement throughout this project.

We are thankful to all the staff member of civil department for their kind support at various stages of the work.

We are thankful to each and every group member who participated in this work to complete the project.

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