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Experimental Study On Effect Of Metakaolin On Performance Of Concrete

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ABSTRACT

In the present study, an attempt has been made to assess the suitability of kaolinite clay collected from hyderabad, India, as pozzolana in making concrete. The characteristics of OPC, FA, CA, and water were studied to assess their suitability to use in concrete. Mineralogical and physical properties natural kaolin clay collected from Cretaceous (CCK & CCT) and Tertiary system (TCN) were analyzed to find out their acceptability as pozzolona to use in concrete. Later, natural kaolin was calcinated at 400ºC, 600ºC and 800ºC temperature over a period of 4 and 6 hour duration to find out optimum calcination temperature and calcination duration to convert natural kaolin into meta-kaolin. Subsequently, an effort has been made to find out the influence of meta-kaolin on characteristics of MK-OPC blended cement such as consistency, setting time and compressive strength. Further, MK concrete mixes were designed using guidelines prescribed by IS: 10262-2009. Subsequently effect of MK on workability of fresh concrete and also properties of hardened concrete such as compressive, flexural and split tensile strength of concrete were analyzed. Later, CCK15MK concrete mixes were made assess its influence on structural performance of reinforced concrete and also durability in terms of acid resistance, sulfate resistance, chloride resistance, etc. Finally microstructure analysis using x-ray diffraction, Scanning Electron Microscopy with Energy-dispersive X-ray spectroscopy was carried out on CCK15, CCT15, and TCN15 mixes to find the influence of meta-kaolin on performance of concrete.

Key words: kaolin clay; metakaolin; Tertiary system.

1.0 INTRODUCTION:

In India, about 502 million tons of cement per year are being produced to match with demand of construction industry. The demand for cement is likely to increase exponentially in future to meet needs of infrastructure development. However, environmental concerns due to carbon dioxide emitting from cement industry brought tremendous stress to minimise cement utilization by using alternative binding material or supplementary cementitious materials to produce concrete. Alternative binding materials may be industrial refuse or by-products and also materials requiring less energy for producing binding material and natural material. Efficient usage of industrial refuse materials such as fly ash , rice husk ash (RHA), ground granulated blast-furnace slag (GGBFS), and silica fume (SF) as cementitious material certainly reduce the problem of disposing industrial waste and preserve the ecosystem. Further, serious deterioration of structures made with OPC concrete is another concern for a rise in pressure to search for alternative binding material or supplementary cementitious material to enhance concrete durability.

In order to address aforesaid concerns, attempts been have been made to use industrial by-products such as Fly Ash, RHA, GGBFS, and SF as mineral admixtures (Pozzolanic material) to enhance quality of concrete. The economic advantage, coupled with improved performance made construction industry to use binary as well as ternary blended cement to produce concrete (Mehta,1987). Fly ash is a major industrial byproduct polluting air, water and soil . Presently, around 184 MT of Fly Ash is being generated in Thermal power units in India. Out of which, almost 55% being utilized as pozzolanic material in making concrete, bricks, pavements, landfilling etc. It has been proved that fly ash is very effective pozzolanic material to partially replace OPC up to 30% to produce concrete with improved performance. Also, around 12MT of slag is being generated from Steel industry in India and it is being used as recycled material in road construction, producing PPC , soil stabilization etc. These pozzolanic materials are improving durability as well as strength of concrete.

Kaolin may not be possessing a pozzolanic character in its natural state due to its crystalline form. However, kaolin gains amorphous form during thermal treatment at temperature ranging from 700 to 8000C and becomes meta-kaolin having excellent pozzolanic properties. In fact, blended calcinated clay and lime was used in making binding material for construction for thousands of years (Stanley 1979 ; Mehta 1987). Structures such as water tank, aqueduct, bridge etc of 4000 years old were constructed using metakaolin-lime blended mortars (Malinowski 1979 ; Mielenz 1983). However, its suitability depends on physical properties, chemical composition, calcination temperature and duration of the calcination process. Hence, an attempt has been made in this work to study characteristics of Cretaceous and Tertiary meta-kaolin available in Tamil Nadu, India and their influence on the performance of concrete.

The utilization of clay in the form of meta-kaolin as a pozzolanic material for mortar and concrete has received considerable attention in recent years. Kaolin is one of the most widely used industrial minerals. The total output of kaolin clay exceeds 25 million tonnes (Koumbou et al., 2009). Natural pozzolans in the form of calcined earth blended with lime have been used to produce cementitious materials during the past over thousands of years (Stanley, 1979 Mehta, 1987). Structures such as water tanks, aqueducts, walls, and bridges of 4000 years old have been constructed using thermally activated clay and lime mortars (Malinowski, 1979 ; Mielenz, 1983).

Kaolin undergoes the de-hydroxylation process during calcination at 550°C to 700°C and converts to meta-kaolin (Mackenzie, 1970; Babushkin et al., 1985 and Sperinck et al., 2011). He C et al., (1995) reported that 648°C to 982°C as optimum temperature for calcination. Subsequently, Al-Rawa et al., (1998) suggested calcination temperature in the range of 730°C to 830°C. Later, Chakchouk et al., (2006) recommended 700°C to calcinate kaolin to use as pozzolanic material. However, Souri et al., (2015) proposed 700°C to 800°C temperature to calcinate kaolin to convert to meta- kaolin. Also, IS 1344-1981(Reaffirmed1999) has specified 700°C to 800°C.

The present work entitled “Experimental Study on Effect of Cretaceous and Tertiary Metakaolin on Performance of Concrete” was carried out, to study physical and chemical characteristics of Cretaceous as well as Tertiary Kaolin clay and to assess their suitability to use as pozzolanic material. Also, to find out optimum calcination temperature, duration of calcination to produce meta-kaolin.

2.0 MATERIALS AND METHODS:

53 grade OPC, river sand as fine aggregate, crushed angular stone of 12mm to 20mm as coarse aggregate, super plasticizer and kaolin clay from Cretaceous and Tertiary system were used to produce concrete.
OPC of 53 Grade (Ultra tech) was used as binder for making concrete. Experiments are carried out to find out physical properties of cement using the methodology prescribed by IS: 4031-1996 (Reaffirmed 2011). River sand available locally was collected and used as fine aggregate to produce concrete. Physical characteristics of fine aggregate were measured using methodology prescribed by IS: 2386-1963 (Reaffirmed 2011). 20mm and 12mm size crushed stone aggregate available locally was used as coarse aggregate. The physical and mechanical characteristics of these coarse aggregate were measured as per methodology recommended by IS: 2386-1963. Treated potable ground water conforming to requirements prescribed by IS: 456-2000 was used for preparing concrete as well as curing.

The super plasticizing admixture (Conplast SP430 from Fosroc) was used to enhance workability of concrete. The characteristics of the admixture are in conformity with IS 9103-1999.The Specific gravity of the admixture is 1.18.

An effort has been made in this work to study characteristics of natural kaolin and its suitability as a mineral admixture to partially replace cement in concrete. The vast Kaolin clay resources are available in Indian sub-continent and it is being used for wide variety of applications. Cretaceous clay from Karai (CCK), Therani (CCT) and Tertiary clay were collected and used as pozzolanic material in the present work. Physical characteristics of kaolin such as particle size, lime reactivity and compressive strength etc were measured to assess suitability of pozzolana for making concrete. XRD technique was used to measure particle size, mineral constituents present and also crystalinity of kaolin. Natural kaolin sample collected from afore said locations were tested for their chemical composition. The chemical analysis was carried for determining various characteristics of kaolin such as Silica+ Alumina+ Iron Oxide, loss on Ignition, Silica, Magnesia, Total sulfate, Total loss on ignition and Alkalis.

2.1 MIX PROPORTIONING OF CONCRETE

Concrete mix was designed using guidelines prescribed by IS: 10262-2009. The concrete mixes were made by replacing 10%, 15%, 20%, and 25% of cement (by weight) by calcinated kaolin. 417 kg/m³ of cement with w/b ratio of 0.45 was used to produce concrete mixes. Maximum 20mm size coarse aggregate is used in present work. Also, chemical admixture (Conplast-430) was used to maintain workability of concrete. The mixes made with meta-kaolin from Karai and Therani were designated as CCK and CCT respectively. Also, mix made with meta-kaolin from Tertiary kaolin- Neyveli was designated as TCN. The number in designation indicates percentage of MK in concrete mix. Further, control mix was made with only OPC as a binder. Details of mix design for M30 grade of concrete are furnished in Table 1.

Table 1: Details of Concrete Mix Proportion of Various mixes (M 30 Grade)

Mix Code	**OPC (kg/m^3)**	**kaolin(Karai) (kg/m^3)**	**FA (kg/m^3)**	**CA (kg/m^3)**	**water (L)**	**admixture**
CC	416	0	751	1169	187	2.91
CCK 10	375	42	748	1164	187	2.91
CCK 15	354	62	747	1163	187	2.91
CCK 20	333	83	745	1160	187	2.91
CCK 25	312	104	744	1158	187	2.91

Concrete cubes having 100 mm dimension were cast to measure compressive strength as well as durability, whereas, prisms with 500x100x100 mm dimension were cast for flexural strength. Further, concrete cylinders were cast with 100mm diameter and 200 mm length to study tensile strength of concrete. Also, a beam having 1700x 150x100 mm dimension was cast to study flexural behavior of RCC. After 24 hours, specimens were demoulded and cured in water before testing. Details of specimen casted are presented in Table 2.

Table 2: Details of Type, Dimensions and Number of Casted Specimens

S. No	Name of test	Type of specimen	Specimen dimensions (mm)	Total specimens
1	Compressive strength	Cube	100 X 100	15
2	Flexural Strength	Prism	500x100x100	15
3	Split Tensile strength	Cylinders	100 X 300	15
5	Ultra-Sonic Pulse Velocity	Prism	500x100x100	10
	Total number of specimens			55

EXPERIMENTAL RESULTS AND DISCUSSION

Characteristics of different types of meta- kaolin and MK-OPC blended binder are presented in this chapter. Also, workability, compressive, flexural and split tensile strength of CCK, CCT and TCN concrete mixes were included. Further, flexural behaviour of CCK-RCC beam evaluated experimentally as well as analytically is also given in this chapter. Also, microstructure analysis and durability of CCK concrete in terms of water absorption, RCPT, acid resistance, chloride resistance and sulfate resistance were furnished in this chapter.

The XRD analysis was conducted on natural kaolin clay. The XRD of CCK kaolin was analysed for various mineral constituents and results are furnished. Result shows that kaolinite is a dominant mineral present in CCK sample. Also, impurities such as Montmorillonite and Quartz minerals are present in CCK clay of Cretaceous system. Peaks appear at °2 theta position of 13 represent Montmorillonite. The kaolinite is represented by peaks having reflection at 2 °theta positions of 12 and 27 with a scale factor of 0.553. Also, Peaks appear at °2 theta position of 21 and 25 represent Quartz mineral (Fig 1).

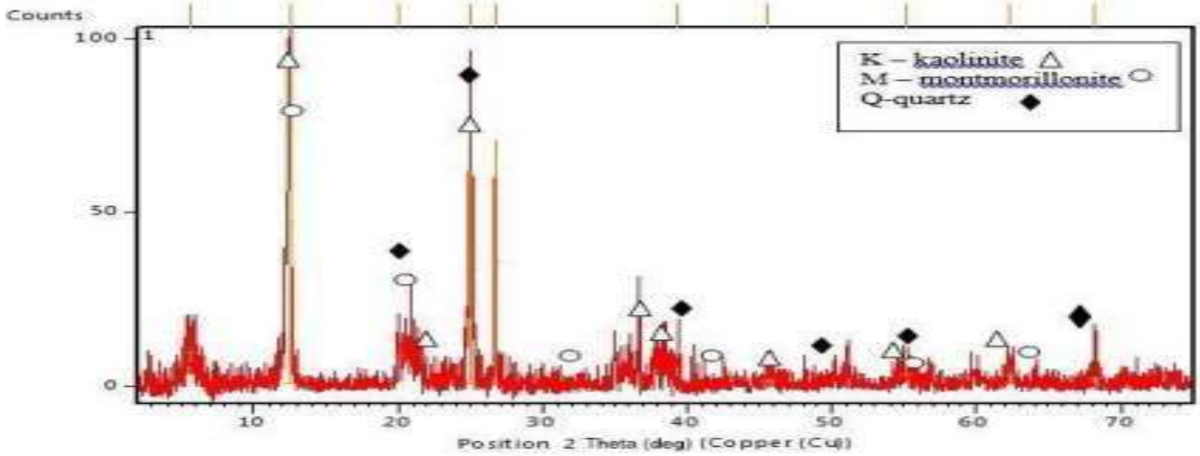

Figure 4.1: X-Ray Diffractogram of Natural CCK kaolin clay

Also, XRD analysis was carried out on CCT kaolin clay and results are furnished. The results show that clay is having minerals such as kaolinite, clinochlore- ferrous and quartz minerals (Fig 4.2). The peaks appear at °2 theta position of 12 and 26 represent kaolinite with a scale factor of 0.275. Further, peaks at 18 and 27 represents quartz and 18 clinochlore ferrous.

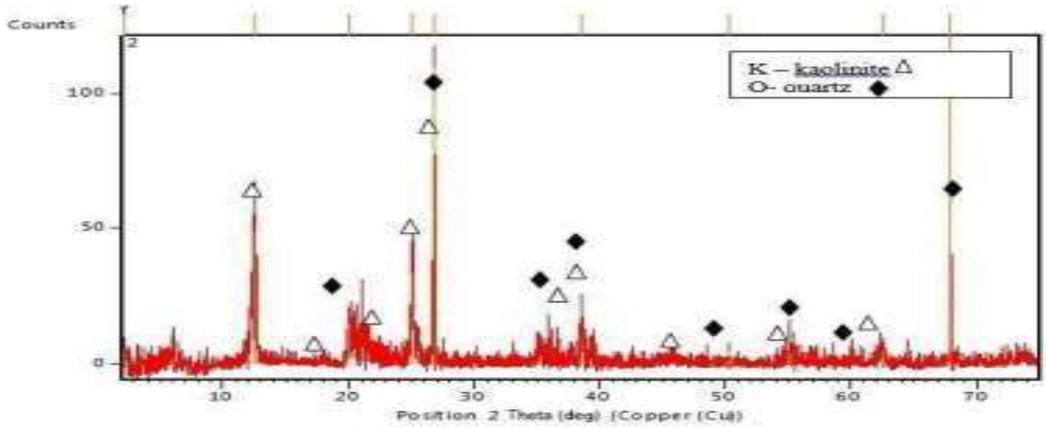

Figure 4.2: X-Ray Diffractogram of Natural CCT kaolin clay

Table 4.4: Chemical Characteristics of CCK, CCT & TCN Kaolin Clay

S. No	Chemical Characteristics	CCK	CCT	TCN	Requirement as per IS 1344- 1981 (Reaffirmed 1999)
1	Silica+ Alumina+ Iron Oxide	83.92%	81.10%	79.88%	70% Min
2	Silica	51.79%	48.66%	48.55%	40% Min
3	Calcium oxide	2.89	1.42	2.38	10% Max
4	Magnesium oxide	1.83%	1.72%	1.32%	3% max
5	Total sulfate	0.34%	0.67%	0.97%	3% Max
6	Alkalis(Na+ K oxides)	0.0049%	0.0011%	0.0001%	3% Max
7	Total loss on ignition	11.01%	15.08%	15.44%	10% Max

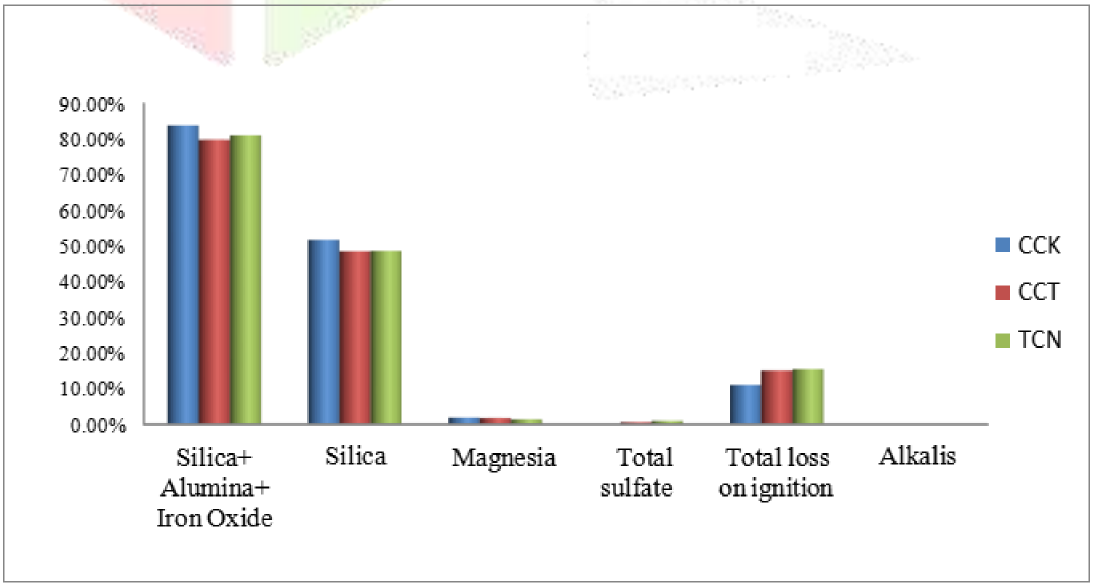

Figure 4: Chemical Characteristics of natural CCK, CCT and TCN kaolin clay

Chemical analysis was carried out on Cretaceous (CCK, CCT) and Tertiary (TCN) kaolin clay samples using procedure prescribed by IS: 1727-1967 (Reaffirmed 1999) to find out various chemical constituents present and their suitability to use as pozzolanic material in making concrete. The results of chemical analysis of natural Cretaceous and Tertiary kaolin clay are presented in Table 4.4. Result reveals that all chemical characteristics except loss on ignition of three clay samples are in conformity with requirement specified by IS: 1344- 1981 (Reaffirmed 1999) to use as pozzolanic materials. However, among three kaolinite clay samples, CCK sample appears to be the best pozzolanic material as it is having higher proportion of silica+ alumina+ iron oxide as well as silica and less total loss on ignition (Fig4). It is followed by CCT and TCN kaolin clay.

The result shows that lime reactivity of Cretaceous clay from Karai as well as Therani and Tertiary clay from Neyveli is significantly higher than requirement specified by IS: 1344-1981 (Reaffirmed 1999). Also, compressive strength of these three kaolin clays is remarkably higher than 80% of strength of corresponding plain cement mortar cubes. Further, physical characteristics indicate that among three kaolin clays, Karai clay (CCK) is the best pozzolan followed by Therani and Neyveli clay.

Table 4.4: Chemical Characteristics of CCK, CCT & TCN Kaolin Clay

S. No	Chemical Characteristics	CCK	CCT	TCN	Requirement as per IS 1344- 1981 (Reaffirmed 1999)
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7	Total loss on ignition	11.01%	15.08%	15.44%	10% Max

The CCK, CCT and TCN kaolin calcinated at 400º C, 600º C and 800º C was used to make binary blended cement. The mortar mixes, namely, MM-CCK, MM-CCT and MM-TCN were made with binary blended cement having 10, 15, 20 and 25% meta- kaolin (by weight of cement) and tested for compressive strength at 28 days. Results of MM-CCK, MM-CCT and MM-TCN mixes are furnished in Table 4.14, 4.15 and 4.16 respectively. Results show that compressive strength of CCK mortar is increasing with calcination temperature. Mortars made with CCK kaolin calcinated at 800ºC yielded highest compressive strength than mortars with kaolin calcinated at lower calcination temperature. Thermal activation of clay minerals at 800ºC leads to dehydroxylation, causing partial break down of structure. This imparts higher pozzolanic reactivity and enhances strength of mortar (Ambroise et al., 1986; Sayanam et al., 1989).

Table 4.14: Compressive Strength of CCK Blended Cement Mortar at 28 days

Mix code	Compressive strength (MPa)		
	Calcination temperature		
	400ºC	600ºC	800ºC
Control mix	50.22	50.22	50.22
MM-CCK10	49.13	49.33	51.21
MM-CCK 15	52.43	52.54	54.55
MM-CCK 20	50.43	51.63	53.22
MM-CCK 25	43.24	43.52	47.04

CONCLUSION

The salient conclusions drawn are presented below:

i)XRD analysis shows that kaolinite is present as a dominant mineral in clay sample collected from Cretaceous and Tertiary system, Tamil Nadu, India. Also, Quartz, Montmorillonite and clinochlore-ferrous are present as accessory minerals in clay samples. Among three clay samples, CCK clay is possessing higher proportion of Kaolinite and it is followed by CCT and TCN clay. Hence, CCK clay may be the best as pozzolanic material to use in concrete.

ii)Further, all chemical characteristics of three clay samples, except loss on ignition are in conformity with requirements specified by IS: 1344- 1981 (Reaffirmed 1999) to use as pozzolanic material. Also, CCK kaolin sample appears to be the best pozzolanic material as it is having higher proportion of silica+ alumina+ iron oxide as well as silica and relatively less total loss on ignition. It is followed by CCT and TCN kaolin clay. iii) Also, lime reactivity of all three kaolin clays is significantly higher than requirement specified by IS:1344-1981 (Reaffirmed1999). Also, compressive strength of these three clay samples is remarkably higher than 80% of the strength of corresponding plain cement mortar cubes as specified by IS: 1344-1981(Reaffirmed 1999). In addition, 8000C and 4 hours are optimum temperature and duration to calcinate Kaolin to produce meta- kaolin.

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