



International Journal of Engineering Research and Science & Technology

ISSN : 2319-5991
Vol. 4, No. 4
November 2015



www.ijerst.com

Email: editorijerst@gmail.com or editor@ijerst.com

Research Paper

PARTIAL REPLACEMENT OF CEMENT WITH METAKAOLIN IN HIGH STRENGTH CONCRETE

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Among many mineral admixtures available, Metakaolin (MK) is a mineral admixture, whose potential is not yet fully tested and only limited studies have been carried out in India on the use of MK for the development of high strength concrete. MK is a supplementary cementitious material derived from heat treatment of natural deposits of kaolin. MK shows high pozzolana reactivity due to their amorphous structure and high surface area. The experimental work has been carried out as partial replacement of cement with MK in M70 grade of concrete at 0%, 10%, 15%, 20%, 25% and 30% of replacements. The mix design was made making the use of Erntroy empirical Shacklock's method. Cubes are tested for durability studies with H₂SO₄ and HCL of 0.5% and 1% concentrations. Cubes, cylinders and prisms are tested for temperature study at 15% replacement. The specimens were heated to different temperatures of 100°C, 200°C, 300°C, 400°C and 500°C for three different durations of 1, 2 and 3 h at each temperature. Conclusions are made from the various results and the discussions there on to identify the effect of partial replacement of cement by MK in the design concrete mix. The results conclude that, the use of Metakaolin Concrete (MKC) has improved the performance of concrete under various conditions.

Keywords: Metakaolin, Concrete, Flexural strength, Compressive strength, Durability

INTRODUCTION

Concrete is one of the most widely used man-made construction material in the world. Metakaolin is the cementitious material used as an admixture to produce high strength concrete. Optimal quality of metakaolin for M70 grade of concrete has been worked out, which can replace the cement in order to get better strength and

durability. Also identification of the drying shrinkage and permeability characteristics of blended cement has been done.

A versatile material, High Strength Concrete (HSC) possesses desirable properties other than high strength. The most dramatic and memorable applications stem from this aspect, however, as high-rise buildings like 311 South Wacker Drive

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create striking visual impressions. This structure, at 969 ft (295 m), was the world's tallest concrete building when completed in 1989, utilizing concrete with compressive strengths of up to 12,000 psi (83 MPa).

Metakaolin is the white powder of $Al_2O_3 \cdot 2SiO_2$ by dehydrating kaolin ($Al_2O_3 \cdot 2SiO_3 \cdot 2H_2O$) at an appropriate temperature (700-900°C). Kaolin is in a layered silicate structure, with the layers binding with each other via the Van Der Waals bond, among which O is bound firmly. Kaolin, when being heated in air, may experience several structural changes, and when being heated to around 600°C, the layered structure of kaolin is damaged due to dehydration to form a transient phase with a poor crystallinity, i.e., metakaolin. As the molecular arrangement of metakaolin is irregular in a thermodynamic metastable condition, it is cementitious under an adequate excitation. With a high activity, metakaolin can be used to manufacture cementitious materials and mix high-strength high-performance concrete.

FORMING METAKAOLIN

The T-O clay mineral kaolinite does not contain interlayer cations or interlayer water. The temperature of dehydroxylation depends on the structure layer stacking order. Disordered kaolinite dehydroxylates between 530 and 570°C, ordered kaolinite between 570 and 630°C. Dehydroxylated disordered kaolinite shows higher pozzolana activity than ordered. The dehydroxylation of kaolin to metakaolin is an endothermic process due to the large amount of energy required to remove the chemically bonded hydroxyl ions. Above temperature range of dehydroxylation, kaolinite transforms into metakaolin, a complex amorphous structure which retains some long-range order due to layer

stacking. Much of the aluminum of the octahedral layer becomes tetrahedrally and pentahedrally coordinated. In order to produce a pozzolan (supplementary cementitious material) nearly complete dehydroxylation must be reached without overheating, i.e., thoroughly roasted but not burnt. This produces an amorphous, highly pozzolanic state whereas overheating can cause sintering, to form a dead burnt, nonreactive refractory, containing mullite and a defect Al-Si spinel. Reported optimum activation temperatures vary between 550 and 850°C for varying durations, however the range 650-750°C is most commonly quoted. In comparison with other clay minerals kaolinite shows a broad temperature interval between dehydroxylation and recrystallization, much favoring the formation of metakaolin and the use of thermally activated kaolin clays as pozzolans. Also, because the octahedral layer is directly exposed to the interlayer (in comparison to for instance T-O-T clay minerals such as smectites), structural disorder is attained more easily upon heating.

The water-cement ratio is the ratio of the weight of water to the weight of cement used in a concrete mix and has an important influence on the quality of concrete produced. A lower water-cement ratio leads to higher strength and durability, but may make the mix more difficult to place. Placement difficulties can be resolved by using plasticizers or super-plasticizers.

The concept of water-cement ratio was developed by Duff A Abrams and first published in 1918, see concrete slump test.

Metakaolin is a calcined product of the clay mineral kaolinite. The particle size of metakaolin is smaller than cement particles, but not as fine as silica fume. When kaolinite, a layered silicate

mineral with a distance of 7, 13 Å between the layer of SiO₂ and Al₂O₃ is heated, the water contained between the layers is evaporated and the kaolinite is activated for exaction with cement.

Metakaolin is refined kaolin clay that is fired (calcined) under carefully controlled conditions to create an amorphous aluminosilicate that is reactive in concrete. Like other pozzolans (fly ash and silica fume are two common pozzolans), metakaolin reacts with the calcium hydroxide (lime) byproducts produced during cement hydration. Calcium hydroxide accounts for up to 25% of the hydrated Portland cement, and calcium hydroxide does not contribute to the concrete's strength or durability. Metakaolin combines with the calcium hydroxide to produce additional cementing compounds, the material responsible for holding concrete together.

LITERATURE REVIEW

Khatib *et al.* (1996) investigated the porosity and pore size distribution of cured Ordinary Portland Cement-Metakaolin paste. Pastes containing 0, 5, 10 and 15 Metakaolin were prepared at a constant water / binder (w/b) ratio of 0.55. Specimens were moist cured for period from 3 days to 365 days. The intruded pore volume and the pore structure were determined by mercury intrusion porosimetry..

Rama Rao *et al.* (1997) concluded that the durability of concrete using rice husk ash as an admixture is better in terms of permeability, abrasion and chemical resistance to sulphate attack. Khatib *et al.* (1998) in their research said that the partial substitution of cement with Metakaolin is investigated in terms of resistance of Metakaolin mortar to Sodium Sulphate (Na₂So₄) Solution and some specimens are cured in water.

Results on strength, pore size distribution, porosity, are reported.

Long T Phan *et al.* (2000) A compilation of fire test data which shows distinct behavioral differences between High-Strength Concrete (HSC) and Normal Strength Concrete (NSC) at elevated temperature is presented. Fraiss *et al.* (2000). The authors show the results of an investigation focusing on the effect of Metakaolin (MK) on the micro-structure of MK-blended pastes. Pastes containing 0%, 10%, 15%, 20% and 25% of MK were prepared at a constant water/binder ratio of 0.55 and cured at 200°C for hydration periods from 1 to 360 days.

Xia Oquian and Zongjinli (2001) studied the stress–strain relationships of concrete containing 0% to 15% of Metakaolin at an incremental rate of 5%. They concluded that incorporation of Metakaolin up to 15% has increased the tensile and compressive strength and also peak strain is increased at increasing rate of Metakaolin up to 15%. Incorporation of Metakaolin has slightly increased the compressive elasticity modulus.

Bo wu, Xiao - Ping Su and Huili (2002) studied the effect of high temperature on residual mechanical properties of confined and unconfined high strength concrete. They varied the temperature from 100°C to 900°C. Also elastic modulus decreases sharply at the higher temperatures.

Chi-Sun Poon *et al.* (2003) an experimental investigation was conducted to evaluate the performance of metakaolin (MK) concrete at elevated temperatures up to 800°C. Eight normal and HSC mixes incorporating 0%, 5%, 10% and 20% MK were prepared.

Srinivasa Rao *et al.* (2004) investigated the effect of elevated temperatures ranging from 50

to 250°C on the tensile strength (splitting and flexural) of HSC made with Portland Cement (PC) and Pozzolana Portland Cement (PPC), for application in the chemical and metallurgical industries or thermal shielding of nuclear power plants. Tests were conducted on 150 mm diameter cylindrical specimens and 100 mm x 100 mm x 500 mm beam specimens..

Abdul Razak *et al.* (2005) in this study, metakaolin (MK) and silica fume (SF) were used as cement replacement materials at 5%, 10%, and 15% by mass. Water/cementitious materials (w/cm) ratios varied from 0.27 to 0.33, and strength testing was conducted up to an age of 180 days.

Chi-Sun Poon *et al.* (2006) this study is to relate the mechanical and durability properties of high performance metakaolin (MK) and silica fume concretes to their microstructure characteristics. The compressive strength and chloride penetrability of the control and the concretes incorporated with MK or silica fume (SF) at water-to-binder (w/b) ratios of 0.3 and 0.5 are determined.

Nabil M. Al-Akhras (2006) this study investigates the effect of metakaolin (MK) replacement of cement on the durability of concrete to sulfate attack. Three MK replacement levels were considered in the study: 5%, 10%, and 15% by weight of cement. The other experimental parameters investigated in the study were: water to binder ratio (0.5 and 0.6), initial moist curing period (3, 7, and 28 days), curing type (moist and autoclaving), and air content (1.5% and 5%). After the specified initial moist curing period, concrete specimens were immersed in 5% sodium sulfate solution for a total period of 18 month.

Bamonte *et al.* (2010) the present investigation deals with high temperature, in order to evaluate the thermal diffusivity and the mechanical decay as a function of the temperature, since there is still scanty information in the literature on the high-temperature behavior of this family of materials.

Rafat Siddique *et al.* (2010) an investigation dealing with the effect of metakaolin (MK) on the near surface characteristics of concrete are presented in this paper. A control concrete having cement content 450 kg/m³ and w/c of 0.45 was designed.

Dinakar *et al.* (2011) examined High Reactive Metakaolin (HRM) for high strength and high performance concrete. According to him Supplementary Cementing Materials (SCMs) such as fly ash, silica fume and GGBS are increasingly used in recent years as cement replacement material. They help obtain both higher performance and economy

Beulah *et al.* (2012) this paper presents an experimental investigation on the effect of partial replacement of cement by metakalion by various percentages, viz., 0%, 10%, 20%, and 30% on the properties of high performance concrete, when it is subjected to hydrochloric acid attack. Vikas Srivastava *et al.* (2012) this study deals with the addition of some pozzolanic materials, the various properties of concrete, viz., workability, durability, strength, resistance to cracks and permeability can be improved.

In this present work is to find the mechanical properties of partially replaced cement with Metakaolin at 0%, 10%, 15%, 20%, 25% and 30% for M70 grade of concrete. To evaluate compressive strength, split tensile strength, Flexural strength and stress-strain curve of MKC

at 15% replacement by exposing to temperature of 100°C, 200°C, 300°C, 400°C and 500°C. Calculated the mix proportion by partial replacement of OPC with 0%, 10%, 15%, 20%, 25% and 30% of MK.

RESULTS AND DISCUSSION

The tests were carried out to obtain compressive strength, split tensile strength, flexural strength and stress-strain curve of M70 grade concrete. The specimens are tested for 28 days for 0%, 10%, 15%, 20%, 25% and 30% replacement of MK for compressive strength and the specimens are tested for 28 days for 0% and 15% replacement of MK for flexural strength, stress-strain curve, split tensile strength. These are presented in tables and graphs were plotted correspondingly.

In the present experimental work the specimens exposed to temperature undergo physical changes and weight loss. The free moisture content is lost initially, followed by physical adsorption of water. The mix design proportions are mentioned in Table 1 and the results are plotted in Figures 1 to 18.

Effect of Variation of Metakaolin on Compressive Strength

From the below figure it is observed that at 15% replacement of cement with MK, concrete attains its maximum compressive strength. When the replacement exceeds 15%, the compressive strength is found to be decreasing slightly.

Effect of H₂SO₄ and HCL acids on Metakaolin Concrete (Durability Studies)

Concrete cubes of 0%, 10%, 15%, 20%, 25% and 30% of MKC of M70 grade concrete exposed to HCL and H₂SO₄ of 0.5% and 1% concentrations are tested for compressive strength for 28 days respectively. The results are presented in the following tables and the graphs are plotted. From the below figure, it is observed that at 15% replacement of cement with MK, concrete attains maximum compressive strength when exposed to 0.5%, 1% HCl at the age of 28 days. When the replacement exceeds 20%, the compressive strength is found to be decreasing slightly.

Compressive strength for M70 Grade of Concrete after 0.5% and 1% H₂SO₄ Acid Curing

From the below figure, it is observed that at 15% replacement of cement with MK, concrete attains maximum compressive strength when exposed to 0.5%, 1% H₂SO₄ at the age of 28 days. When the replacement exceeds 20%, the compressive strength is found to be decreasing slightly.

Temperature Studies

Effect of Temperature on Compressive Strength of Metakaolin Concrete

From the below figure, it is observed that the compressive strength increases at 100°C temperature when compared to the strength obtained at normal room temperature for 0% and 15% replacement of MK. The increase in

Table 1: Design Mix Proportions (M70 Grade)					
Design Method	Maximum Size Of C.A	Mix Proportions (By Weight)			
		Cement	F.A	C.A	W/C Ratio
ERNSTROY & SHACKLOCK	20 mm	1	1.155	2.145	0.30

Figure 1: Compression Strength of Concrete vs. % of Metakaolin

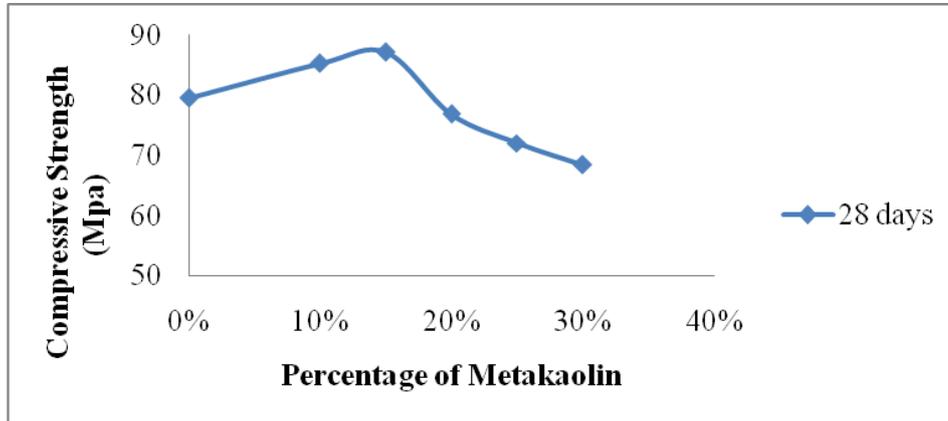


Figure 2: Compressive Strength of concrete vs. % of MK at 0.5% and 1% HCl

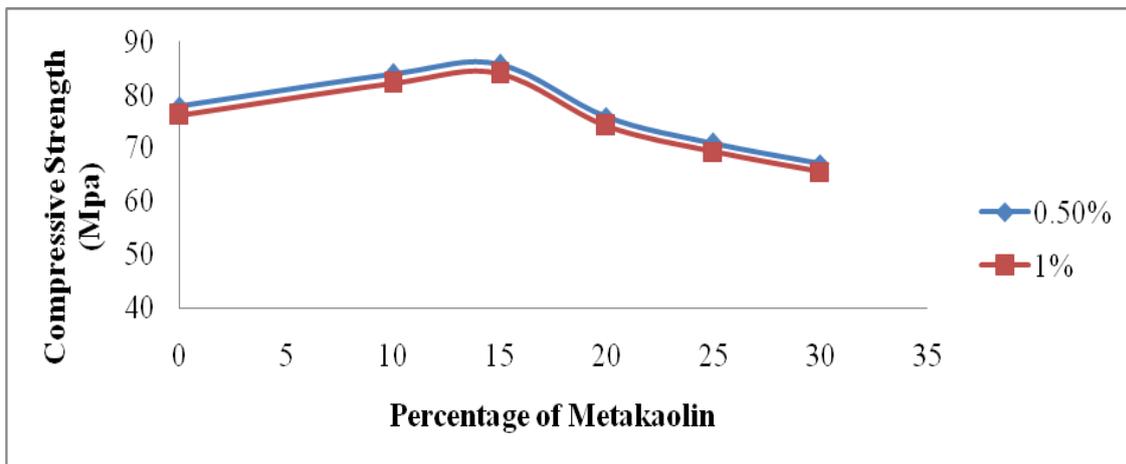


Figure 3: Compressive Strength of concrete vs. % of Metakaolin at 0.5%, 1% H2SO4'

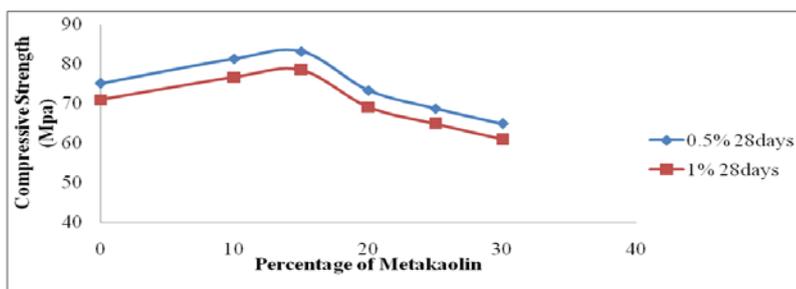


Figure 4: 28 Days Compressive Strength of concrete (%) vs. Exposed Temperature (°C) of 0% MKC

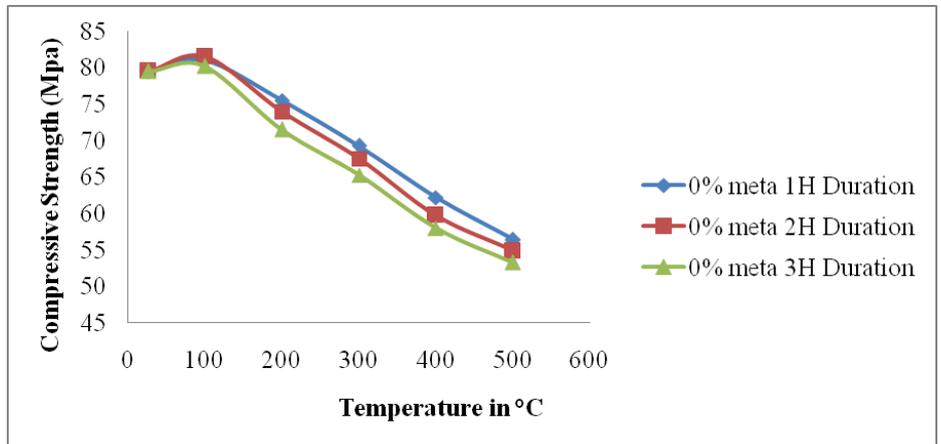


Figure 5: 28 Days Compressive Strength of concrete (%) vs. Exposed Temperature (°C) of 0% MKC

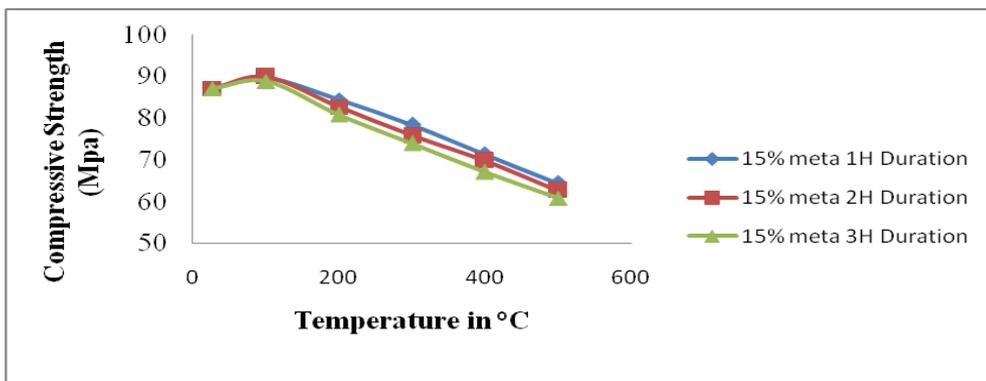


Figure 6: 28 Days Compressive Strength of concrete (%) vs. Exposed temperature (°C) of 0% and 15% MKC

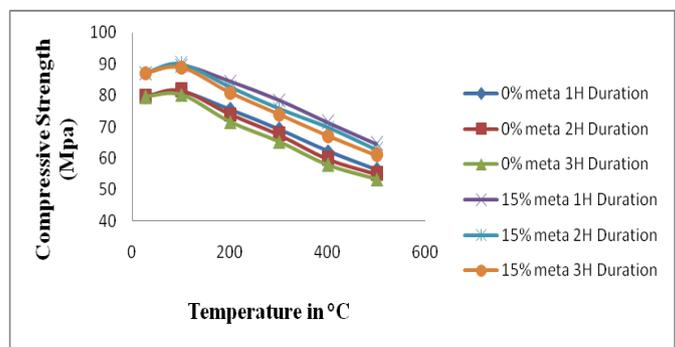


Figure 7: 28 Days Split Tensile Strength of concrete (%) vs. Exposed Temperature (°C) of 0% MKC

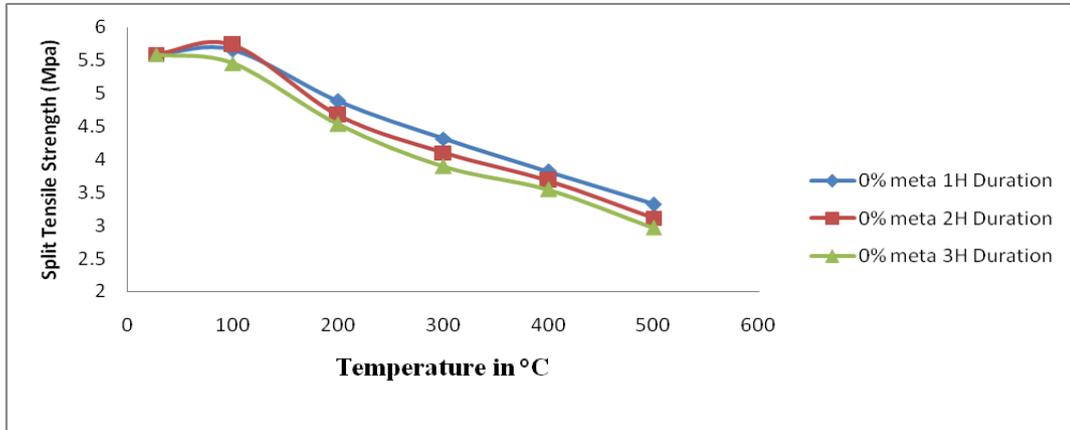


Figure 8: 28 Days Split Tensile Strength of concrete (%) vs. Exposed Temperature (°C) of 15% MKC

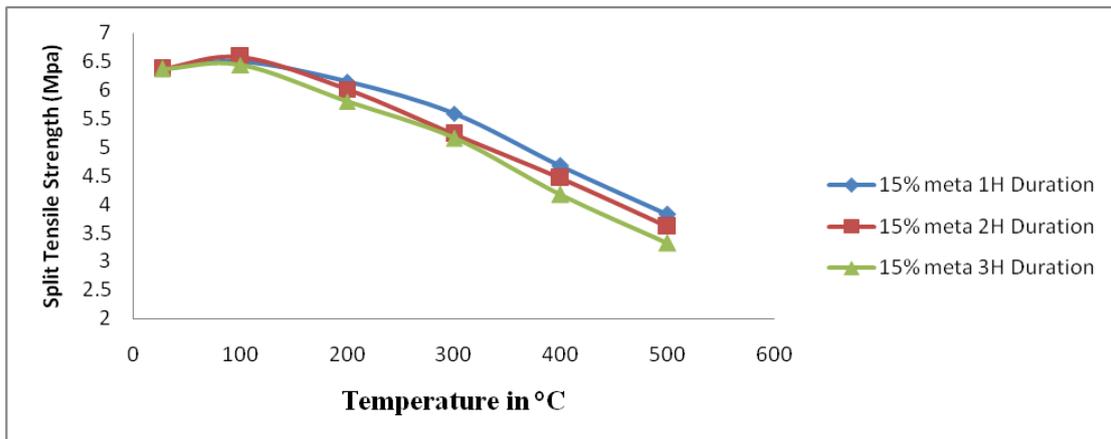
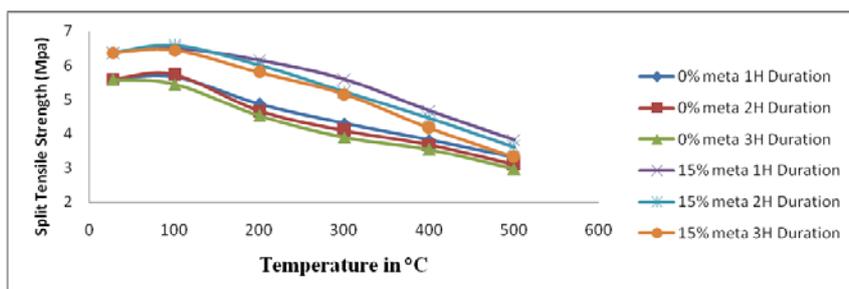


Figure 9: 28 Days Split Tensile Strength of concrete (%) vs. Exposed Temperature (°C) of 0% and 15% MKC



compressive strength associated with the increase in temperature is attributed to the increase in the surface forces between gel particles (Vander wall forces) due to the removal of moisture content. The compressive strength decreases from 100°C to 200°C and further decreases at 500°C.

Effect of Temperature on Split Tensile Strength of Metakaolin Concrete

From the below figure, it is observed that the split tensile strength increases at 100°C temperature when compared to the strength obtained at normal room temperature for 0% and 15% replacement of MK. The increase in split tensile strength associated with the increase in temperature is attributed to the increase in the surface forces between gel particles (Vander wall forces) due to the removal of moisture content.

Effect of Temperature on Flexural Strength of Metakaolin Concrete

From the below figure, it is observed that the flexural strength increases at 100°C temperature when compared to the strength obtained at normal room temperature for 0% and 15% replacement of MK. The increase in flexural strength associated with the increase in temperature is attributed to the increase in the surface forces between gel particles (Vander wall forces) due to the removal of moisture content.

Effect of Temperature on Stress-Strain Curve of Metakaolin Concrete

The stress-strain behavior of cylinder specimens for 0% and 15% of metakaolin cured for 28 days age and subjected to elevated temperature from 100 to 500°C apart from room temperature were as shown below. The various graphs plotted are as shown in below figure.

Figure 10: 28 Days Residual Flexural Strength of concrete (%) vs. Exposed Temperature (°C) of 0% MKC

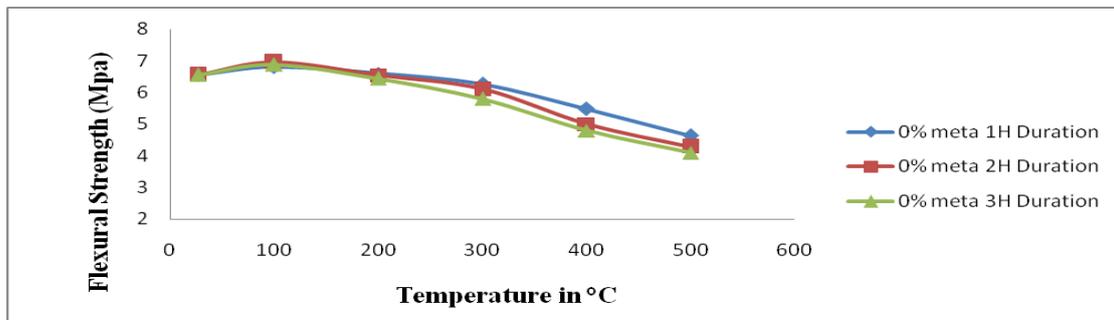


Figure 11: 28 Days Flexural Strength of concrete (%) vs. Exposed Temperature (°C) of 15% MKC

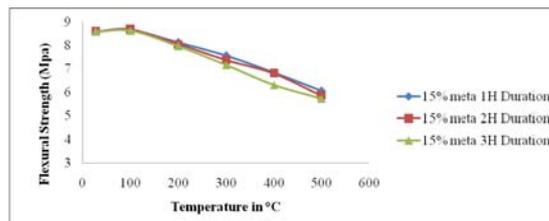


Figure 12: 28 Days Flexural Strength of concrete (%) vs. Exposed Temperature (°C) of 0% and 15% MKC

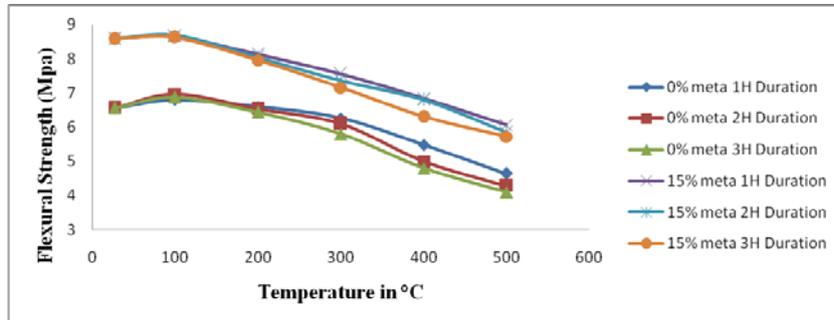


Figure 13: Strain curve of concrete at room temperature

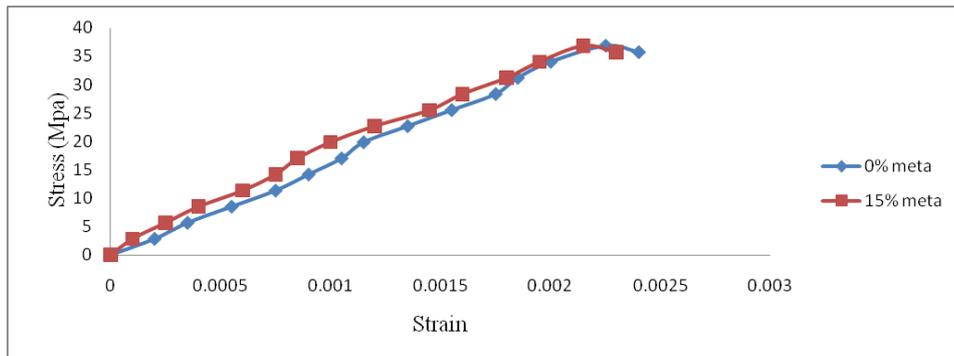


Figure 14: Stress-strain curve of concrete exposed to 100°C for different exposure durations

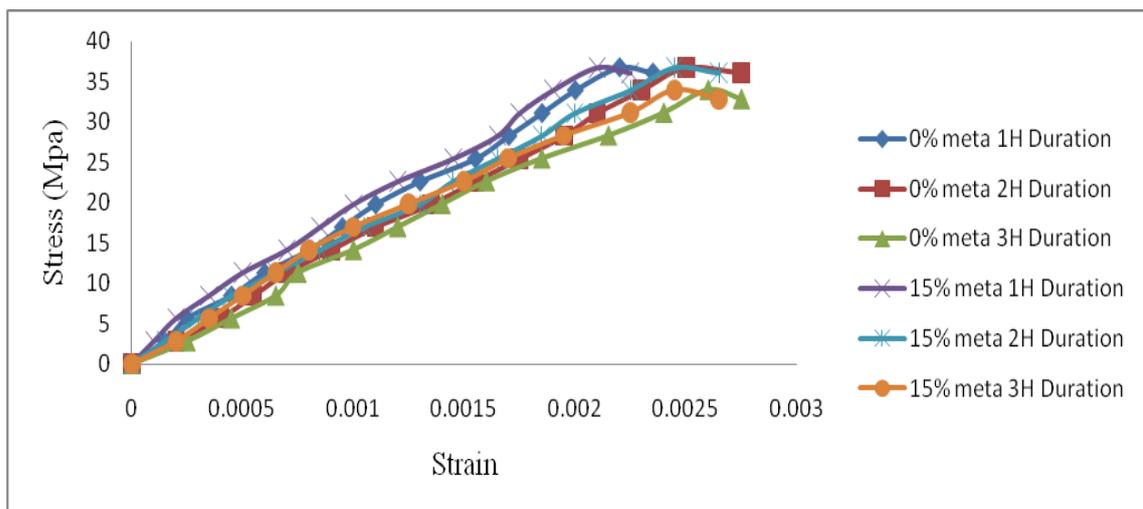


Figure 15: Stress-strain curve of concrete exposed to 200°C for different exposure durations

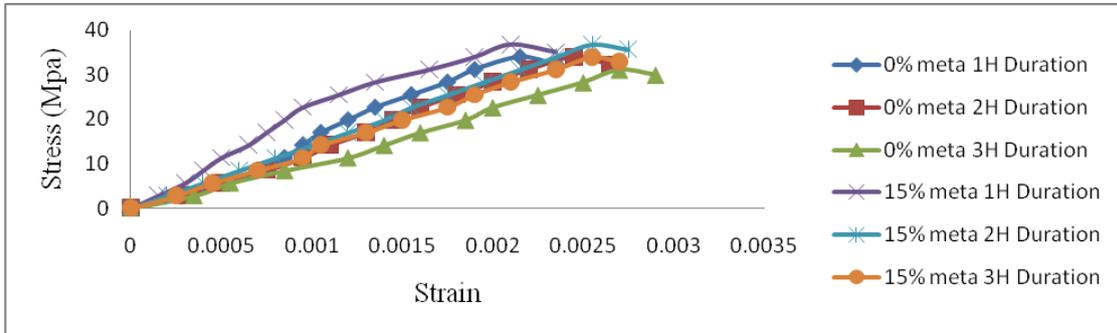


Figure 16: Stress-strain curve of concrete exposed to 300°C for different exposure durations

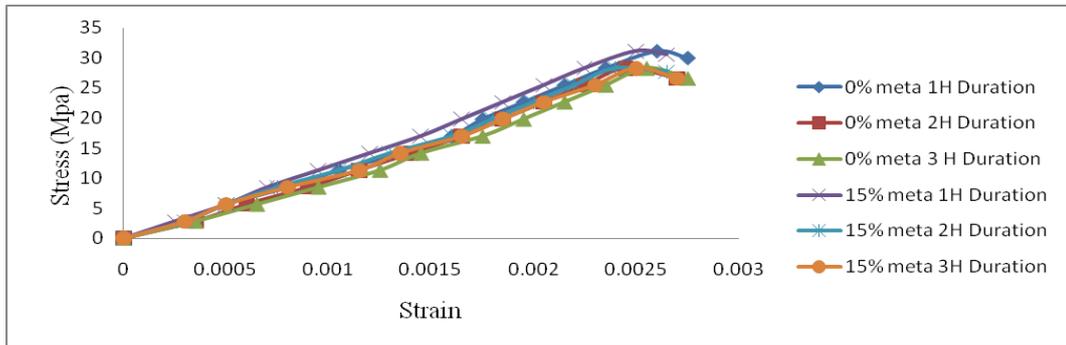


Figure 17: Stress-strain curve of concrete exposed to 400°C for different exposure durations

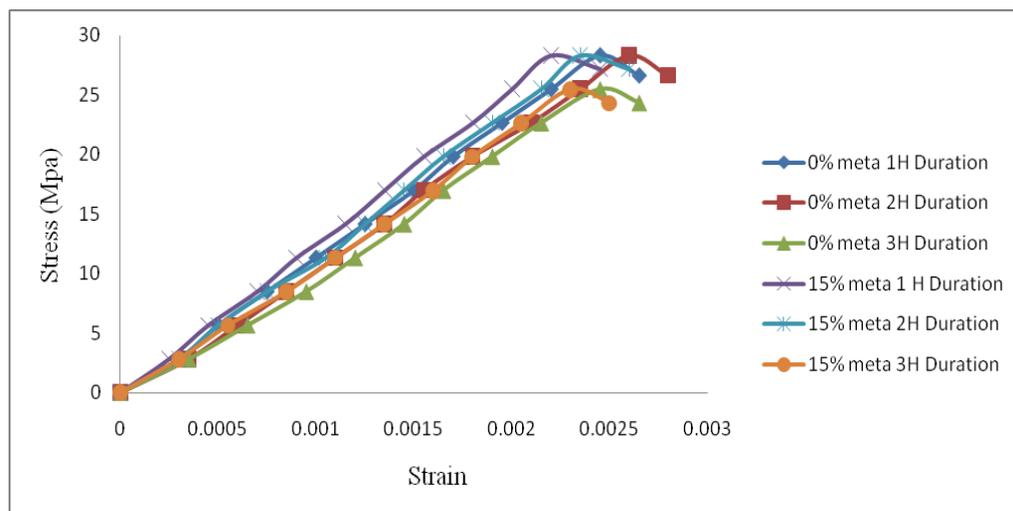
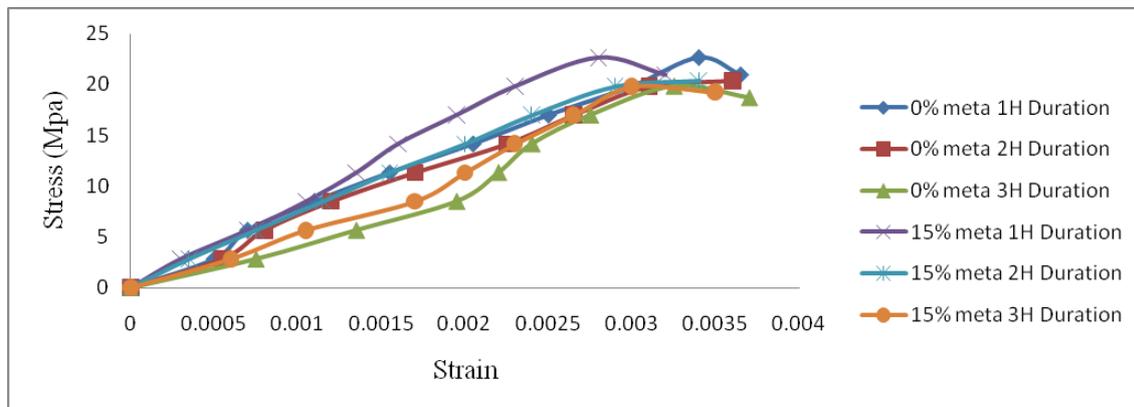


Figure 18: Stress-strain curve of concrete exposed to 500°C for different exposure durations

CONCLUSION

Based on the experimental investigation carried out, the following conclusions are made.

- Workability of concrete decreases with the increase in Metakaolin replacement level.
- The compressive strength, flexure strength and split tensile strength of conventional concrete and concrete with MK as partial replacements are compared and observed and concluded that the strength of the conventional concrete is slightly lower than the MKC.
- The compressive strength of concrete is increased when cement is replaced with Metakaolin. The compressive strength is maximum at 15% of replacement.
- The split tensile strength of concrete is increased when cement is replaced with Metakaolin. The split tensile strength is maximum at 15% of replacement.
- The flexure strength of concrete is increased when cement is replaced with Metakaolin. The flexure strength is maximum at 15% of replacement.
- At room temperature and 100°C exposure, the stress-strain relationship is similar to the conventional concrete and MKC behavior. However the trend is different for temperature exposure of 200°C to 500°C.
- The compressive strength of concrete showed better result at 15% replacement of MK for 0.5% and 1% HCl at the age of 28 days of strength.
- The compressive strength of concrete showed better result at 15% replacement of MK for 0.5% and 1% H₂SO₄ at the age of 28 days of strength.
- The effect of HCl on strength of the Metakaolin concrete is lower than the effect of H₂SO₄.
- The strength increases at 100°C temperature and thereafter it starts losing its strength as the temperature increases.

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International Journal of Engineering Research and Science & Technology

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