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Performance Characterization of Concrete Using Metakaolin as Supplementary Cementing Material

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ABSTRACT

This research presents the mechanical, durability and micro structural properties of concrete using metakaolin by partial replacement of cement. In this context, metakaolin replaced at 10%, 20% and 30% by weight of cement water cement ratio 0.47. For this purpose, mix design was prepared according to IS 10262: 2019 for M25 and OPC 43 grade of concrete. Various mixes of cubes and cylinders were prepared to check the compressibility and split tensile strength after 7 and 28 days of curing. To determine the durability properties of concrete, water absorption test was also performed after 28 days. Once the tests were performed, samples from broken pieces of cubes are taken for micro structural analysis. This was analyzed by Scanning Electron Microscope (SEM). It was observed that 20% of metakaolin was considered to be suitable as replacement of cement. The compressive strength and tensile strength at 7 and 28 days found to be higher as compared to other percentages.

Key Words: Metakaolin, Compressive strength, Water absorption, Split tensile strength, SEM.

Introduction

Since its beginning, construction is most important part of the civil engineering profession. Civil engineers got involved in the development, construction, and operation of transit facilities, such as underground systems, street railways and elevated, as cities evolved (Mohod & Kadam, 2016).

The Concrete the mostly used and adaptable material for construction because concrete is often less expensive than steel, is rust and fire resistant (non-corrosive and fire resistant), and can be manufactured with locally available materials. There is constantly a desire for stronger, more versatile, cost-effective concrete and environment friendly material. Blended cement concrete has been used in this case to meet the requirements. Cement manufacture, along with all other concrete components, is hazardous to the environment because enormous amounts of CO_2 are released during the process (Kalaignan & Reddy, 2016). As a result, finding an appropriate material for Cement replacement is critical in the current setting of growing environmental awareness (Seelapureddy et al., 2020)**.**

The word 'Meta' in the term means 'to change,' and Kaolin is a stone with a high percentage of Kaolinite.. Kaolin is a fine white clay mineral that has long been used in the manufacture of porcelain. Kaolin is thought to have originated from the Chinese Kaolin, which loosely translates to "white hill" and has been related to the name of a Chinese mountain that delivered the first kaolin to Europe. Kaolin clays are referred to as kaolinite in mineralogical terms. The most common component of kaolin is hydrated aluminium disilicate $AlSi_2O_5\ (OH)_4$, which is a common mineral. This clay is used in the production of adhesives, paper, bricks, paint, rubber, ceramics, refractories, cement, plastics, among other things. Except for those linked to building, all of these uses need high-grade kaolinite clay. As a result, the construction industry is encouraged to focus on the benefits of utilizing this clay, given its relative availability,

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particularly in quickly growing parts of the world, and in terms of minimizing the effect of global warming related to Portland cement manufacturing.

The formation of metakaolin given below: (Ilić et al., 2010)

$$\underset{\text{(aluminium silicate)}}{Al_2O_3.\ 2SiO_2.\ 2H_2O} \xrightarrow{\text{calcination at } 650^{\circ} \text{ to } 800^{\circ}\text{ C}} \underset{\text{(metakaolin)}}{Al2O3\ .2(SiO2)} + 2H2O$$

Metakaolin fills the empty spaces between the cement particles in concrete mixes, resulting in concrete structure is more impermeable. The C-S-H gel is formed when calcium hydroxide reacted with metakaolin. The reaction has given below: (Chandak & Pawade, 2020)

$$\text{Cement} + \text{Water} \longrightarrow C - S - H \text{ gel} + Ca(OH)_2$$

$$Ca(OH) + \text{Metakaolin} \longrightarrow C - S - H \text{ gel}$$

Globally, kaolinite clay extraction and metakaolin production plants are only found in the United States, India, and China, while limited in Africa. In some locations, there is a lack of industrial reaction to the use of metakaolin and other SCMs such as fly ash and slag. In May 2015, the United Arab Emirates (UAE) implemented new laws requiring that at least 60% of all major infrastructure projects and substructures utilize slag or ash-containing cement. If such regulations adopted in a number of countries, particularly in Africa, will help industry in using metakaolin.

Their findings showed that the substitution of metakaolin as cement replacement can increase both compressive and tensile strength compared to normal concrete. The compressive strength of SCC was found to be increased by 53.3MPa with 15% inclusion of MK and tensile strength of SCC was increased by 3.6MPa with 10 % of MK(Shahidan 2017). evaluated that 15% of metakaolin is sufficient for replacement of cement. It improves the compressive strength and split tensile strength (Narmatha and kala, 2017). It is possible to manufacturing high-performance concrete with low metakaolin reactivity (Salimi et al., 2019). MK can be used as alternative binder material for protecting coastal/marine concrete structures against chloride damage (Pillay et al., 2020)**. R**eplacing cement with metakaolin may considerably extend the life of buildings. It is frequently recommended that supplementary cementing materials such as silica fume or blast furnace slag be used to improve the durability of concrete in maritime environments. Metakaolin provides for a considerable improvement in chloride diffusion even having low purity (Bucher et al., 2021)**.** Findings shows that 10% MK with 1% super plasticizer in pervious concrete sample exhibits 4 times greater compressive strength at 7 days as compared to normal control mix (Supit and Pandei, 2019).

Experimental Programme

Cement

In this study, OPC-43 grade was used. The cement was tested in accordance with IS: 4031-1988, and its qualities were found to be in accordance with IS: 8112-1989.

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Metakaolin

Metakaolin was procured from HS Mineral and Chemicals Baroda, Gujrat. The kaolinite clay was heated at temperatures of 600 °C to 900 °C. The calcined clay was crushed to make a fine powder. It was light mineral with specific gravity of 1.903, pinkish in colour and has texture like clay.

Chemical examination of the sample provided by the producer indicated that the brightness of metakaolin is in the range of 74±1 (%) with a bulk density of 300 – 500 (gm/L). The moisture content of sample is < 0.5 %. Other oxide constituents such as SiO_2, Al_2O_3, Fe_2O_3, MgO also necessary in cement manufacturing was found to be 52±1, 42±1, < 1.3, and < 0.5 percent, respectively.

Table:1 Chemical composition

Grade	Range
Brightness (%)	74±1
Bulk density (gm/L)	300 - 500
Color on panel	Pinkish
Grit content ((325#)	< 0.5
Moisture (%)	<0.5
D (50)	1.5
Oxide constituents	Range
SiO_2 %	52 ±1
Al_2O_3 %	42 ±1
Fe_2O_3 %	< 1.3
TiO_2 %	0.5 Max
CaO %	< 0.5
MgO %	< 0.5
Na_2O% K_2O%	0.5 – 2.5
L.O.I	0.8 to 1.5
POZZOLAN REACTIVITY –mg $Ca(OH)_2$	> 1000

Fig.1 Metakaolin powder

Coarse Aggregates

In this study the coarse aggregates of size 10 mm and 20 mm was used and it was tested as per IS 3114 – 2013. The sieve analysis and properties of coarse aggregates are shown in tables 2 and 3 respectively.

Table: 2 Properties of coarse aggregates

Sr. no.	Properties	Coarse aggregate (20mm)
1	Specific gravity	3.29
2	Water absorption	0.8%

Table: 3 Sieve analysis of coarse aggregates

IS sieve sizes	Percent passing		Percent passing		Combined 100%	Requirement as per IS:3114-2013
	20 to 10 mm	10 mm down	50% 20 mm	50% 10mm		
20 mm	95.5	100	47.5	50	97.8	95 – 100
10 mm	2.27	77	1.14	39	39.21	25 - 55
4.75 mm	0	12.4	0	6.17	6.17	0 - 10

Table:4 Physical properties of fine aggregates

Fine Aggregates

In this study maximum size of 4.75 mm size fine aggregates was used. The sieve analysis and physical properties of fine aggregates was shown in table 4.

Methodology Adopted for Mix Design

Mix design is the process of selecting acceptable components for concrete and establishing their proportions in order to generate a concrete that meets the project requirements, such as workability, compressive strength, and split tensile strength, as economically as possible. Concrete proportioning is an essential aspect of concrete technology because it assures both quality and economy. The first stage in achieving the aim of creating concrete with specified performance characteristics is to selecting component materials; the second step is to use a technique called mix design to arrive at the best combination of components.

Mix Composition

The concrete mixes were prepared with constant cementitious material, coarse aggregates and fine aggregates by using various percentages of metakaolin content.

Table:5 Mix Composition (Standard Surface Dry Aggregate)

Cement kg/m^3	Water kg/m^3	Fines kg/m^3	Coarse kg/m^3	w/c ratio
408	208	604	1492	0.47
1	0.5	1.48	3.65	-

Results and Discussions

Compressive Strength

Compressive strength and percentage change with respect to CM (control mix) was shown in table 24 and 25. The compressive strength for CM at 7 days containing 0% metakaolin was 33.55 N/mm^2. Whereas mix containing 10% and 20% of MK showed strength of 34.05 N/mm^2 and 36.78 N/mm^2 respectively and giving percentage increase in strength of 10.57% and 10.96% w.r.t control mix. We can see from this pattern that as the amount of Metakaolin in the mix increases, the compressive strength of the mix increases as well. Mix having 30% of metakaolin showed compressive strength of 33.22 N/mm^2 and giving percentage increase in strength of 9.01% with respect to control mix.

Sr. no.	Properties	Fine aggregates
1	Specific gravity	2.61
2	Fineness modulus	3.16
3	Water absorption	0.8%

If we compare compressive strength at replacement level i.e., 10%, 20% and 30% respectively, we can see that mix with 20% MK shows higher compressive strength than other mixes. Further increase in metakaolin percentage reduced 7 days compressive strength below the target strength

It is found mix having 20% of metakaolin gives higher compressive strength.

Compressive Strength at 28 days

Compressive strength and percentage change with respect to CM (control mix) was shown in table 26 and 27. The compressive strength for CM at 28 days containing 0% metakaolin was 37.73 N/mm^2. Whereas mix containing 10% and 20% of MK showed strength of 38.72 N/mm^2 and 39.70 N/mm^2 respectively and giving percentage increase in strength of 10.26% and 10.52% w.r.t control mix. We can see from this pattern that as the amount of Metakaolin in the mix increases, the compressive strength of the mix increases as well. Mix having 30% of metakaolin showed compressive strength of 33.22 N/mm^2 and giving percentage increase in strength of 9.36% with respect to control mix.

If we compare compressive strength at replacement level i.e., 10%, 20% and 30% respectively, we can see that mix with 20% MK shows higher compressive strength than other mixes. Further increase in metakaolin percentage reduced 28 days compressive strength below the target strength. It is found that mix having 20% of metakaolin gives higher compressive strength.

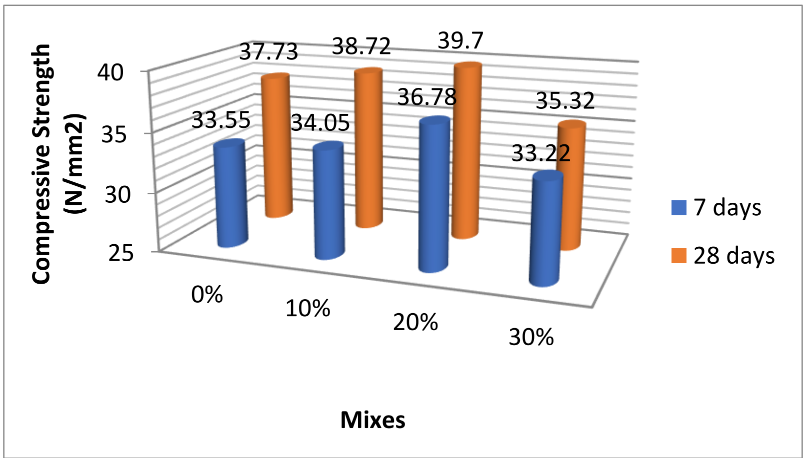
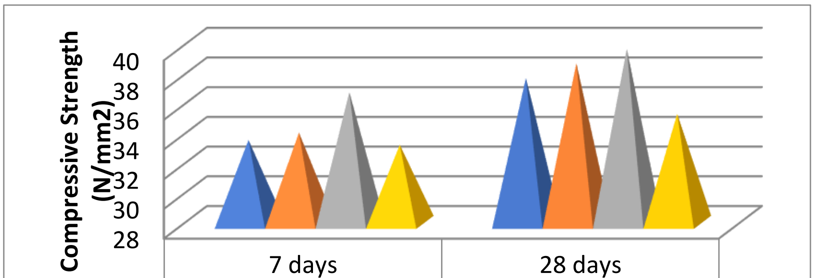


Fig: 2 Compressive strength Vs Mixes



	7 days	28 days
0%	33.55	37.73
10%	34.05	38.72
20%	36.78	39.7
30%	33.22	35.32

Fig:3 Compressive strength Vs Ages

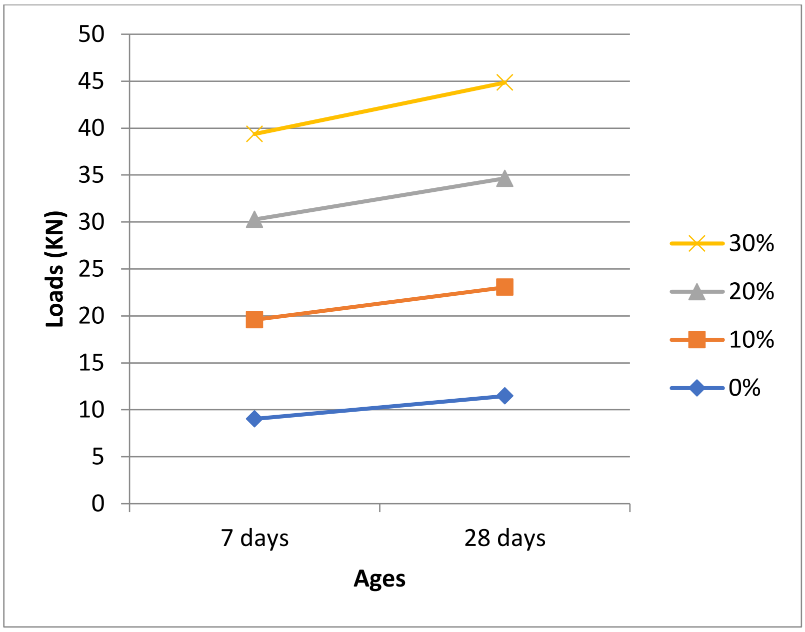


Fig:4 Loads at 7 and 28 days

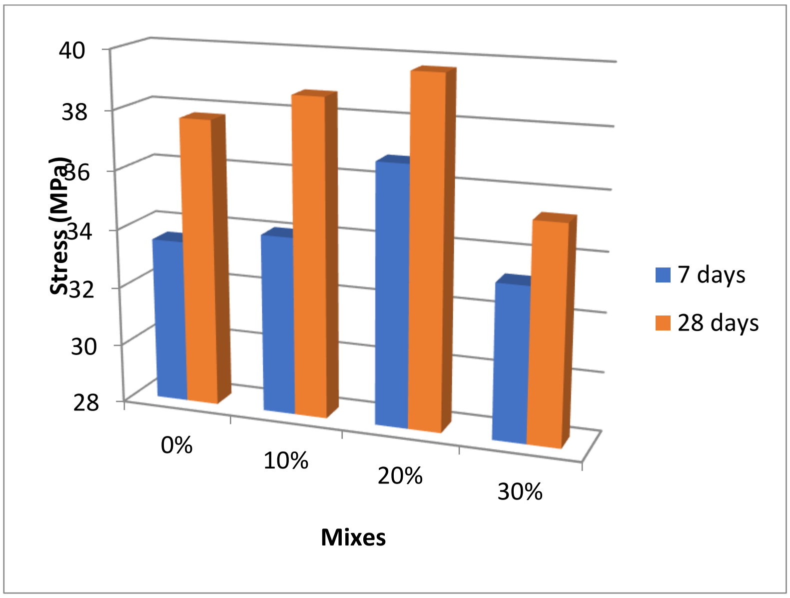


Fig:5 Stress at various mixes

Split Tensile Strength

Cylinders of M-25 grade concrete of size 30 cm length and 15 cm diameter were casted evaluating split tensile strength after 7 and 28 days of curing period. As various mix proportions were prepared same as of compressive strength.

Split tensile Strength at 7 days

Split strength and percentage change with respect to CM (control mix) was shown in table 29 and 30. The compressive strength for CM at 7 days containing 0% metakaolin was 33.55

N/mm^2. Whereas mix containing 10% and 20% of MK showed strength of 10.56 N/mm^2 and 10.68 N/mm^2 respectively and giving percentage increase in strength of 11.6% and 11.8% w.r.t control mix. We can see from this pattern that as the amount of Metakaolin in the mix increases, the split tensile strength of the mix increases as well. Mix having 30% of metakaolin showed tensile strength of 9.11 N/mm^2 giving percentage increase in strength of 10.8% with respect to control mix.

If we compare split tensile strength at replacement level i.e., 10%, 20% and 30% respectively, we can see that mix with 20% MK shows higher tensile strength than other mixes. Further increase in metakaolin percentage reduced 28 days tensile strength below the target strength. It is found that mix having 20% of metakaolin gives higher strength.

Split tensile Strength at 28 days

Split strength and percentage change with respect to CM (control mix) are shown in tables 31 and 32.The compressive strength for CM at 28 days containing 0% metakaolin was 11.46 N/mm^2. Whereas the mix containing 10% and 20% of MK showed strength of 11.58 N/mm^2 and 11.61 N/mm^2 respectively, giving a percentage increase in strength of 10.10% and 10.13% w.r.t control mix. We can see from this pattern that as the amount of Metakaolin in the mix increases the split tensile strength of the mix increases as well. Mix having 30% of metakaolin showed tensile strength of 10.20 N/mm2, giving a percentage increases in strength of 8.90% with respect to the control mix.

If we compare split tensile strength at replacement level, i.e., 10%, 20% and 30% respectively, we can see that the mix with 20% MK shows higher tensile strength than the other mixes. Further increase in metakaolin percentage reduced 28 day tensile strength below the target strength. It is found that a mix having 20% of metakaolin gives higher strength.

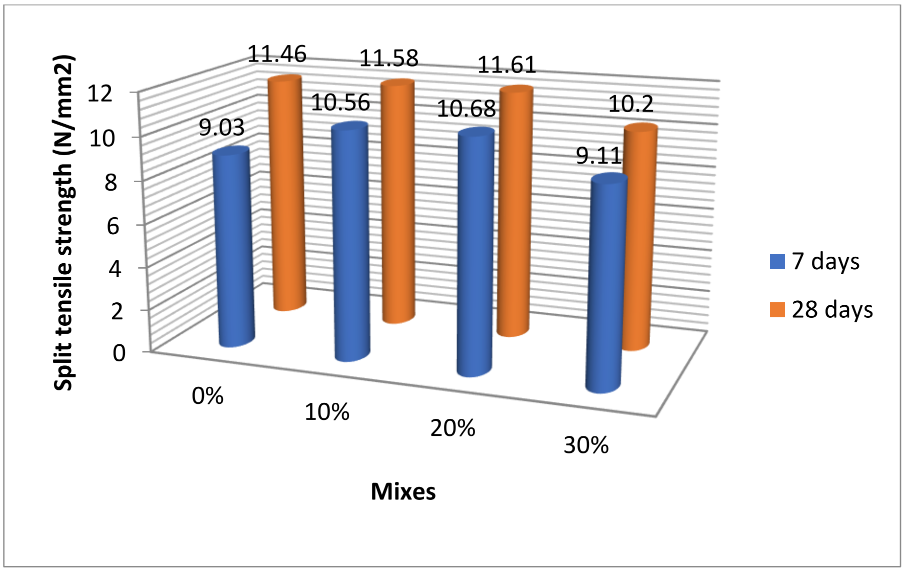


Fig.7 Split tensile strength Vs Mixes

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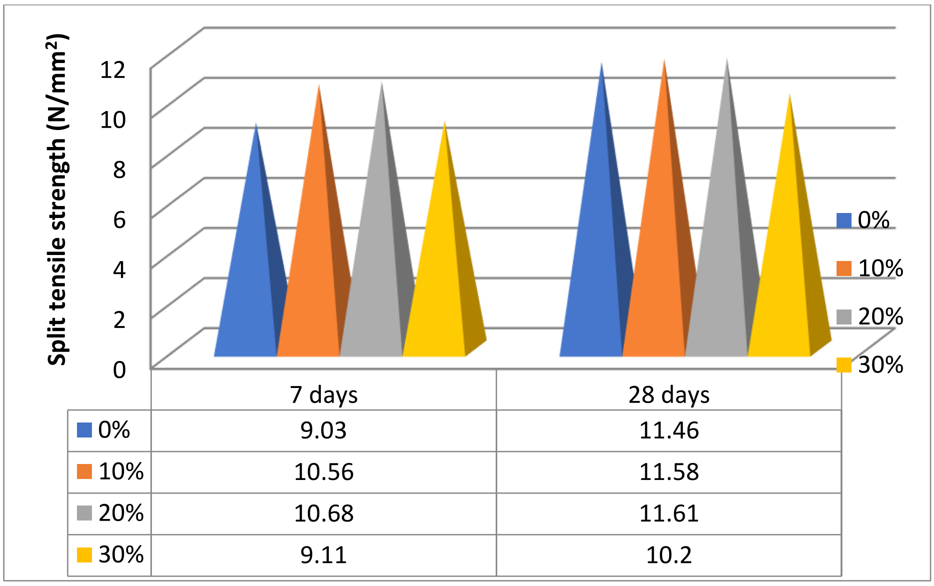

Fig.8 Split tensile strength Vs ages

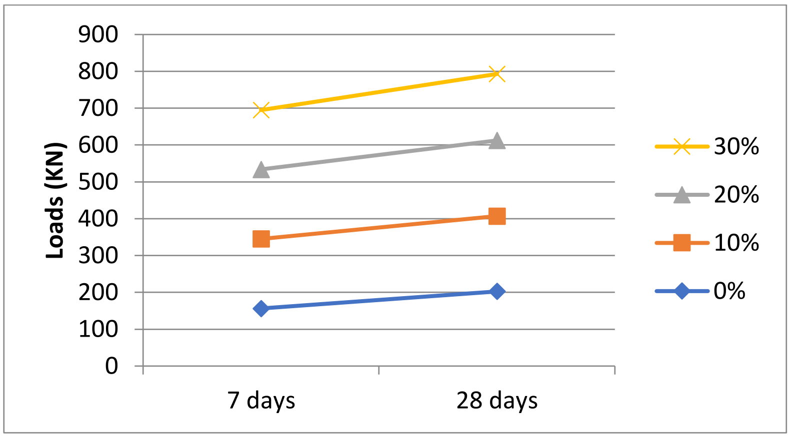

Fig.9 Loads at 7 and 28 days

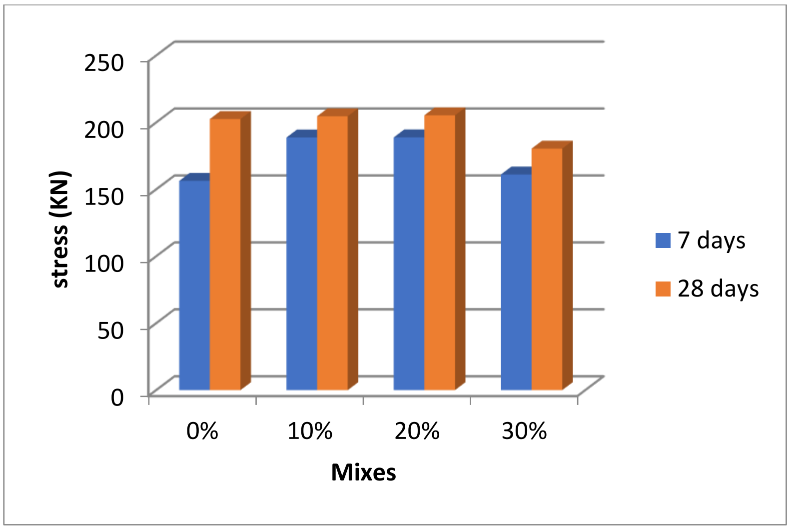

Fig.10 Stress at various mixes

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Water absorption

Table 34 and 35 presented results for water absorption of the mixes after 28 days of curing period.

There was no apparent difference in water absorption as the amount of metakaolin in the concrete mix was increased. As a result, it was discovered that the presence of metakaolin had no adverse impact on water

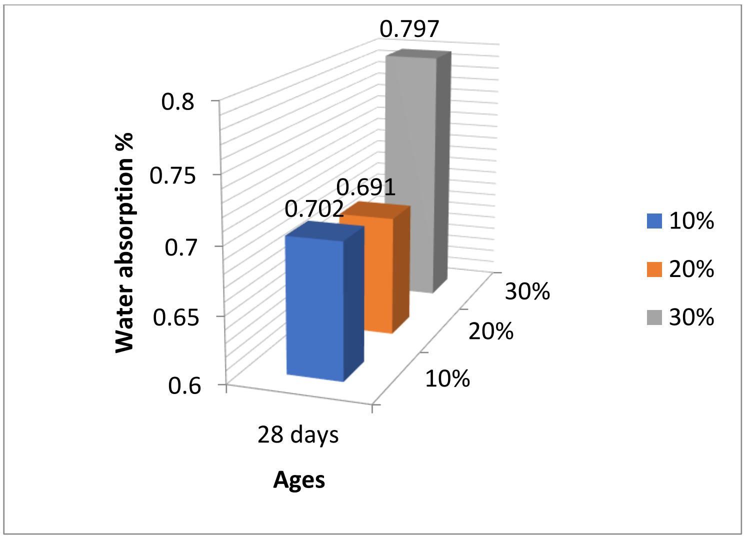

Figure :11 Water absorption Vs ages

Scanning Electron Microscope

In hardened concrete specimens containing MK, SEM methods were used to examine pore microstructure and estimate pore size and distribution. The SEM specimens first broken down into small pieces of 3-5 mm in size, constituted mainly of mortar and fine particles. They were then dried and stored in sealed containers until they were tested using a SEM. SEM analysis of different samples after 28 days of curing.

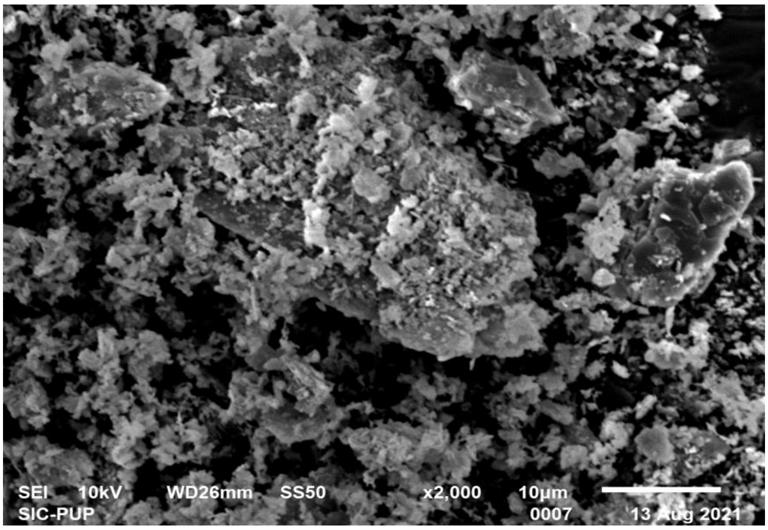

Fig.12 SEM image of Metakaolin

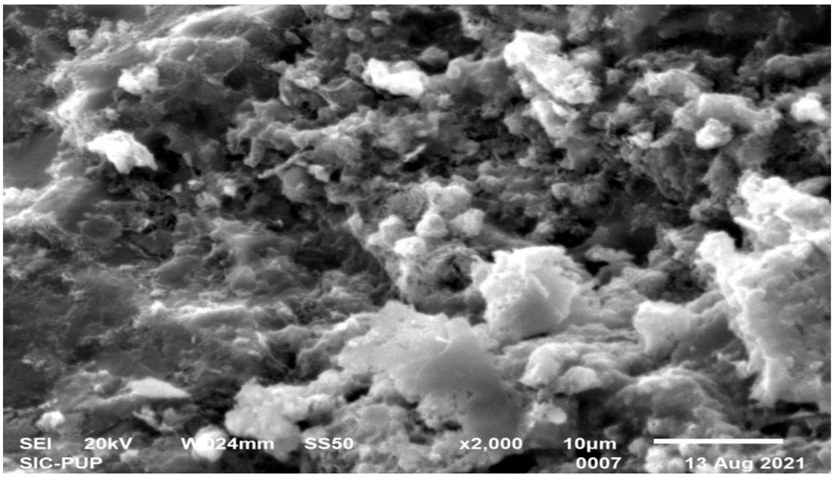

Fig. 13 Control mix SEM image after 28 days

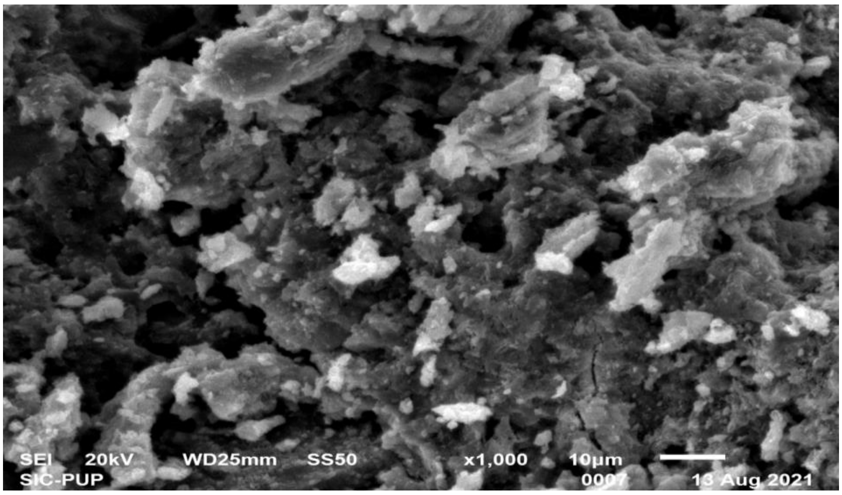

Fig. 14 10% metakaolin SEM image after 28 days

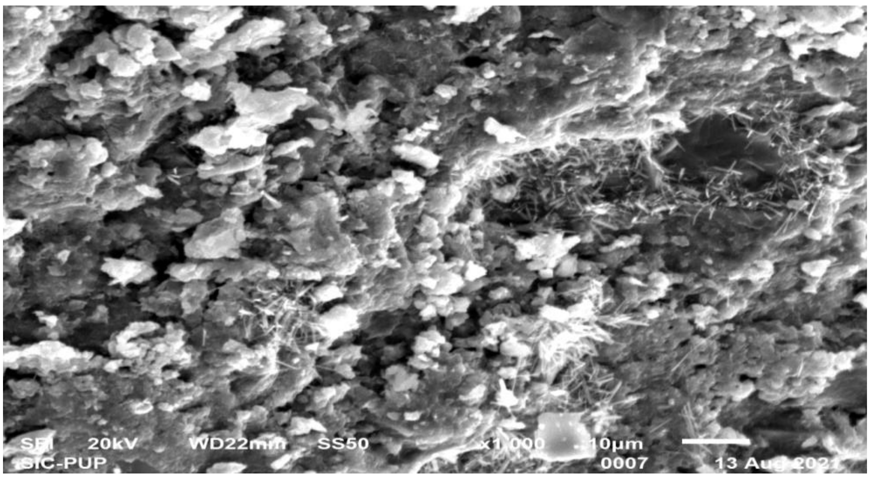

Fig.15 20% metakaolin SEM image after 28 days

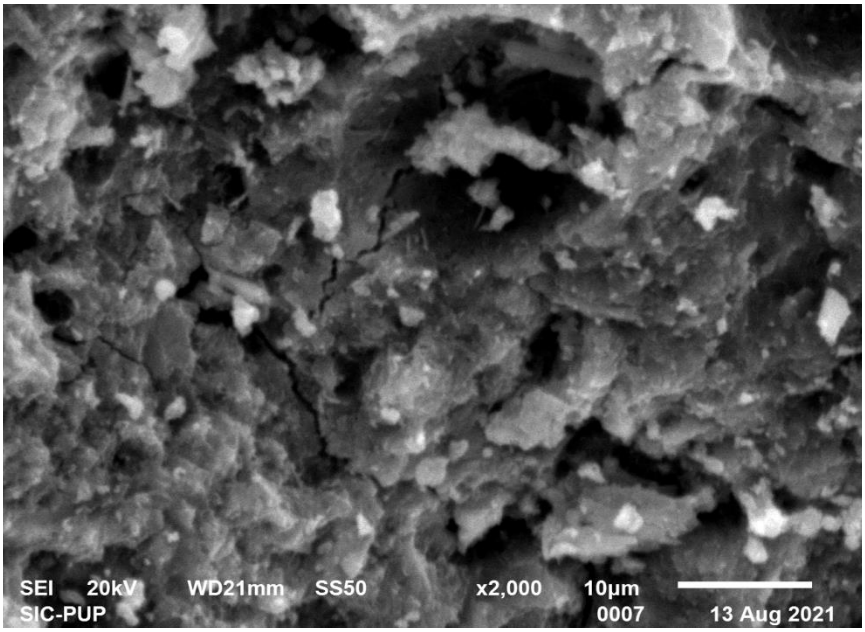

Fig.16 30% metakaolin SEM image after 28 days

Microstructural compounds generated with and without replacement metakaolin were characterised using SEM. The pozzolanic reaction and gel formation are revealed by the study. To directly examine the evolution of cement hydration and pore structure, SEM magnification was adjusted at 1000 and 2000 times. After 28 days of ageing, SEM observation was done on the control specimen, as indicated in the figures above. CH, as well as pore interconnectivity, was found.

At 28 days, SEM observation was conducted to specimens with varying quantities of replacement metakaolin (10%, 20%, and 30%), as shown in figures 19, 20, 21, and 22. Clearly, replacement metakaolin formed a more compact, denser pore structure in cement-based paste specimens. On the surface of M10, M20, and M30 specimens, hydration formed; the microstructure of these samples restricted the mobility of chloride and other ions, resulting in increased compressive strength and reduced crack stretching.

Figure 14 shows the microstructure of M10 specimens, which is rather dense and homogenous. Particles could act as filler, converting big capillary pores into small, discontinuous capillary pores. The compressive strength test results, quantity of efflorescence, and SEM observation all indicated that the M10 and M20 specimens had higher pozzolanic activity. Furthermore, when compared to control specimens, the peak value for M30 specimens dropped considerably, indicating a poor hydration response. This trend is similar to compressive and split tensile strength results, in which the hydration reaction of specimens with more than 20% metakaolin is lower than that of control specimens due to the influence of reduced efflorescence resistance.

Conclusions

- The compressive and tensile strength at 7 – day was found to be maximum when cement was replaced by 20% of MK. It also showed that replacement of cement with metakaolin resulted in lower strength at early ages than Control Mix, according to 7 – day compressive and tensile strength. The strengths of compressive and tensile were found to be 36.78 N/mm2 and 10.68 N/mm^2 respectively.

- At 28 – days, compressive strength of concrete mixes having 20% of MK as supplementary cementitious material was higher than the target strength for M25 grade of concrete.
- Metakaolin with a concentration of more than 20% is not shown to be practical.
- 20% of MK was found to be feasible as replacement of cement by weight.
- The addition of metakaolin had no apparent effect on water absorption.
- SEM analysis was also be conducted and observed that samples with more than 20% metakaolin is lower than that of control specimens.

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