

Microstructural Analysis of Triple Blended High-Performance Concrete Incorporating Ceramic Waste and Metakaolin

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The analysis of micro structural property of triple blended high-performance concrete provides essential information about the internal molecular bonding and Mechanical Properties of the concrete. The mechanical property of the concrete can be improved by setting up porosity, homogeneity, microstructural arrangement of ingredient etc. The micro structural analysis helps to improve the desirable property of high-performance concrete to open the application in Prestressed, Prefabricated concrete member, High Rise buildings, Bridges etc. The disadvantage of high-performance concrete is high cement content which increases the overall cost, to reduce the cost of the concrete without compromising the mechanical properties and durability of the concrete, in this study the waste material metakaolin and ceramic waste is incorporated as a partial replacement of cement. The objective of the present study to assess microstructural assessment using X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Electro Static Discharge (ESD), Interfacial Transition Zone (ITZ) etc. The Outcome of this experimental study shows greater feasibility of using waste material into the concrete mix for not only making the Concrete Cost effective but also environment protective too.

Keywords: High Performance Concrete; Micro Structure, Mechanical Property, Waste Material, Micro analysis.

1. Introduction

The Concrete is the highest consumed material in the field of Construction. The demand of Concrete is increasing day by day due to rapid construction work. Now, the main target goal of Researcher and Scientist is to provide concrete which will not only be cost effective but also can be environment friendly too. The High-Performance Concrete is a emerging new

generation concrete which exhibits High Strength, good durability, sufficient ductility and low maintenance. The mechanical properties of the concrete mainly based on their chemical composition, properties of aggregate, microstructural arrangement, and the bonding between ingredient of concrete. The relationship between microstructure of cement-based product and strength give us the prediction of manipulation of various ingredient to achieve our target. Adding waste material into the concrete becomes very heterogeneous and complex microstructure. In this kind of microstructure, it is very challenging to define the micro level characteristics of new ingredient with the conventional ingredient of concrete. The transition zone of the concrete mixture is the weakest region in the concrete because it is interfacial region between hydrated cement paste and coarse aggregate. In many cases, the properties of interfacial region make a direct impact on concrete strength and durability and it has been also seen in many research studies done in past. The high-performance concrete is applicable for many civil structures like skyscrapers bridges etc. The use of high-performance concrete incorporates with waste material make a directly impact in lowering the construction cost causing a reduction in the cross section of individual structural element. This can be advantageous in terms of allowing the structure for the reduction of consumption of steel reinforcement and long span request. The High-Performance concrete is more durable and stiffer than conventional concrete. The brittleness of HPC is more therefore it is susceptible to brittle failure and cracking. For this experimental study, we have considered two waste material which are by product of Ceramic Industry and China Clay as a replacement of cement. The past studies have shown the advantages and disadvantages of adding this waste material into the concrete mix as a partial replacement of cement in which mainly positive results can be seen if the experimental study is done on trial-and-error based approach. Metakaolin is very fine material which is helpful to fulfill the voids and pores in concrete mixture. Hence, the bonding between particles improves and results into higher strength. The Target strength of this study is taken as 60 MPa for different proportions. Metakaolin is obtained when kaolinite clay is heated at very high temperature ranges 600 °C to 800 °C. The ceramic waste is generally found from the ceramic manufacturing industry and demolition of structure. For reaching to a fruitful conclusion, it is mandatory to have microstructural analysis despite getting good results in mechanical properties experiments. On the microscopic assessment, mechanical characteristic and sorptivity were evaluated. The composition and morphology is mainly taken into consideration while evaluating microlevel study. This study gives many valuable insights by analyzing the influence of waste material on strength, durability, and microstructure of High-Performance concrete.

2. MATERIALS

A. Materials

a. Metakaolin:

It is the supplementary cementitious material which is produced the process of hydroxylation. The clay mineral is heated at very high temperature and results into formation of metakaolin. The particle size of metakaolin is too fine approx. 2.5 µm. This shows very good bonding property and protect the concrete corrosion. This material is added into the concrete mix to enhance the strength and performance.

- Physical Composition:

Material Color: Off White

Specific Gravity: 2.60

Physical form: Powder

Avg. Particle Size: less than 2.5 μm

- Chemical Composition (Major Element): Al_2O_3 : 21.50%

SiO_2 : 57.50%

L.O.I: 16.13%

b. Ceramic Waste:

This waste material is the byproduct of glazes, stains, and clay in which some of the toxic metals like copper, cadmium and chromium are basically incorporated. These wastes are very harmful to earth so it is better to reuse this waste material into some product to avoid environmental impact. This material has potential to use this into concrete mix as a partial replacement of cement.

- Chemical Composition (Major Element):

Al_2O_3 : 13.43%

SiO_2 : 70.00%

L.O.I: 8.37%

c. Portland Cement:

Portland cement is one of the core binding agents of the concrete material. The mechanical and chemical strength of the concrete directly or indirectly depends on the strength of the cement. The cement has a vital role in providing strength as well as performance to the concrete. In this experimental study, OPC 53 grade of cement have been used throughout.

d. Fine and Coarse Aggregate:

The strength and performance of the concrete also depends on the quality of fine and coarse aggregate. The shape and size of the coarse aggregate make a direct impact on the strength and performance of the concrete mixture. The coarse aggregate having size between 9.5 mm to 12.5 mm have been used in this study. Coarse aggregate plays a vital role in the strength of concrete because it covers large volume in the concrete mixture and fine aggregate fulfils the pores and voids in the concrete mixture.

e. Admixture:

The admixture used in this study which is for increasing the strength and performance of the concrete. The admixture namely Perma Plast PC-405 have been used in the study for increasing strength and performance of the concrete. The core benefit of using this admixture apart from strength is additional control over segregation and bleeding for better bonding. The amount of admixture was taken 1.7% by the weight of cement.

3. EXPERIMENTAL DATA

a. Concrete Mix Design with Different Mix Proportions

Description	Mix Design Proportion				
	M-1	M-2	M-3	M-4	M-5
OPC 53 (Kg)	507	430.95	405.6	380.24	354.9
Metakaolin (kg) (Replacement of Cement)	0	25.35 (5%)	50.7 (10%)	76.06 (15%)	101.4 (20%)
Ceramic Waste (kg)	0	50.7 (10% of Cement)			
Fine Aggregate (kg)	802	802	802	802	802
Coarse Aggregate (kg)	965	965	965	965	965
Water (Ltr.)	162	162	162	162	162
Admixture (Ltr.)	4	4	4	4	4

Table 1: Different Mix Proportions with Waste Material

b. Scanning Electron Microscopy (SEM) Analysis

SEM is mostly used analytical technique because of the ease of sample preparation, the information with manifolds diversity can be collected easily. The resolution is so high and associated with High field depth, broad range, and continuous range of magnification. The Scanning electron microscopy of concrete specimen for Mix-4 is shown in Fig. 1. As per the Images, the morphology found normal with equal dispersion of concrete ingredient. The Cement paste is investigated with higher magnifications in which the hydration elements and particles of non-hydrated cement are clearly visible. SEM technique allows the microstructure of concrete to be characterized [1].

i) SEM after 7 days curing on different specimen:

Figure 1 depicts the SEM results of concrete mixture after 7 days of curing. The Image shows that hexagonal bonding of Calcium Hydroxide (CH) and Thin Sheet-like structure of CSH can be observed. Figure 2 and 3 represents the SEM results of Mix-2 and Mix-3 after 7 days curing. The High amount of ettringite formed by reaction between tricalcium aluminate (C_3A) and retarder gypsum. This may cause cracking and disorder in long term. This can happen in case unreacted calcium sulphate remains in the mixture. The figure 4 shows that pores and voids in concrete microstructure were filled almost completely by particles of Waste material metakaolin and ceramic waste. The presence of ettringite is less and bonding between ingredient is high due because of reduction in pores and voids. The figure 5 depicts higher presence of hydrated phase like CSH and ettringite. The higher side of these may directly impact on overall strength and performance of concrete in long term.

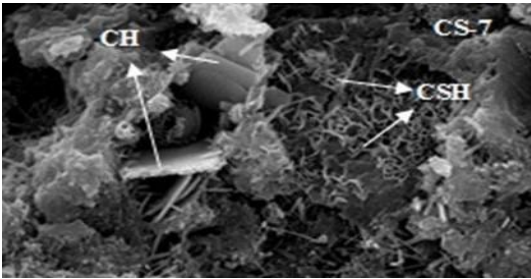


Figure-1 SEM Image for M-1 after 7 days Curing

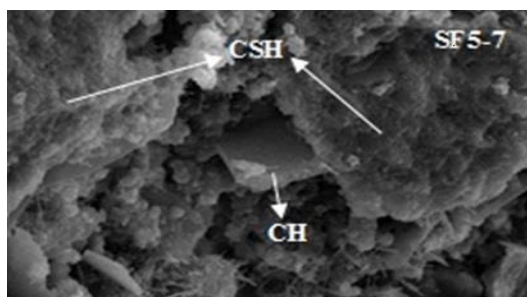


Figure-2 SEM Image for M-2 after 7 days Curing

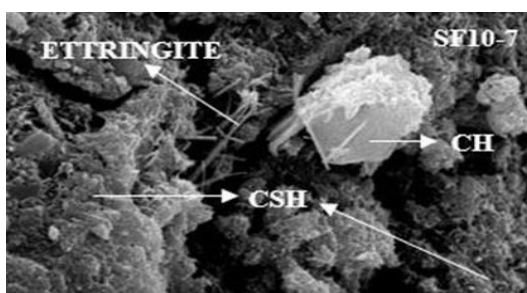


Figure-3 SEM Image for M-3 after 7 days Curing

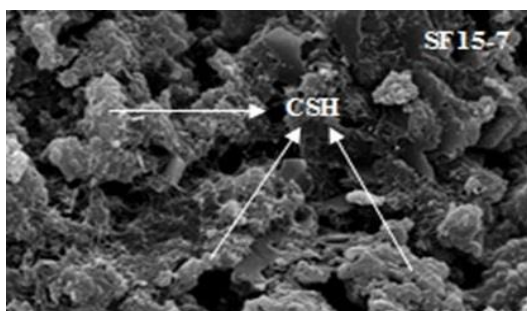


Figure-4 SEM Image for M-4 after 7 days Curing

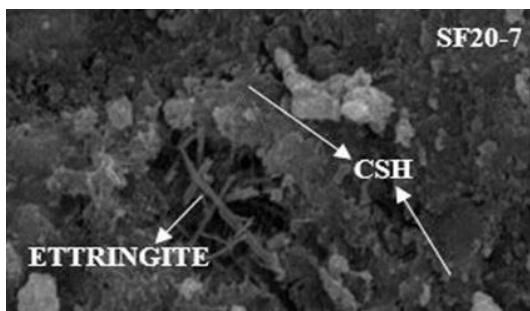


Figure-5 SEM Image for M-5 after 7 days Curing

i) SEM after 28 days curing on different specimen:

Figure 6 represents hexagonal calcium hydroxide present in concrete mixture and thin sheet
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like CSH and needle like ettringite can be observed. Figure 7 and Figure 8 shows the similar results of SEM after 7 days curing. Figure 9 represents the increasing amount of tri calcium silicate and di calcium silicate which is good sign for mechanical strength and durability of concrete. For mix-4, ettringite is less in the images after 28 days curing. Hence, the strength of concrete is on higher side with durability. In the figure-10, there is high amount of ettringite and CSH is available which can affect the strength and durability of the concrete mixture.

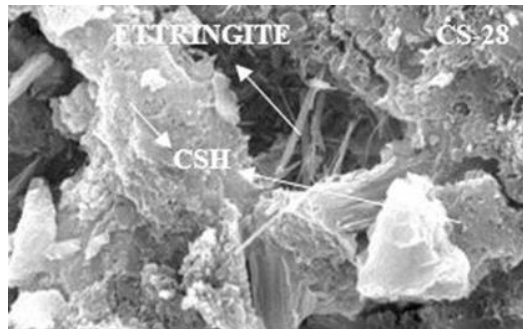


Figure-6 SEM Image for M-1 after 28 days Curing

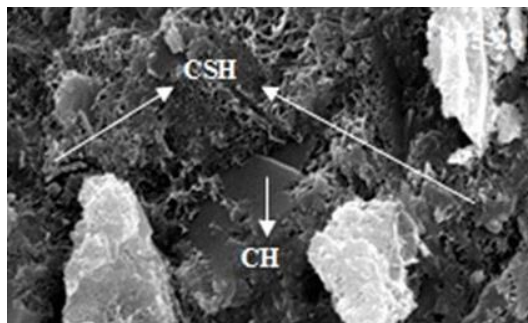


Figure-7 SEM Image for M-2 after 28 days Curing

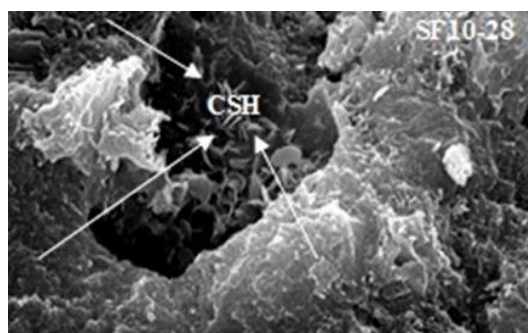


Figure-8 SEM Image for M-3 after 28 days Curing

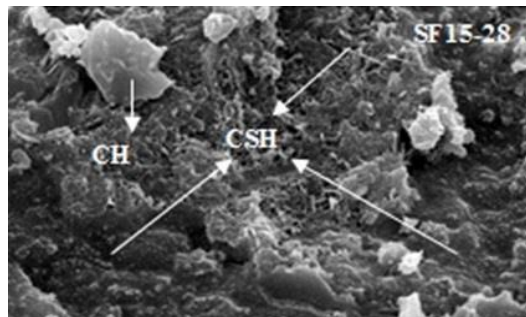


Figure-9 SEM Image for M-4 after 28 days Curing

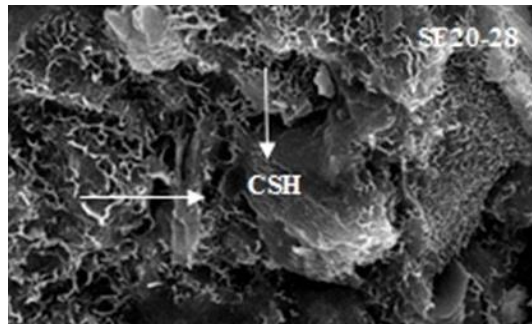


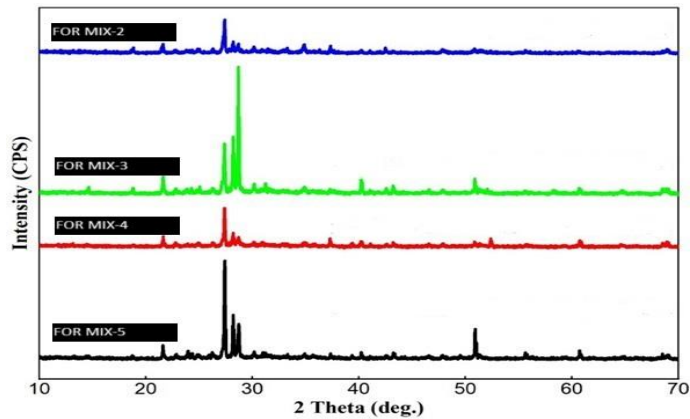
Figure-10 SEM Image for M-5 after 28 days Curing

c. X-Ray Diffraction (XRD) Analysis

X-Ray diffraction method is one of the accurate methods used for qualitative and quantitative analysis of hydrated cement concrete samples. This method is based on Bragg's Law. Using this method, a graph is plotted between angle and Intensity of X-Ray. The angle is taken where the wave gets diffracted and simultaneously intensity of x-ray is determined. XRD analysis was performed on specimen to identify how concrete microstructure affects after adding metakaolin and ceramic waste material as a partial replacement of cement. The Test was done after 28 days curing. The primary peaks were due to presence Silicon dioxide (SiO_2), Calcium Carbonate (CaCO_3) and Calcium Hydroxide $\text{Ca}(\text{OH})_2$. The results clearly stated that adding metakaolin and Ceramic waste had no discernible effect on Calcium Carbonate or Calcium Hydroxide. As per the observations, the following are the causes of the peaks that could be found at various angles:

- 27° and 31° are caused due to tricalcium silicates (C_3S).
- 55° are caused due to Calcium Oxide (CaO).
- 28° are caused by Calcium Hydroxide ($\text{Ca}(\text{OH})_2$).
- 48° are caused due to dicalcium Silicate (C_2S)
- 22° are caused by Calcium Sulphate (CaSO_4)
- 24° and 28° caused due to Calcium Carbonate (CaCO_3)

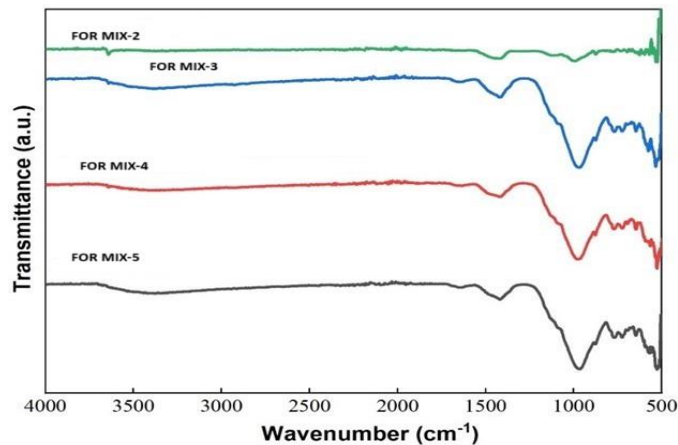
- 22° and 25° are caused by Magnesium Oxide (MgO)



Graph-1 X-Ray Diffraction Observations

d. Fourier-Transform Infrared Spectroscopy (FTIR)

The FTIR analysis is conducted to determine the functional group of the products that are generated during the process of hydration. Graph-2 shows the FTIR spectra on different samples tested after 28 days of hydration in the mode of transmittance. During the experiments, the identical infrared bands have been seen in the all samples with consistent wave number value and varying intensities. The Peak at 3640 cm^{-1} found due to the stretching O-H bond in $\text{Ca}(\text{OH})_2$. The Value of 1665 cm^{-1} is found due to V2 bending of the bound water and absorbed water. The Peak at 1425 cm^{-1} is occurred by V3 stretching of C-O bond in Calcium Carbonate. The peak at 1455 cm^{-1} is occurred by Si-O stretching of CSH. The Peak at 730 cm^{-1} is occurred by the V2 stretching of C-O in CaCO_3 . In this graph, the two peaks are very important for evaluating the hydration process of cement phase. It is clearly seen that 3450 cm^{-1} and 965 cm^{-1} exhibits high intensities in Ordinary Portland Cement compared to other samples.



Graph-2 FTIR Observations on different Mixes

4. CONCLUSIONS

Based on above observations and results, following conclusions can be given:

- The Use of Metakaolin and Ceramic waste can enhance the mechanical strength and performance of concrete. The increase in strength is attributed to the production of additional CSH due to the reaction of Metakaolin with Calcium Hydroxide.
- SEM images clearly showed uniform dispersion and interlocking of ingredient of concrete with the selected industrial waste material Metakaolin and ceramic waste.
- The SEM pictures show a continuous matrix which means a dense microstructure with homogenous matrix of ingredients.
- In XRD analysis, all the peaks have been observed. The hydration of cement product and incorporated waste material enhances at curing age of 28 day.
- The Overall minerology of internal ingredient found extremely locked with less voids and pores by adding waste material
- The use of industrial waste in the concrete mixture will lead to green concrete results into decrease in greenhouse gases and helped to improve strength and durability.
- The use of waste material in the concrete mixture will have directly impact on economic factor.

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