Analysis of Microstructural Behaviour of Rice Husk Ash Blended Cement Mortar

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Abstract- *The stated work investigates the microstructural properties of Rice Husk Ash and the viability of replacing cement with it. Cement was replaced by Rice Husk Ash (0–25 percent at 5% intervals) to investigate its impact on cement mortar's mechanical and physical characteristics, including the evaluation of the microstructure. Samples used were tested after 3, 7, and 28 days of curing. It was noted that when a sample has a consistent water content, it produces a dry mix when more than 10% of the cement was replaced by RHA. owing to dry mix the mortars attempt to hydrate itself was unsuccessful, and the strength starts declining as the Rice Husk Ash percentage rises.*

It was also noticed that consistency of cement was continuously increasing with increase in percentage of Rice Husk Ash when samples were prepared for checking consistency of cement at varied quantities of Rice Husk Ash. The compressive strength was seen to increase up to a 15% Rice Husk Ash, and results above this percentage were also superior to those of the similar samples with constant consistency.

Peaks in the Rice Husk Ash 's XRD pattern revealed quartzite and coesite minerals, both of which have crystalline natures that signify silica concentration. The weight loss percentage of Rice Husk Ash was evaluated using thermal gravimetric analysis (TGA), and the phase transition was examined using differential scanning calorimetry (DSC) between 600 and 1000 C. As the Rice Husk Ash mixed cement mortar cured up to 3,7 and 28 days, the microstructures were also examined using SEM and EDS analysis.

Keywords: XRD-X ray diffraction, TGA-Thermal Gravimetric analysis, DSC-Differential scanning calorimetry.

1. INTRODUCTION

Any nation's economic development depends heavily on cement, and India's cement sector is the world's second-largest producer of cement after China. India uses 195 kg of cement per person annually. Currently, India's installed cement capacity is 500 million tons per annum, producing 298 million tons of cement annually. India's cement production is anticipated to reach 500 million tons by 2021 and 800 million tons by 2030 due to the country's expanding infrastructure.

Ash from the rice husk industry is a waste by-product. When utilized as fuel in boilers, rice husk produces Rice Husk Ash as a by-product. In India, it is widely available. Rice Husk Ash has been discovered to have a significant level of reactivity toward cement and lime. In the study, it was discovered that Rice Husk Ash maintained if not improved the mechanical and durability qualities of concrete and mortar made of blended cement. As a result, it can be utilized as a substitute material in concrete and mortar as partial cement replacement for the sustainable development.

The use of Rice Husk Ash as an additional cementitious material or non-reactive filler might lessen the adverse effects on the environment (such as air pollution and soil pollution). Due to CO_2 emissions, cement production severely harms the environment. Pozzolanic materials can be used in place of cement to lessen the environmental impact. Because pozzolanic material lacks cementing properties and reacts in the presence of free lime and moisture to generate C-S-H gel, a complete substitution of cement by Rice Husk Ash is not feasible. Using RICE HUSK ASH as a mineral additive, 10 to 15 percent of the cement might be replaced. The solid mixture's packing state has a major impact on the physical qualities, Surface area, morphology, particle size, and specific gravity are the main variables linked to the physical feature of Rice Husk Ash. The amount of amorphous silica, which combines with free lime [$Ca(OH)_2$] in the presence of water during hydration, has an impact on the chemical character.

The observation of the calcium silicates hydrate phase is of primary concern. The calcium silicate hydrate mineral structure changes weak crystal from fibres to a net-like structure. According to previous research on the use of fly ash in cement mortar, the mechanical strength increased when cement was replaced by fly ash up to 20% and declined once this limit of cement-fly ash mortar was reached (after curing for up to 28 days). Additionally, they had examined the XRD patterns of several fly ash-cement mixtures that had been cured for up to 28 days, and they had determined the phases of $Ca(OH)_2$, $CaCO_3$, $Ca_2Al(OH)_2xH_2O$, Ca_2SiO_4, ettringite, and SiO_2.

The need of partial replacement of cement aroused due to various factors like

- With increase in the rate of construction and pricing The high cost of cement used as binder has led to a search for alternative.
- In order to reduce the depletion of natural limestone deposits used up for manufacturing cement.
- To lower the energy demand and emission of CO2 responsible for global warming related to cement usage.
- In order to ensure a safe and effective solution to deal with industrial and agro waste byproducts such as fly ash, rice husk ash, sugarcane bagasse.

It was found that some of these waste products contain amorphous silica and hence act as pozzolanic materials which enhance properties of concrete and mortar. Fly ash has been used as a pozzolanic material in most research projects, according to the literature. There was little activity in using Rice Husk Ash for construction. Microstructural characterization of Rice Husk Ash has received little attention, and the cause of strength increases, and decreases are not well understood.

As a result, an effort has been made to research on microstructural characterization of Rice Husk Ash, and Rice Husk Ash-blended cement mortar. The analysis is crucial in understanding Rice Husk Ash's characteristics as well as how this waste might be applied to construction projects for sustainable development.

2. MATERIALS AND METHODOLOGY

2.1 Materials used and their properties

Cement acts as the binding material in concrete used in construction, the basic property of cement is that it first sets and then ultimately hardens and adheres to the other materials binding all of them together. The cement from a single batch used throughout for this study is Ordinary Portland Cement 43 Grade selling under the name CONCRECEM, The Manufacture of the cement is Birla Corporation Limited (M.P Birla Group) and Production Unit Is Satna Cement Works, Satna, M.P.

Water imparts workability when the mortar is in paste form and then it is required for the process of hydration which ultimately leads to strength development. To create a robust and usable cement mortar, water is mixed to cement and sand. The hydration reaction of cement cannot occur without water. Additionally, water must be added to create a paste that may be used on both overhead and vertical surfaces. The excessive workability brought on by too much water will result in the mortar dripping off in addition to the mortar losing strength. The best way to determine the water to cement ratio primarily depends on conditions existing at site.

Sand is used as a filler material in the mortar and comprises maximum percentage of volume of mortar, Sand is not typically added to mortar to boost strength. Instead, it is primarily utilized as an inert substance to enhance the amount of mortar for economic reasons. Sand can be used in the proper quantity to create inexpensive mortar without compromising mortar strength. The fundamental idea behind using all cementing ingredients is that sand splits the paste of the cementing components into a thin film. Sand provides the necessary amount of surface area for the cementing material film to adhere and spread, also it aids in keeping mortar from shrinking. Additionally, it stops mortar from breaking when setting. The density of mortar is increased by using well- graded sand. Additionally, it is asserted that a chemical reaction occurs to create a hardened mass between the components of the cementing ingredients and the silica (SiO2) found in sand grains.

In the investigation for preparing cement mortar sand used was standard river sand having specific gravity 2.7 and moisture content 1.4%, respectively. Rice husk ash is an agro based waste by-product which is collected from boiler feeds and is available abundantly in India, it constitutes of high amount of silica and behaves as pozzolanic material hence can be used as a material for partially replacing cement as supplementary cementitious material or non-reactive filler in mortar to decrease the cost of production as well as maintain or increase the mechanical or durability properties of mortar up to some extent.

The rice husk ash used in this investigation was bought from Baba Traders, a Rice Husk Ash manufacturer and supplier situated in Kolkata, West Bengal. The manufacturers quoted that the supplied rice husk ash was generated by burning rice husk at a controlled temperature from 700°C to 900°C and sieved through a 90-micron sieve to remove coarser unburnt remains of carbon particle.

The specific gravity of rice husk ash was determined using Le-Chatelier flask as apparatus and kerosene oil as medium having density 0.71gm/cc according to IS 1727:2004 and the specific gravity was found to be 2.1.

2.2 Microstructural testing methods.

In this thesis microstructural characteristics of Rice Husk Ash and accordingly the feasibility of partial replacement of cement with Rice Husk Ash in mortar has been investigated, also effect of this replacement on mechanical and physical properties of cement mortar has also been studied. Various types of testing and analysis have been performed on cement, rice husk ash, grinded mortar to know microstructural behavior and elemental composition like-

1) Thermal Gravimetric Analysis to observe weight change variation with temperature of rice husk and ordinary Portland cement.
2) Differential Scanning Calorimetry to observe the chemical change with temperature of Rice Husk Ash and Ordinary Portland Cement.
3) X-Ray Fluorescence analysis to determine the elemental composition of incinerated rice husk ash.
4) X-Ray Diffraction technique to determine the chemical composition of Rice Husk Ash and Ordinary Portland Cement.
5) Scanning Electron Microscopy and Energy-Dispersive Spectroscopy for analyzing the chemical composition and microstructure of finely grinded blended cement mortar.

There are various analysis software present in the market for refinement and analysis of these raw data files. For this investigation the software used is X'Pert High Score Plus which provides various features and has a huge elemental database for phase identification. To get refined results, determine phase and obtain chemical composition information a specific set of procedures need to be performed in the respective software-

1) Background Correction- To see the XRD signal in a two-dimensional (2D) XRD measurement, the background must be eliminated, which is made up of scattering from the apparatus and the main X-ray beam rather than the sample.
2) Stripping K alpha 2- X-ray diffraction requires monochromatic X-rays, as Bragg's law makes clear. Copper K-alpha, which is more intense than K-beta and provides superior resolution, is employed in the procedure because it is used to identify and examine material structures. The composite link model is used to extend the model and remove the K doublet. The data are consequently split into two smooth components, K-1 and K-2. The applied penalty's weight is automatically tailored for both smoothing and K2 removal.
3) Identifying the peaks-The peak intensities in an XRD experiment will vary since intensity is proportional to the number of scatterers per unit area of a certain atomic plane. Typically, the peak's strength decreases with increasing plane indices (greater angles in the pattern). We must examine the nature of the Bragg's peaks that emerge in the XRD pattern in order to use it to determine the nature of the materials. If the peak is quite broad, the substance is amorphous with short range ordering. The material is crystalline if the XRD pattern shows sharp peaks.
4) Executing Search and Match- To determine phase and obtain chemical composition information.

3. RESULTS AND DISCUSSION

For investigating the microstructural behavior of rice husk ash blended cement mortar and determining optimum cement replacement by rice husk ash in respect to strength and durability several tests had been conducted whose results are as represented below

3.1 Effect of Rice Husk Ash replacement on standard consistency

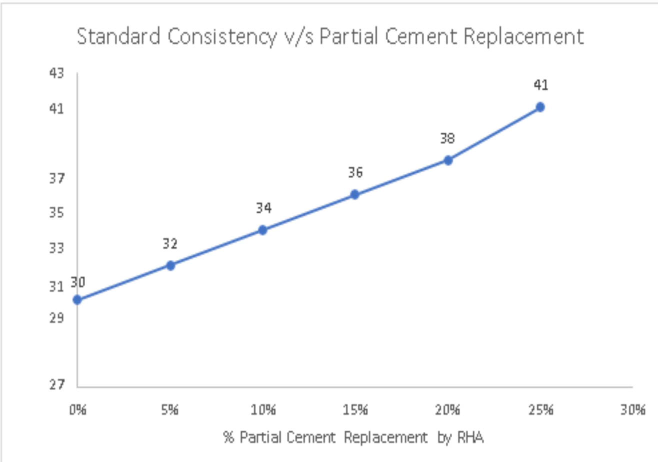

Chart -1: Graph for Standard Consistency v/s Partial Cement replacement by RHA

On performing the test to obtain the standard consistency corresponding to different partial replacements of cement by pozzolanic material Rice Husk Ash, the following results were obtained. The graph illustrates initially a nearly linear increase in the standard consistency as we increase the percentage replacement of Rice Husk Ash and also it indicates that at higher replacements the rate of increase of standard consistency also increases.

This above observation indicates towards the hygroscopic nature of the rice husk ash by the virtue of which it absorbs more water in comparison to the cement hence increase the water demand, this can be due to high specific surface of Rice Husk Ash in comparison to cement and should be kept in mind while determining water requirement for cement mortar in field.

3.2 Compressive strength test results

While performing compressive strength testing the samples were divided into two groups on the basis of their standard consistency-

Group 1- (C group) In this group samples the standard consistency was assumed as constant and equal to the standard consistency of unblended Ordinary Portland Cement (30%) while calculating the water requirement for compressive strength testing irrespective of the partial replacement of cement by Rice Husk Ash.

Group 2- (V Group) In this group samples the standard consistency corresponding to each partial replacement was first calculated and the water requirement for compressive strength testing was calculated accordingly for each partial replacement.

Total 18 cube specimens were made for each case of partial replacement out of which 9 cubes were made according to C Group and the other 9 were made according to V group, out of these 9 specimens 3 specimens were tested after 72 hours for calculating 3 days Strength, the next 3 specimens were tested after 168 hours for calculating 7 days strength and the remaining 3 specimens were tested after 672 hours for calculating 28 days strength.

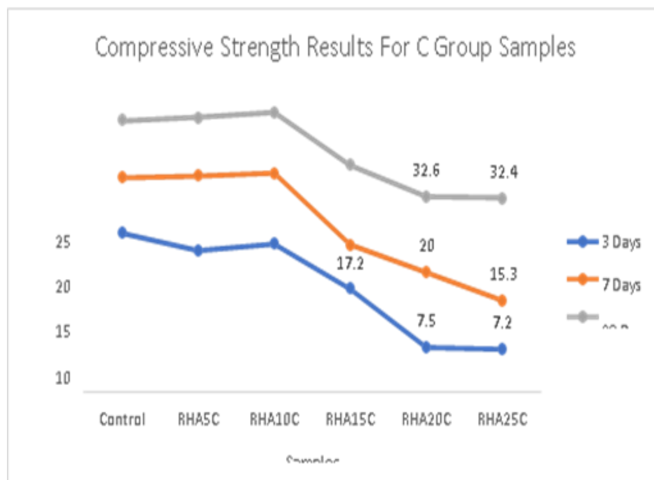

Chart -2: Graph for Compressive Strength Results for C Group Samples

On analyzing the Compressive strength results it was observed that the 3 days compressive strength of blended cement mortar irrespective of partial replacement percentage is always lesser than the 3 days compressive strength of control sample, this observation indicates toward the slow nature of pozzolanic reaction that is occurring in blended cement mortar and is responsible for late strength development of compressive strength.

Further on investigating it was observed that the 28 days compressive strength is increasing on increasing the percentage partial replacement up to partial replacement of 10% after which it shows a down fall pointing towards the conclusion that optimum level of partial replacement in the case of constant standard consistency is 10% to achieve highest compressive strength.

On analyzing the Compressive strength results it was observed that the 3 days compressive strength of blended cement mortar irrespective of partial replacement percentage is always lesser than the 3 days compressive strength of control sample, this observation again indicates toward the slow nature of pozzolanic reaction that is occurring in blended cement mortar and is responsible for late strength development of compressive strength.

Further on investigating it was observed that the 28 days compressive strength is increasing on increasing the percentage partial replacement up to partial replacement of 15% after which it shows a down fall pointing towards the conclusion that optimum level of partial replacement in the case of variable standard consistency is 15% to achieve highest compressive strength.

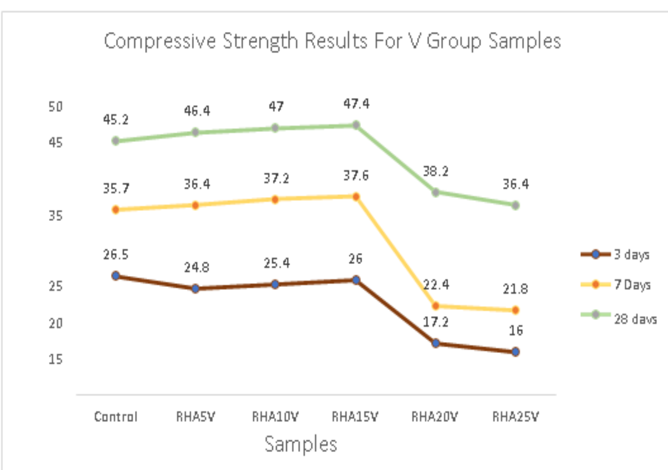

Chart -3 Graph for Compressive Strength Results for V Group Samples

3.3 Compressive strength test results comparison of V group specimens and C group specimens.

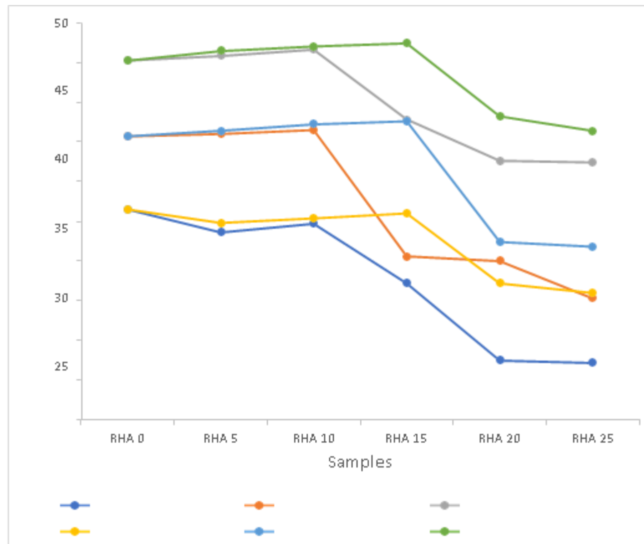

Chart -4: Graph for Compressive strength test results comparison for V group samples and C group samples

On comparing the results of compressive strength tests corresponding to both groups it was observed that the maximum 28 days compressive strength for group C samples having constant standard consistency was obtained for 10% replacement and had a value of 46.6 MPa whereas the maximum 28 days compressive strength for group V samples having variable standard consistency was obtained for 15% replacement and had a value of 47.4 MPa and after it in both cases there was a downfall in compressive strengths with respect to control sample, The main cause can be that there wasn't enough water for hydration. At replacement levels of 20 and 25 percent, the materials had very little bonding, which made them less workable.

In addition to this, all samples with variable water content produced higher compressive strength results than equivalent samples made with constant water content. It can be since there is enough water in the mixtures to finish hydration of the cementitious paste. The production of extra C-S-H gel because of the reaction between free lime present in cement paste and active silica present in Rice Husk Ash is another factor contributing to the increased strength gain.

3.4 XRD analysis results

Samples of Ordinary Portland Cement and Rice Husk Ash were investigated by XRD technique in order to determine their chemical composition and raw data files were obtained, Then the results obtained from XRD machine in the form of raw data images were refined in X'Pert High Score Plus Software and plotted in graphical form in OriginPro software.

To analyze the chemical structure of materials, XRD Patterns of OPC were plotted. The pointed peaks of graph indicate towards the crystalline nature of OPC. The main peaks of Tricalcium Silicate C3S (Ca3SiO5) Dicalcium Silicate C2S (Ca2Sio4) and Silica (SiO2) were observed, along with it peaks of Gypsum (CaSO4), Magnesia (MgO) were also observed.

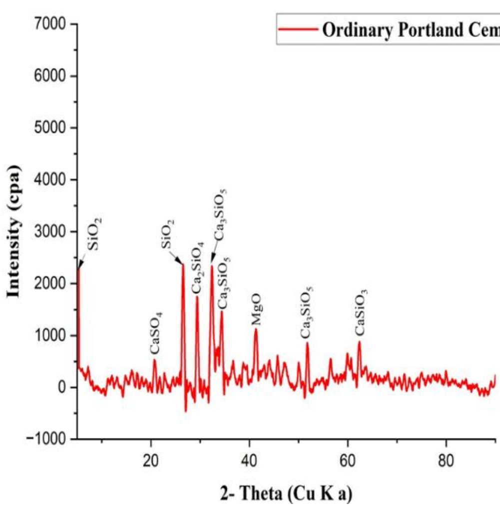

Chart -5: Refined XRD analysis graph for Ordinary Portland cement (OPC)

To analyze the chemical structure of materials, XRD Patterns of Rice Husk Ash were plotted. The pointed peaks of graph indicate towards the crystalline nature of Rice Husk Ash. The main peak of Silica (SiO2) was observed showing that intensity of silica is very high in comparison to other constituents, along with it peaks of Lime (CaO), Magnesia (MgO), Alumina (Al2O3) were also observed. The silica was observed at different diffracted angles and was mainly observed in the forms of coesite, quartz, and tridymite mineral.

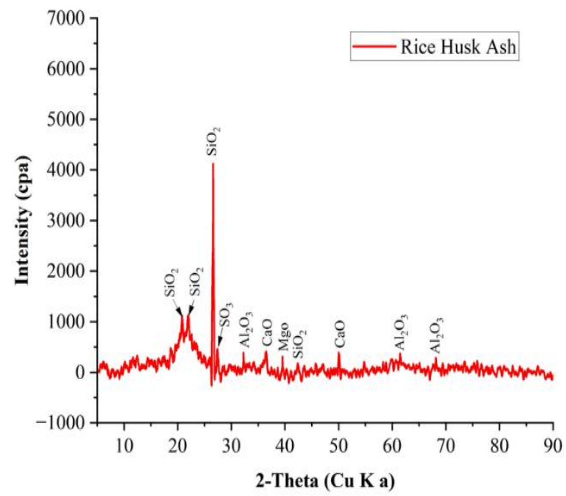

Chart -6: Refined XRD analysis graph for Rice Husk Ash (RHA)

3.5 TGA-DSC Analysis Results

In the Fig. 7 below, the rice husk thermograms are displayed. Three peaks that can be identified as stages in the decomposition are demonstrated. An exothermic shift occurs after a brief endothermic curve (first peak) at 200°C. At 500°C, the second peak shifts from exothermic to endothermic. 650°C marks the third peak, which was followed by an exothermic shift.

The first weight loss of 10% below 110°C and the subsequent rapid weight loss of 56% that began above 400°C. The weight loss exceeds 83 percent at 900°C. The first peak is followed by a 5% weight loss, which is seen to represent absorbed water loss. The degradation of cellulose is indicated by the second peak, which is exothermic and i followed by fast weight loss. The breakdown of cellulose and lignin occur at the same time. At the third peak, the weight of rice husk continued to decrease, which was due to the continuous degradation of lignin and another organic component.

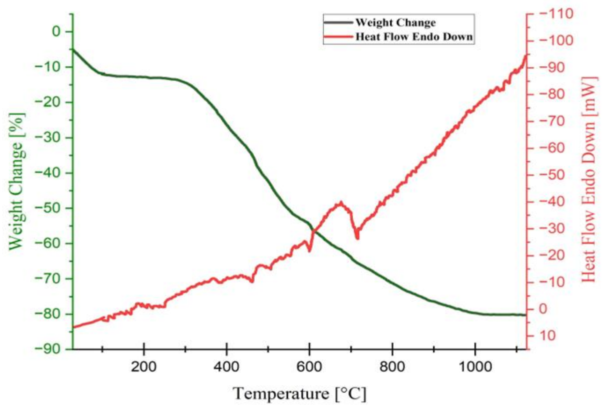

Chart -7: TGA-DSC Curve of Rice Husk

4. CONCLUSION

After detailed investigation following conclusions are drawn from this study-

In the calcination of Rice Husk in order to obtain the rice husk Ash, silica content was increased from 28.12% to 92.30% as noted by X-Ray Florescence Technique.

A study of Rice Husk Ash's morphology was also conducted and it was found to be porous and soft in nature. The main peaks as obtained in X-Ray Diffraction analysis were SiO_2, Al_2O_3 and CaO.

Ca, Si and O were noted as the primary components of OPC, whereas Si and O are the primary components of Rice Husk Ash in EDS analysis.

On performing Thermo Gravimetric analysis of Rice Husk from 200 to 350°C, a sudden decrease in weight change percentage was seen, and from 350°C to 400°C, it was practically linear. Between 600 and 800°C, exothermic to endothermic transitions were seen in the Differential Scanning Calorimetry.

The maximum compressive strength was discovered at 10% substitution with mortar made from Rice Husk Ash mix with the same consistency in contrast to 15% replacement level with mortar made from Rice Husk Ash mix in the various consistency blends. All blended mixes of different consistency had greater compressive strength than the corresponding blended mixes made with constant water content. As Rice Husk Ash level rose in blended mixtures, EDS analysis revealed decreasing Ca peaks and increasing silica content peaks.

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BIOGRAPHIES

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