

Performance and Micro Structural Analysis of Portland Slag Cement Mortar Induced with Pozzolanic Additives



P.Raja Priya, V.Kannan

Abstract: Portland cement is a kind of cement used where the high strength and durability is needed. Also, this type of cement is essentially used to control the CO₂ emission during the manufacturing process of the concrete. This cement is made up of slag with the activator such as alkalis in the form of sodium hydroxide or sodium silicate. However, this addition is increasing the overall cost of the production of concrete. In this research, a new attempt has been made to use the natural activators of Rice Husk Ash (RHA) and Natural Steatite Powder (NSP). This research aims to determine the effects of RHA and NSP with Portland slag cement by partial replacement with 5%, 10%, 15% and 20% of RHA and NSP. The influence of the RHA and NSP on the mechanical properties of the mortar was evaluated by measuring the compressive strength and the split tensile strength. The durability properties of the specimens were analyzed by water absorption, sorptivity and acid attack tests. The analysis of the microstructure of the specimens was done by scanning electron microscope (SEM) and Fourier Transform Infrared Spectra analysis (FTIR). It was observed that the maximum compressive strength and split tensile strength was in 5% RHA and NSP blended mortar. The durability results showed that the 10% RHA and 10% NSP had lesser water absorption and sorptivity values. From the results of micro structural analysis it was observed that replacing cement with 5% RHA and 5% NSP results in improvement of microstructure of cement mortar.

Keywords: Mechanical properties, Microstructure analysis, Natural steatite powder, Portland slag cement mortar, Rice husk ash.

I. INTRODUCTION

Mortars prepared from slag-cement blends exceed or equal the strength of ordinary Portland cement mortar at 7 to 28 days [1]. The replacement of fine aggregate with iron slag enhanced the compressive strength and split tensile strength. The improvement in the compressive strength and split tensile strength of the mix with the incorporation of iron slag is primarily due to the pozzolanic reaction and slag being finer than sand, results in particle packing behaviour. The mortars with the replacement of fine aggregate with iron slag have

better sulphate resistance due to low C₃A content. The microstructure is also improved due to reduced permeability [2]. The slag addition in cement contributes 70% compressive and flexural strength improvement in later age [3]. Use of slag reduces the amount of cement content as well as heat of hydration in a mortar mix. As the heat of hydration is less the use of high volume slag as a replacement of cement, provides lower impact on environment. Thus, the construction work with slag concrete becomes economical and also makes a sustainable concrete [4]. Since the heat release of slag reaction is slow and low compared to that of Portland cement, the risk of cracking in massive concrete can be minimized, which in turn improves the durability of concrete. Concrete made from slag cement shows denser microstructure [5].

Rice husk ash contains about 10-20% ash. The ash has 60-97% silica, highly porous, light weight with very high surface area [6]. The silica in the rice husk ash provides excellent thermal insulation. The mechanical properties in terms of flexural and tensile strength have been significantly improved with the addition of RHA. When RHA added to the cement concrete, it reduces the weight of concrete up to 15% after 90 days curing. Due to high specific surface of RHA the dosage of superplasticizer had to be increased along with RHA fineness to determine the desired workability [7].

The compressive strength and flexural strength of pervious concrete is increases up to 10% replacement of the cement with RHA [8]. Pozzolanic reaction of RHA depends on particle size, age and cement replacement percentages. Pozzolanic reaction of RHA blended mortar is higher than OPC mortar after 90 days of curing. Flexural strength, water absorption and porosity performance of mortar containing small RHA are significant due to higher degree of pozzolanic activity. The microstructure of the RHA mortars is denser than that of the control mortar [9]. Water required RHA blended cement mortar is more than the water required for conventional mortar [10]. The replacement of ultrafine natural steatite powder with cement reduces initial setting time and final setting time and improves the mortar compressive strength. The UFNSP replacement percentage should not exceed 20% [11]. The mix with 5% Rice husk ash, 5% metakaolin and 5% natural steatite powder gave the best improvement in the strength of concrete specimens [13]

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This research work was conducted by replacing Portland slag cement with various percentages of RHA and NSP and the properties and mechanical behaviour were discussed. Furthermore the microstructural analysis was also examined.

II. MATERIALS AND PROPORTIONING

A. Cement

Slag cement is manufactured as per BIS specification IS 455 1989 and the quantity of slag to be added shall be in the range of 25% to 70%. Tests were carried out on various physical properties of cement and the results are shown in Table 1.

Table-I: Properties of Cement

Grade	PSC
Consistency	32%
Initial Setting Time	180 minutes
Final Setting Time	240 minutes
Specific gravity	2.87

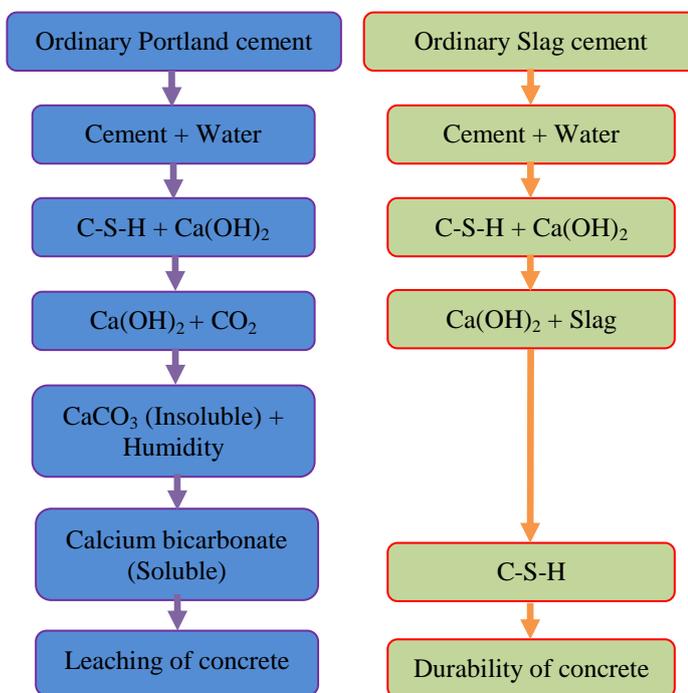


Fig.1. Cement hydration mechanism

B. Rice Husk Ash

RHA was collected from an agro industry. The collected RHA was sieved through 300micron sieve and the size was further reduced to less than 45micron sieve by grinding it in ball mill.

Table-II: Properties of RHA

Colour	Grey
Shape	Irregular
Particle size	<45micron
Appearance	Fine powder
Specific gravity	2.3

C. Natural Steatite Powder

Steatite is a type of metamorphic rock which is largely composed of mineral talc and is thus rich in magnesium. Natural steatite powder has the advantages of reduced initial and final setting time and consequently increases the compressive strength. The specific gravity of NSP is 2.70

Table-III: Proportion of NSP

Composition	Natural steatite powder
SiO ₂	62.67%
CaO	0.2%
Al ₂ O ₃	0.24%
Fe ₂ O ₃	0.3%
MgO	33.26%
L.O.I	3.33%

D. Fine Aggregate

Natural river sand was used as fine aggregate conforming to EN 12620-2002. The results obtained from sieve analysis indicate that the sand conforms to Zone III. The fineness modulus of sand was 3.5. The specific gravity was 2.67.

E. Water

Portable water was used for entire work.

F. Super plasticizer

The superplasticizer, ESP 400 was used to increase the workability. It is a brown liquid with specific gravity of 1.18. The dosage of super plasticizer was constant at +1% of weight of cement throughout the mix.

G. Mix proportion

The mortars were prepared with the mix proportion of 1:3. The W/B ratio was taken as 0.43 based on the consistency of cement. The RHA and NSP were replaced with varying percentage as shown in Table 4.

Table-IV: Program of mix proportion.

Designation	Cement (g)	NSP (g)	RHA (g)	Fine Aggregate (g)	W/B ratio
M1	80	-	-	240	0.43
M2	72	4	4	240	0.43
M3	64	8	8	240	0.43
M4	56	12	12	40	0.43
M5	48	16	16	240	0.43

III. METHODOLOGY

A. Casting and Curing

After proper mixing, the mortar was filled in the moulds of size 50 mm x 50 mm x 50 mm for compression testing and cylinders of 50 mm diameter and 100 mm length for split tensile test after proper oiling. Then machine vibration was given for 5 minutes.



The mortar was allowed to be in the mould for a period of 24 hours at a temperature of $27 \pm 2^\circ\text{C}$ and 90% relative humidity and demoulded. After demoulding the specimens were subjected to water curing for about 28 days.



Fig.2. Specimens after casting

B. Mechanical Properties

The compressive strength test was carried out on cubical specimens of 50 mm size. The cubes were tested as per IS: 516-1979. The tests were done on compression-testing machine by placing the specimen at the centre of the testing machine and the load is applied on axially opposite faces at the rate of 140 MPa / minute. Cylindrical test specimens 50 mm diameter and 100 mm long were casted. The specimens were tested as per IS 5816-1999. For each mixes 3 specimens were casted and the average of the mixes is taken for the calculation.

C. Water absorption

The 50 mm x 50 mm x 50 mm size cube after casting has been immersed in water for 28 days curing. The test was carried out as per IS 1124-1974. To determine the water absorption the cubes were dried in oven at a temperature of 85°C for 24 hours and their initial weights were noted. The samples were then immersed in water for 24 hours and its saturated surface dry weight was recorded as the final weight. Water absorption of specimens was determined by the formula as follows.

$$\text{Percentage of water absorption} = \frac{(W_2 - W_1)}{W_1} \times 100$$

Where,

W_1 = Oven dried weight of cubes in grams.

W_2 = Wet weight of cubes after 24 hours in grams

D. Sorptivity

The sorptivity is a simple and rapid test to determine the tendency of water absorption by the mortar by capillary suction. The test was performed on 50 mm x 50 mm x 50 mm cubes. The cubes were tested as per ASTM C1585:13. The samples were dried for 7 days in hot air oven at 50°C . The sides of the specimens were sealed using electricians tape to achieve unidirectional flow. Weights of the specimen after sealing were taken as initial weight. After taking the initial

weight, at time 0 the specimen was immersed to a depth of 5-10 mm in water. At 15 minutes and 30 minutes the sample was removed from the water, excess water was removed with a damp paper towel or cloth and the sample weighed. It was then replaced in water and stop watch was started again. The gain in mass per unit area over the density of water is plotted versus the square root of the elapsed time. The slope of the line of best fit of these points was reported as the sorptivity.

E. Fourier Transform Infra Red spectra analysis

FT-IR analysis helps to understand the behavior of the materials. It identifies the chemical compounds in the sample. The test was used to identify the traces and to enhance quality control. It offered quantitative and qualitative analysis for both organic and inorganic samples. FT-IR is used to find the chemical bonds in the molecule by producing an infrared absorption spectrum. The spectra produced were used to screen and scan the sample. It served as an effective analytical instrument for detecting the functional groups and characterizing covalent bonding information.

F. Scanning electron microscope analysis

The scanning electron microscope (SEM) is a type of electron microscope which produces images of a sample by scanning the surface with a beam of electrons. The electrons interact with atoms in the sample and produce various signals which contain information about the topography and composition of the sample. The electron beams position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer. The SEM mode also detects secondary electrons that are emitted by atoms excited by the electron beam. The number of secondary electrons that could be detected depends on the specimen topography. The samples were scanned, the secondary electrons were collected and the images showing the topography of the sample's surface were created.

IV. RESULTS AND DISCUSSION

A. Cube compression test

The compressive strength of the mortar mixes made with rice husk ash and natural steatite powder at 28 days is shown in figure 3. When compared to the reference mix there was an increase in the compressive strength of the mortar with the inclusion of RHA and NSP except for the mix M5. The strength observed at the mix M2 was the highest which was about 33.57% more than the reference mix. The strength of M3 mix was 28.46% more than the reference mix. For M4 mix the strength was 18.24% more than the reference mix. The strength of M5 mix was 28.3% less than the reference mix. The maximum strength was obtained at 10% replacement (5% RHA and 5% NSP) which can be judged as optimum mix.

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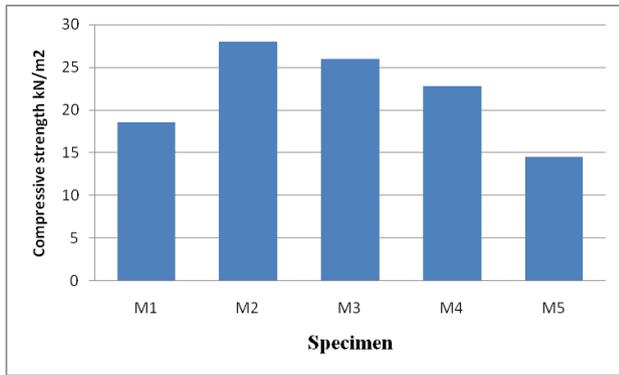


Fig.3. Compressive strength of specimens at 28 days

B. Split tensile test

The tensile strength of the mortar mixes made with rice husk ash and natural steatite powder at 28 days was given in figure 4. When compared to the reference mix there was an increase in the tensile strength of the mortar with the inclusion of RHA and NSP except for the mix M5. The strength observed at the mix M2 was the highest which was about 50.6% more than the reference mix. The tensile strength of M3 mix was 38.4% more than the reference mix. For M4 mix the tensile strength was 11.6% more than the reference mix. The tensile strength of M5 mix was 14% less than the reference mix. The maximum strength was obtained at 10% replacement (5% RHA and 5% NSP) which can be judged as optimum mix.

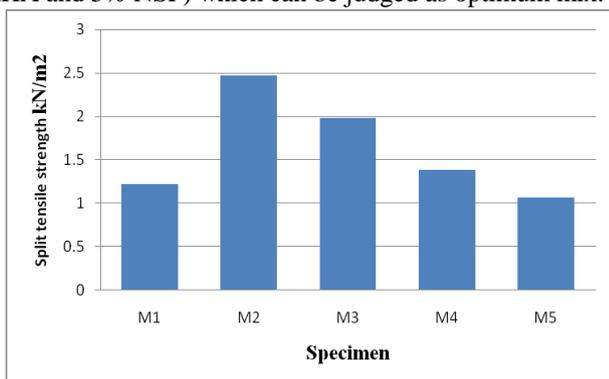


Fig.4. Split tensile strength of specimens at 28 days

C. Water absorption

From figure 5 it was clear that the water absorption values decreased with the addition of RHA and NSP up to 10% and then it increases. The lesser values of water absorption indicated that the mortar is denser which in turn will increase the durability. The specimen M4 have shown higher percentage of water absorption.

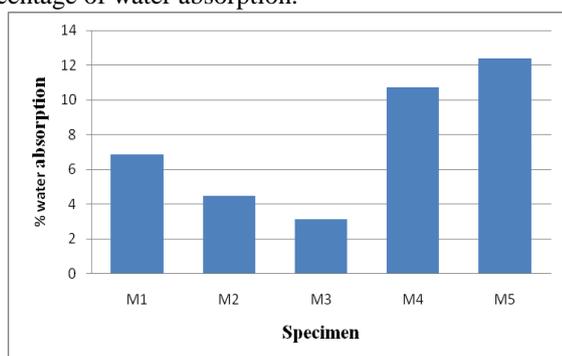


Fig.5. Percentage water absorption of specimens at 28 days

D. Sorptivity

From the figure 6 it was identified that the sorptivity value was minimum for M3 specimen in which the percentage of RHA and NSP is about 10%. The lesser values of sorptivity implies that the mortar is denser which in turn increase the durability.

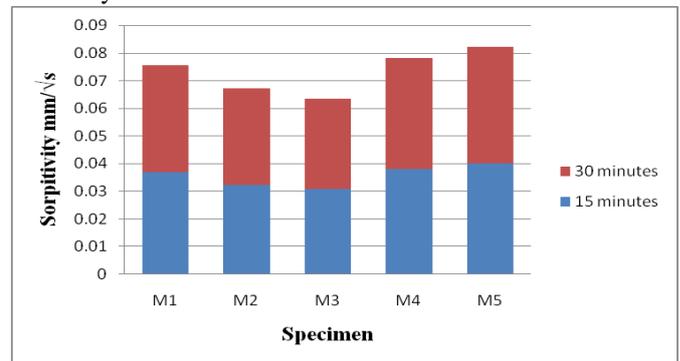


Fig.6. Sorptivity values of specimens at 28 days

E. Fourier Transform Infra Red spectra analysis

The FT-IR spectra of the samples were given in Figure 9, 10, 11, 12 and 13. The absorption bands in the range 460.99 cm⁻¹ and 1018.41 cm⁻¹ was common in all samples which was the characteristic of calcium carbonates. The IR spectrum of M2, M3, M4, M5 samples containing RHA and NSP had a peak in the range 669.30 cm⁻¹ and the presence of calcium silicate hydrates (C-H-S) is clearly seen. The signal around 1649 cm⁻¹ is may be due to the deformation modes of O-H groups and absorbed water molecules. From 3000 cm⁻¹ to 1649 cm⁻¹ bands used to identifying the H-OH stretching and H-O-H bending, and generally known as water bands. 1018.41 cm⁻¹ and 877.6 cm⁻¹ bands are used to identify Si-O-Si and Si-O-Al bonds of tri calcium silicate and tri calcium aluminates respectively. 660 cm⁻¹ and 538 cm⁻¹ bands assigned to the Si-O (di-calcium silicate) banding respectively. The band 1140 cm⁻¹ was overlapped by a broad peak characteristic of calcium silicate hydrates. A narrow band which is centered around 750 cm⁻¹ is present in the spectra of samples with high alkali content. The absorption band in the range 650 cm⁻¹ confirmed an inconsiderable amount of gypsum in the samples. Thus the bonding between the molecules was clearly known from the peaks based on the bands in the particular wave number.

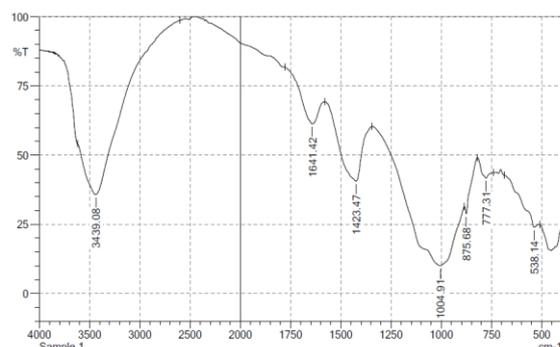


Fig.7. FT-IR sample of M1 specimen

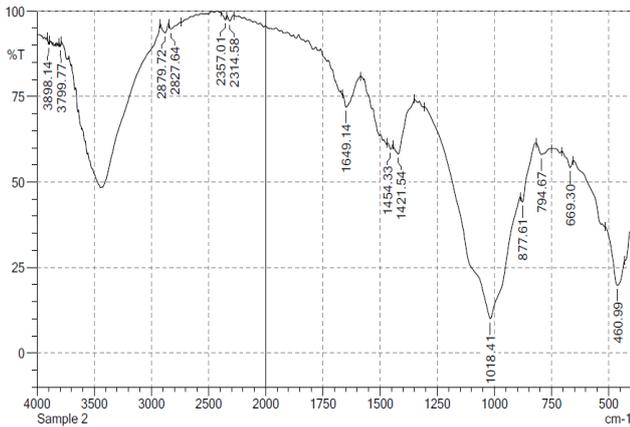


Fig.8. FT-IR sample of M2 specimen

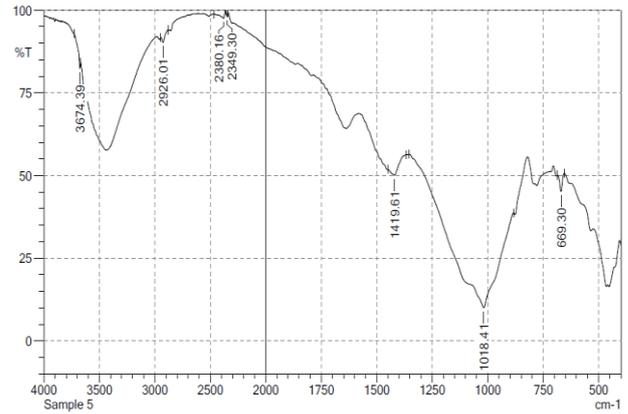


Fig.11. FT-IR sample of M5 specimen

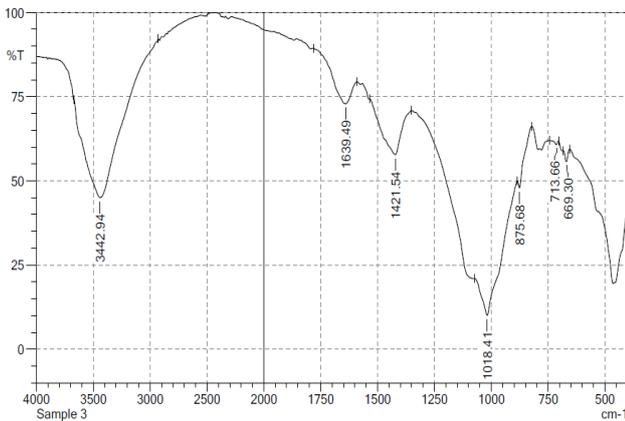


Fig.9. FT-IR sample of M3 specimen

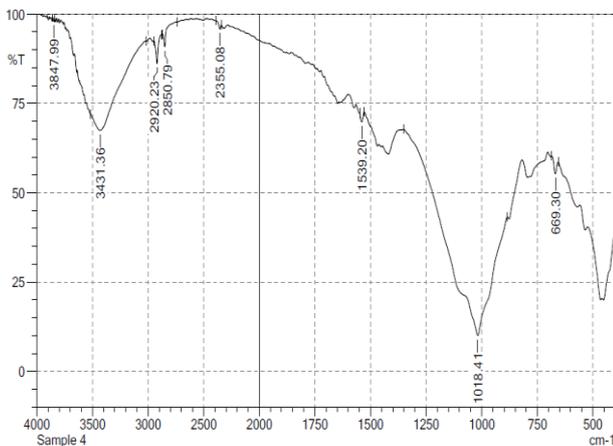


Fig.10. FT-IR sample of M4 specimen

F. Scanning electron microscopic analysis

From figure 12, a needle like structural particle is observed in M1 with pore structures indicating the insubstantial formation of C-S-H and their bond between the C-S-H gel. The SEM image of RHA and NSP blended concrete showed that the uniform structure and it may be due to the fact of the higher surface area of the materials. RHA particles were finer than other materials. Higher amount of RHA particles may be used to activate the pozzolanic reaction, and subsequent strength and durability enhancement. The finer RHA also used to form secondary C-S-H gel. From the SEM analysis report, it can be also noted that the mortar packing density is high due to the absence of large size grains; and the proper hydration process due to the pozzolanic reactions. The NSP used in this experiment developed both C-S-H and M-S-H gel. The magnesium hydroxide (brucite) released during cement hydration may be consumed as a result of interaction with NSP to form M-S-H gel. The intensity of brucite increased as percentage replacement of NSP increases. The calcium hydroxide released during cement hydration may be the end result of interaction with the active amorphous silica present in RHA to form C-S-H gel which contributed to the mechanical and durability properties of mortar. The pozzolanic activity of RHA consumes portlandite forming additional C-S-H phase. M2 specimen showed a good structural bond than specimens due to denser hydration product (portlandite). While increasing RHA content, water demands is increased. It may be due to the higher surface area and formed a layer of RHA particles around anhydrous cement grains which delayed hydration of cement. Therefore the intensity of portlandite decreased in M3, M4 and M5 specimens which reduced the pozzolanic activity and showed disintegrated microstructure. From the results it was observed that replacing cement with 5% RHA and 5% NSP resulted in improvement of microstructure of cement mortar.

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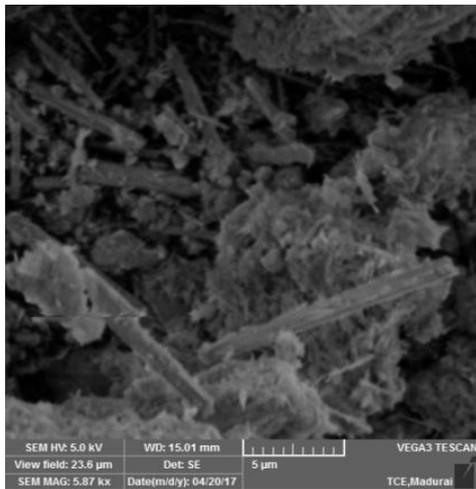


Fig.12. SEM images of Specimen M1

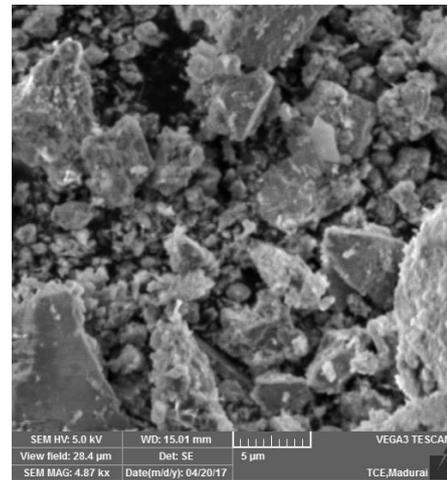


Fig.15. SEM images of Specimen M4

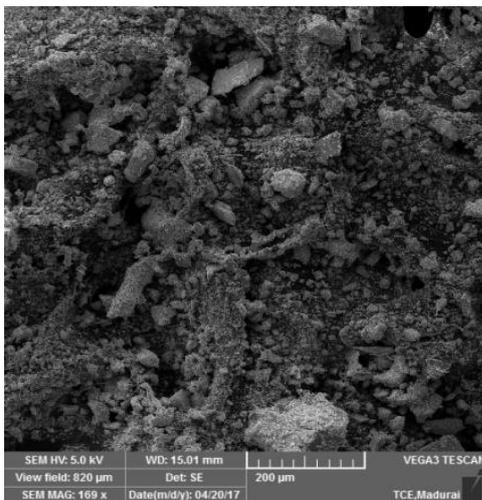


Fig.13. SEM images of Specimen M2

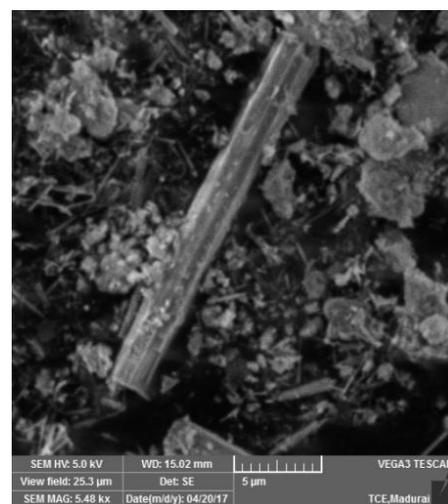


Fig.16. SEM images of Specimen M5

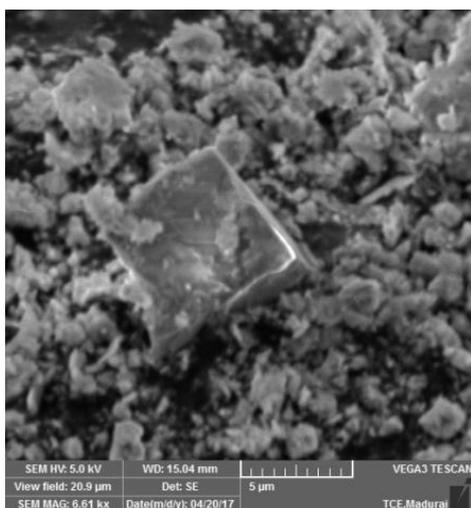


Fig.14. SEM images of Specimen M3

V. CONCLUSION

The following conclusions may be drawn based on the experimental results.

1. It is observed that the greatest increase in compressive and split tensile strength were achieved by substituting cement with 5% RHA and 5% NSP for which the average compressive strength of 28.0 N/mm² and split tensile strength 2.47 N/mm² of was achieved.
2. The durability studies revealed that the water absorption and sorptivity reduces till M3 mix and then it increases.
3. M3 specimen showed lesser water absorption and sorptivity values compared to other specimens.
4. The M2 specimen is showed lower percentage in loss of strength of about 4.29% when immersed in sulphuric acid. Thus it was concluded that the M2 mix was highly resistant to the acids.
5. From the SEM analysis, M2 specimen is showed good micro structural bond compared to other specimens due to denser hydration product (portlandite).

6. The FT-IR test is also revealed that the higher strength of M2 specimen was due to the correct proportion of portlandite and brucite.

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Dr.V.Kannan is a professor and Head of the department of civil engineering in Francis Xavier Engineering College, Tirunelveli. He did his PhD from Anna University Chennai in the year of 2014. His area of research is advanced concrete technology, particularly self compacting concrete and structural elements study. He has sixteen years of experience in his own field. He has published many international journals. Also, he has published a textbook and acting as a reviewer for many reputed journal companies. Further, he is a life time member in various professional bodies. Apart from the academic works, he is also involving in many civil engineering consultancy works from the past ten years.