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Hydration Study Of Rice Straw Ash Admixtured Cement Using Ftir And Sem

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Abstract:Rice straw ash is used as a mineral admixture to cement in the present study. Rice straw is collected from local paddy field and burnt in a clay pit . The ash was collected and grounded to a fine powder. The grounded ash was sieved using a 75 µm test sieve and replaced partially with cement at 5%, 10%, 15% and 20% by weight. The admixtured samples were hydrated for 1hour to 4 weeks and FTIR , SEM, and compressive strength analysis were carried out. The results were analyzed in the light of available literature.

Keywords: Cement, Compressive Strength, FTIR, Rice Straw Ash ,SEM.

INTRODUCTION

Rice has great significance in Indian subcontinent. Producing about one fourth of global rice production India is the second largest rice producer in the world with 101 MT. as on 2013. The rice productionis increased from 20MT in 1950 to 101 MT in 2013 showing a fivefold increase in six decades. Recent researches in agriculture has resulted in 350% growth in the yield per hectare of rice[1]. The cultivation of rice results in two residues namely rice husk and rice straw which are having attractive applications. By burning rice straw we get a secondary byproduct called Rice straw ash(RSA).

India is also the second largest producer of cement in the world with an annual output of 223.4MT in 2012-2013[2]. The phenomenal increase in the cement production is a big concern for the environment since about 930kg of $CO_2$is emitted per 1000 kg of cement produced either directly when calcium carbonate is heated to produce lime and CO_2or indirectly through the use of energy in production involves the emission of CO_2Cement industry is accountable for 5%of manmade CO_2 emission of which 50% is from chemical process and 40% from burning fuels[3].

As CO_2 is acknowledged as a major contributor for climate change steps are being taken to reduce the emission of $CO_2$by all possible ways. One way to reduce the CO_2 emission from cement industry is to use pozzolanic materials to replace cement to a considerable percentages. Enormous works were carried out to identify pozzolanic properties in byproducts and their suitability to use them with cement. Fly ash and Rice Husk Ash were successfully recommended as an admixture for construction [4], [5].Straws and husks of other plants and their ashes were investigated for using them in cement in recent days.

Sugarcane bagasse ash is used in compressed earth blocks and its mechanical strength was analyzed by Sofia A Lima(2012)[6]. The pozzolanic properties of wheat straw ash was studied by Hasan Biricik(1999)[7]. John Kevem supplemented corn husk ash with cement and suggested by analyzing the mortar strength that 10% CHA substitution produce the same strength as that of OPC. By studying the compressive strength of concrete[8], Surajit minsh, suggested that 10% Rice straw ash considerably increases the strength of concrete[9].

For the present study an attempt has been made to admixture RSA with cement and hydration kinetics were analyzed for various time periods using FTIR and SEM.

MATERIALS AND METHODS

Rice straw is composed of both organic and inorganic matter. The prominent organic matter present in rice straw is silica. The silica is absorbed from the ground and gathered in the straw where it makes the structure. When the organic materials are burnt, the silica is left which has pozzolanic in nature.Burning of rice straw under the controlled temperature result in 14% ash of rice straw[10].

In this study commercially available Ordinary Portland Cement (OPC)(43 grade) is used. Rice straw is collected from local paddy field and burnt in a clay pit. The ash is collected and ground toa fine powder. The RSA is sieved using a 75µm test sieve and stored in a desiccator for further use. The entire study was carried out using ground water. The chemical composition of the OPC and RSA were found using Bruker S4 pioneer XRF spectrometer and is listed in Table I.

Table I: Chemical composition of RSA and OPC

Oxides	RSA	OPC
SiO_2	66.72	25.9
CaO	4.78	59.31
Fe_2O_3	0.76	4.11
Al_2O_3	3.26	5.33
MgO	1.48	0.81
K_2O	9.16	1.08
Na_2O	1.12	0.23
TiO_2	0.68	0.6
MnO	0.7	0.11
P_2O_5	0.59	0.2
SO_3	1.35	1.67
LOI	9.4	0.65

For SEM and FTIR analysis, anhydrous OPC is replaced with RSA at 5%, 10%,15% and 20% by weight. The samples were mixed thoroughly and water is added at a water to cement ratio of 0.45. The mixture is mixed for two minutes in a plastic container and allowed to hydrate for fixed time intervals of 1hour (1h),1 day(1d), 1 week(1w) and 4 weeks(4w). The samples were soaked in acetone to remove any evaporable water content and oven dried for two hours at 120 ℃. Then the samples were grounded using agate mortar by applying constant pressure to maintain uniform grain size and stored in a desiccator. FTIR Spectra were recorded with the help of Perkin Elmer Rx1 spectrometer and SEM micrographs were recorded with JEOL JSM 5610LV scanning electron microscope with a maximum accelerating voltage of 20 kv at high vacuum mode.

For measuring compressive strength the samples were mixed with river sand sieved with 600µm test sieve and water at a water to cement ratio of 0.45 and sand to cement ratio of 1.3. The samples were made to a paste by thorough mixing and filled into a 7cmX 7cm X 7cm mould. After 24 hours the specimen were demoulded and cured for different ages. Compressive strength was measured with the help of Unico compressive strength testing machine..

RESULT AND DISCUSSION

The chemical composition (Table I) shows that RSA has 66% of SiO_2 and has a combined percentage of SiO_2, Al_2O_3,and FeO_3 more than 70% (70.84%). This indicates that it is a good pozzolanic material satisfying ASTM C 618 guidelines.

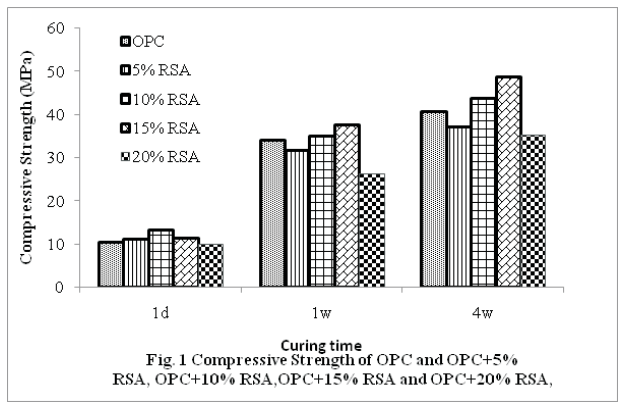

Fig. 1 Compressive Strength of OPC and OPC+5% RSA, OPC+10% RSA,OPC+15% RSA and OPC+20% RSA,

The compressive strength of OPC and OPC with 5%, 10%,15% and 20% RSA were presented in Fig.1. It is visualized from the compressive strength plot that the strength of OPC increases gradually from 1d to 4w. The 15% RSA sample has produced stronger structure than the OPC at 1w and 4w. The 5% admixtured sample shows a decrease in the strength from the 1 day sample onwards up to 4w and for the 20% sample also the strength is less than OPC which may be due to lack of availability of water for hydration process to proceed. The 10% sample shows no significant improvement over OPC in strength . So for the present study only 15% RSA sample is considered for SEM and FTIR analysis.

The FTIR spectra of OPC and 15% RSA admixtured samples hydrated for different periods were presented in Fig 2 and 3 respectively. The prominent absorption bands and their frequency assignment are tabulated in Table II . Cement hydration produce one amorphous or partially crystalline phase of CSH and two crystalline phases of ettringite and portlandite [11].. The main phase responsible for cementing nature is CSH and it can be identified by silicate absorption bands in FTIR spectra. The band at 917cm $^{-1}$ represents ν_3 mode of asymmetric stretching of silicate. It shifts to higher wavenumbers as time progresses is an evidence of hydration[12]. The bands at 519 cm^{-1} and 470 cm^{-1} represent ν_4 mode of out of plane bending and ν_2 mode of in plane bending of silicate respectively. A new band found after 1 day sample at 846 cm $^{-1}$ is due to ν_4 mode asymmetric stretching of silicate in CS_2. The band at 519 cm $^{-1}$is decreased as time progresses and the band at 470 cm $^{-1}$ is increased in intensity indicating the silicate polymerization and strength development which is in well agreement with the compressive strength measurement[13]

Table II: FTIR absorption band assignments

WAVENUMBER (cm^{-1})	VIBRATION MODE	COMPOUND
3629	ν	OH in $Ca(OH)_2$
3422	ν_1	Water
1650	ν_2	Water
1430	ν_3	Carbonate
1102	ν_3	Sulfate
917	ν_3	Silicate
875	ν_4	Carbonate
669	ν_4	Sulfate
519	ν_4	Silicate
470	ν_2	silicate

Ettringite is the first hydration product to form during hydration The ettringite presence and its development during the course of hydration is found by sulfate bands. In 1h sample the band at 1102 $cm^{-1}$is due to ν3 mode of asymmetric stretching of sulfate and the band at 669 due to bending mode vibration of sulfate. At 1h sample the band has shown intensity and begin to decrease as hydration time increased is a indication of sulfate conversion to ettringite[14] . In the 28 day sample the sulfate band is not present indicating the swift nature of sulfate reaction extending only up to one day.

The band at 1480 cm^{-1}may assigned to $\nu_3$mode asymmetric stretching of carbonate. The band at 875cm^{-1} is due to ν4 mode of out of plane bending of carbonate. The band at 1480 cm^{-1} increases from 1 day to 4w and remains same afterwards indicating that the carbonate reaction is extending only up to 4w[15]. The band at 3629 cm^{-1} may be assigned to stretching mode of vibration of OH in $Ca(OH)_2$. The $Ca(OH)_2$ increases up to 1w and its intensity remains same afterwards indicating the conversion of CH

The band at 3422 cm^{-1} and 1650 cm^{-1} are assigned to ν4 and $\nu_2$mode of vibration of water respectively[16]. The band intensities increased upon hydration and at 4w both have broad and strong intensity. The slight shift of 3422cm^{-1} indicates the conversion of ettringite to monosulfate.

The FTIR spectra of 15% RSA hydrated sample exhibits all the above mentioned absorption bands indicating the hydration progress. When comparing with OPC the band at 970 cm^{-1} , 462 cm^{-1} were more prominent than the OPC indicating faster silicate polymerization which is a consequence of hydration reaction. This was clearly visualized in SEM micrographs and well in line with compressive strength measurements.

The SEM micrographs of OPC and 15% RSA admixtured, hydrated samples for 1h, 1d, 1w and 4w are presented in Fig 4 and 5 respectively.

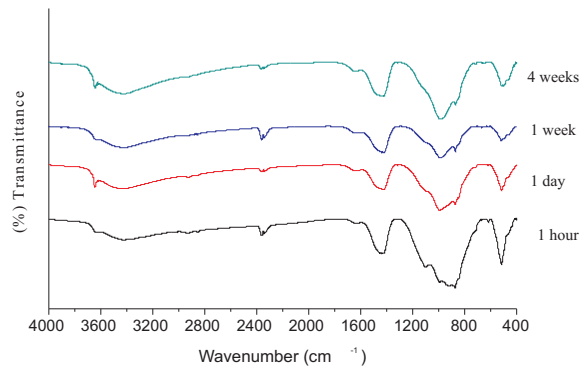

Fig 2. FTIR Spectra of OPC hydrated for 1h, 1d 1w, and 4w

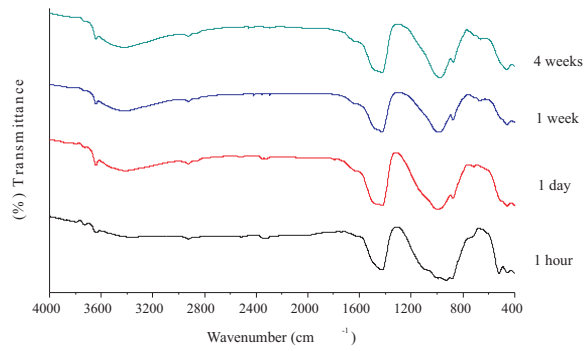

Fig 3. FTIR Spectra of OPC + 15% RSA hydrated for 1h, 1d 1w, and 4w

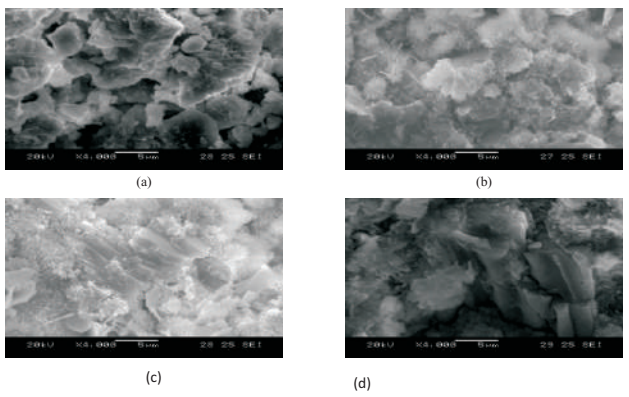

Fig. 4. SEM micrographs of OPC hydrated for (a) 1hour, (b) 1day, (c) 1week and (d) 4weeks

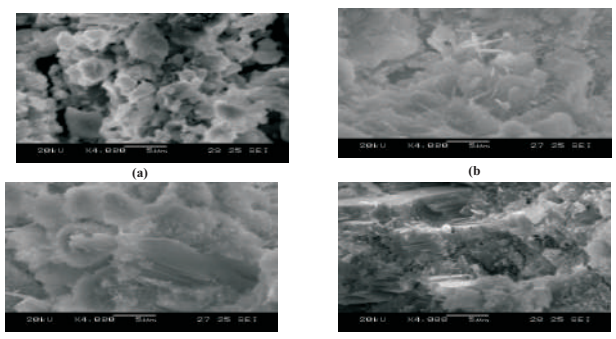

Fig. 5 SEM micrographs of OPC+15% RSA hydrated for (a) 1hour, (b) 1day, (c) 1week and (d) 4 weeks

In 1h hydrated OPC sample (Fig.3) the initiation of hydration reaction is visible with the formation of ettringite colloidal form and CH. The gel like structure around the cement particles is the CH. In 1d sample of OPC (Fig. 4b) ettringite formation is found in needle like structures. At 1w sample (Fig. 4c) well-formed network of the CSH structure is clearly visualized and ettringite crystals begin to disappeared along with CH crystals. The 4w hydrated samples (fig 4d) a silicate hardening phase is seen and the structure becomes bulk with little pores which is the indication of the completion of hydration reaction[18]. In the 1w sample 15% RSA admixtured cement (Fig. 5c) the development of CSH formation is more than the control samples. Similarly in 4w sample (Fig. 5d) silicate polymerization is more prominent than the OPC sample hydrated for the same period which is the indication that the hydration in 15% RSA admixtured samples is hydrating more quickly than the control.

CONCLUSION

1.Rice straw ash samples exhibits pozzolanic properties and comes under ASTM C 618 and also the LOI is less than 10%
2.The water to cement ratio increases with the ash content.
3.For the w/c of 0.45, 15% RSA incorporated cement samples shows higher strength.
4.By increasing the w/c ratio the percentage of ash utilization in cement industry may be increased beyond 15%

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