MICROSTRUCTURAL CHARACTERIZATION OF RICE HUSK AND RESIDUAL ASH FOR THE PRODUCTION OF SUPERIOR BLENDED CONCRETE

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Abstract

Several industrial by-products such as fly ash, silica fume and slag are widely used as supplementary cementitious materials in concrete. In addition to attain designed characteristic strength of structural concrete, it is very imperative to maintain the strength throughout service life of the structure. Supplementary cementitious materials are commonly used in the modern cement manufacturing process to achieve durable and sustainable concrete. Earlier research studies have clearly reported that higher strength and significant reduction in permeability of concrete containing agricultural waste products such as sugarcane bagasse ash and rice husk ash compared to control concrete. However, utilization of rice husk ash and sugarcane bagasse ash in blended cement production is limited to a greater extend as a consequence of lack of understanding and characterization of these materials. Microstructure of rice husk and silica distribution in the residual ash due to controlled burning is not available in the existing literature. This paper reviews reactive silica formation in the rice husk and its residual ashes based on the microstructure and its influence on the pozzolanic performance. Moreover, rice husk ash was processed based on suitable methodology and compressive strength of mortar was found to be high compared to control specimens.

Keywords: *Rice Husk; Microstructure; Pozzolanic Reactivity; Blended Cement*

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1 INTRODUCTION

A number of by products and solid waste materials which have reactive amorphous silica as main constituent in their chemical composition can be used as an alternative cementitious or pozzolanic materials (cement replacement materials). According to ASTM C219-13[1], a pozzolan is defined as "*a siliceous or siliceous and aluminous material, which in itself possesses little or no cementitious value but will, in finely divided form and in the presence of moisture, chemically reacts with calcium hydroxide at ordinary temperatures to form cementitious hydrates.*" Amorphous silica present in the supplementary cementitious materials reacts with available calcium hydroxide in the cementitious system (from cement hydration process) in the presence of water and produces calcium silicate hydrate. Because of the formation of additional C-S-H, increase in long-term strength is observed in blended cement concrete compared to ordinary Portland cement concrete. However, pozzolanic performance of any supplementary cementitious materials is significantly governed by reactive silica content in its chemical composition. Most of supplementary cementitious materials including rice husk ash, bagasse ash are industrial by-products with wide variation in characteristics and cannot be used directly as pozzolanic material in concrete. Minimum level of processing is considered necessary to achieve the status of pozzolanic material as per standards (ASTM C618-12a).[2] Burning process significantly influences the pozzolanic activity of rice husk ash (RHA), sugarcane bagasse ash and metakaolin (Brindley and Nakahira, 1958; Bahurudeen and Santhanam, 2014; Rashad, 2013).[3-5] Chopra et al. (1981) observed that the reactivate silica was retained in rice husk ash up to 700 ºC controlled burning and high pozzolanic performance was also observed.[6] Moreover, further enhance in temperature led to crystallization of silica to crystobalite. Nair et al. (2008) investigated pozzolanic reactivity of rice husk ash using ^{29}Si MAS NMR spectroscopy and the patterns for different burnt samples of rice husk ash were analysed.[7] A broad peak was detected at -111 ppm along with a small peak at -102 ppm for 500 and 700 ºC burnt samples. It was reported as a clear evident and higher pozzolanic reactivity was observed as results of dense silicate network and reactive silanol groups in the residual ash. Brindley and Nakahira (1958) studied the phase transformation of kaolinite with respect to controlled burning and reported the formation of silicon-spinel and mullite.[3] Morat and Comel (1983) studied the effect of calcination temperature and based on obsevation 700–800 ºC was suggested as calcination for higher pozzolanic performance.[8]

It is very clear that burning process significantly govern on the reactivity of rice husk ash. However, earlier research studies were only focused on the residual ashes, it is essential to observe the microstructural changes on rice husk with respect to different range of temperature. Only limited studies are available in the current literature and a comprehensive review on the microstructural characterization of rice husk leads to in-depth understanding on its pozzolanic reactivity. This paper reviews available information on pozzolanic characteristics of rice husk ash based on microstructure of rice hull and significant changes during controlled burning process are highlighted.

2.MICROSTRUCTURAL

CHARACTERIZATION OF RICE HUSK

The main focus of the present study is to understand an in-depth scientific insight on the microstructure of rice husk. Microstructural characterization is very imperative to understand reactive silica distribution on its residual ashes after controlled burning as well as to comprehend the corresponding strength development and pozzolanic performance in concrete. Moreover, silica distribution on these residual fibres as well as ashes is explained further from the earlier research in the following sections.

2.1 Rice hush and residual ash

Rice husk or hull is basically the outermost layer of the rice or paddy grain. It gets separated out from the main rice kernel during the milling process. Rice husk which is also known as lemma (shown in Figure 1) and it covers the entire rice seed which contains the embryo including plumule and radical as well as endosperm, and a variety of other important components of the rice seed.

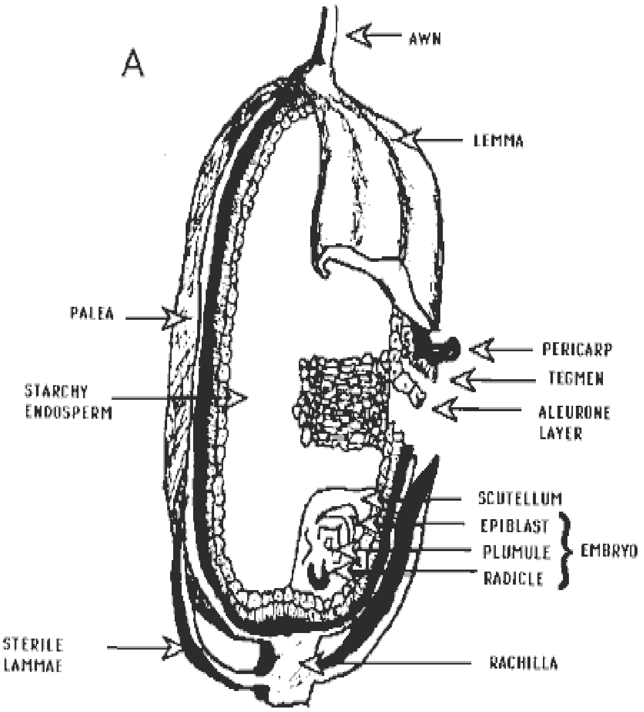


Fig 1: Detailed structure of rice seed and hull [9]

Scanning electron Microscopy (SEM) images of the exterior surface of rice hull was investigated by Watson and Dikeman (1977)[10] and an SEM micrograph of exterior hull is shown in Figure 2. It is interested to note that a clear distinguish on the dentate rectangular elements that were found to be present over the exterior surface of the rice husk and few of these had mechanical indentations as illustrated in Figure 2. The exterior of the Rice Husk was found to be moderately dissimilar than those of other cereal grains.

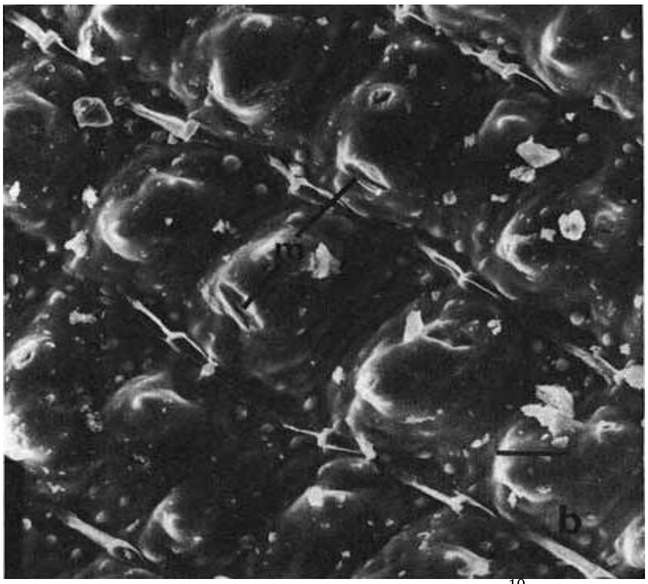


Fig 2: SEM Micrograph of rice hull[10]

Surface microstructure of a virgin rice hull was initially investigated by Brindley and Nakahira[10] using SEM and shown in a study Figure 3. It was found that there were a number of longitudinal fibers which were interspaced in the interior of the matrix of lignin and glucose. This structure was found to be quite analogous to that of a composite material in which fibers are placed regularly in the matrix. It is interested to note that in the rice husk, fiber part constitutes of silica, cellulose and lignin which were found to present in the form of matrix.

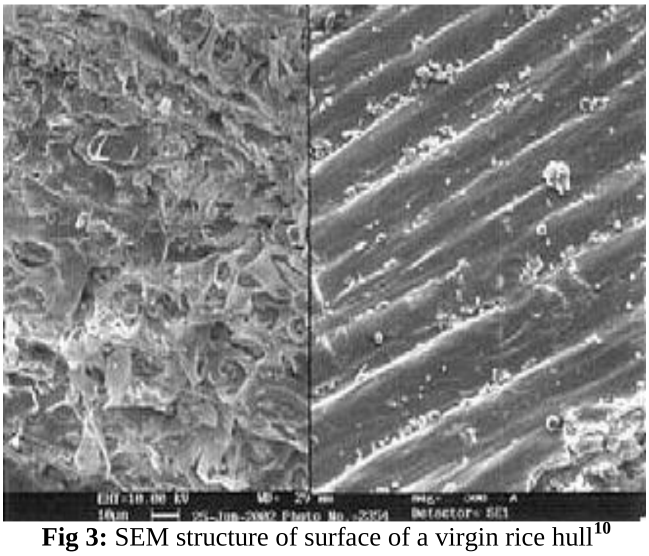


Fig 3: SEM structure of surface of a virgin rice hull**[10]**

SEM micrograph of rice husk that were burnt at temperature up to 350 ºC clearly reveals the changes on the microstructure compared to virgin rice husk. Significant changes in the microstructure are reported in the previous research studies. Most important, button-like structures or bumps as shown in Figure 4 which were interspaced with small pores were found to be present on the surface. Occurrence of these bumps and pores was clearly described in the earlier studies as well as explained as a result of the escaping the volatiles from the surface due to the rapid thermal degradation. [11] The presence of bumps was further explained by researchers by means of the obstruction provided by silica fibers to de-volatilization. The parts from where volatiles escaped, presence of craters can be seen on the surface that were exposed to controlled heat or burning process. It is interested to note that when the temperature was raised up to 850 ºC, the number of pores and bumps were found to considerably increase as shown in Figure 4.

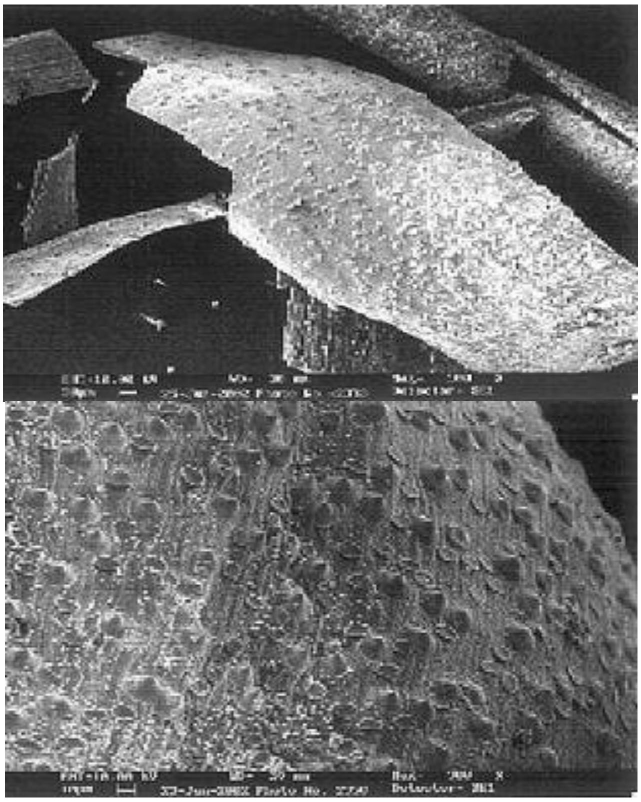


Fig 4: Surface of a particle heated to 350 °C (left); Rice husk heated to 850 °C (right)[11]

SEM micrograph of rice husk was observed and cross section is shown in Figure 5. Elongated hypodermal fibers were evidently found to be present in the micrograph. The complete inner region of hull was found as fibrous and striated

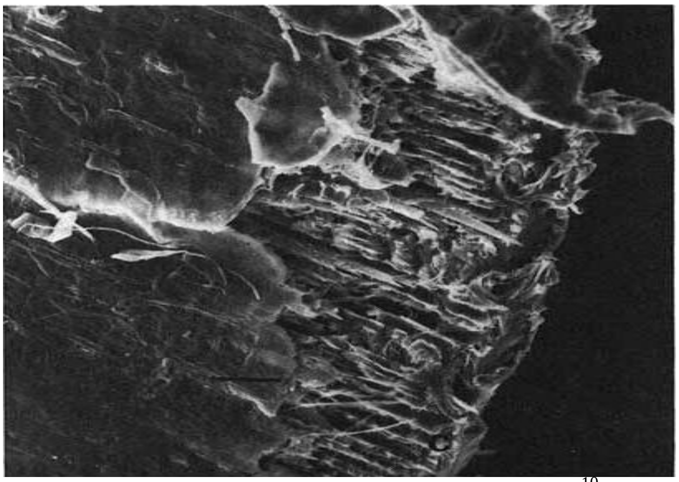


Fig 5: SEM micrograph of cross section rice hull[10]

Afterwards, to find the effects on the cross section of rice husk particle due to controlled burning, the husk was heated up to 350 ºC. A number of pores were created due to the pyrolysis as shown in Figure 6. Pyrolysis has selectively consumed the cellulosic material of the matrix leaving only the silica fibers at the rear. When the temperature was further raised up to 850 ºC the pores were transformed into channels as cellulosic material as favorably deleted during the pyrolysis as shown in Figure 6.

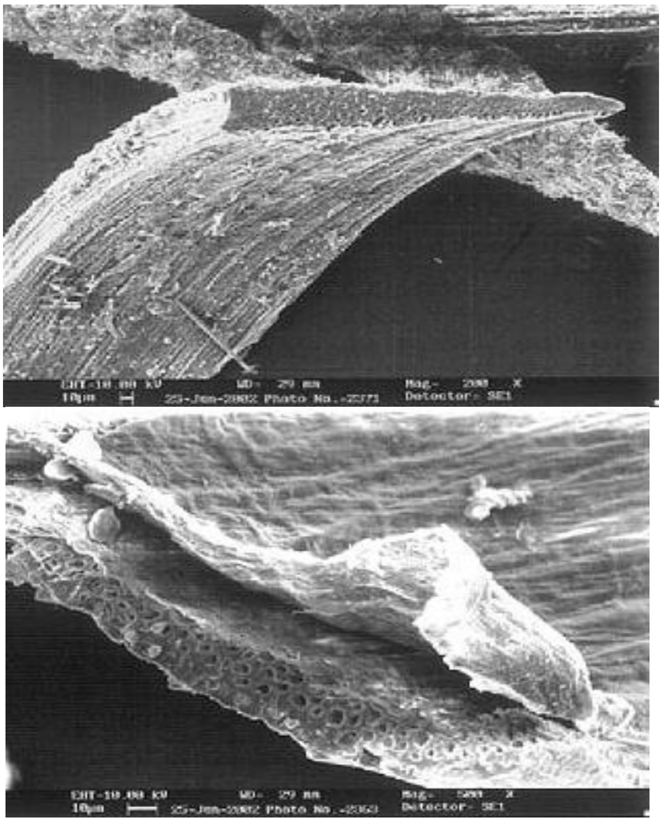
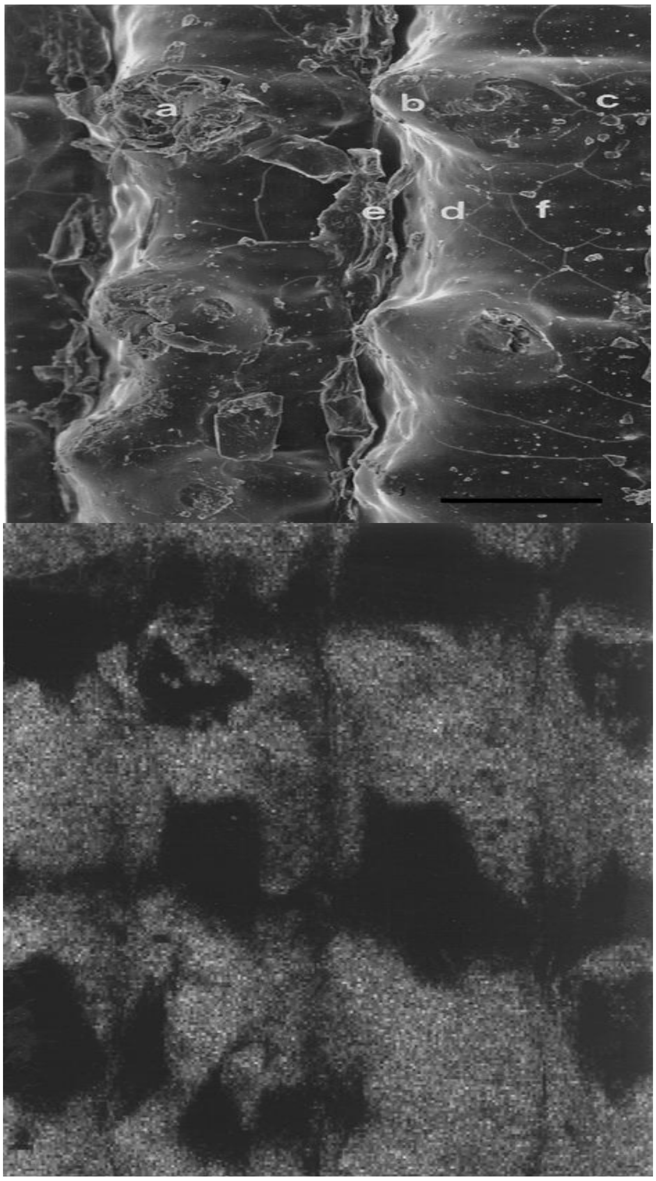


Fig 6: Formation of pores as a result of pyrolysis: Cross-section of particle heated to 350 °C (left); Cross-section of rice husk particles heated to 850 °C (right) [11]

Confocal laser scanning microscopy (CLSM) experiments generally combine the He-Ne laser with the benefits of confocal optics. Observation from CLSM experiments combined with the SEM interpretations proposed that the rice husk or hull structure is a comparable to a composite material in which the silica tubes are jam-packed with cellulose substance. Moreover, these were found to be mainly constitutes the strong phase and the matrix part which consists of the lignin. When these rice hull particles were exposed to higher temperature, the silica tubes remain unaffected. On the other hand there are large numbers of pores as a result of pyrolysis were also observed.

On the other hand, investigation on dissemination of the silica in rice husk is very imperative to identify silica rich components and carbon rich particles. In another study[12], silica distribution was mainly analyzed using FE-SEM image and elemental mapping of silica of the superficial part of lemma as represented in Figure 7. The researcher reported that brightness in the figure basically represents the presence of silica. The higher is the intensity of brightness greater is the silica concentration. Furthermore, another notable interpretation in the study is that the area which has very little or no brightness contains negligible amount of silica. The dome-shaped outcrops (see Figure 7) are the part where silica concentration was found to be higher in the study. In addition to this, FE-SEM image and the elemental plotting of rice husk across its thickness was analyzed. A notable observation from the study that the brightness was found to be more intense in the external portion of the dome-shaped outer epidermal cells which evidently represents considerable amount of silica concentration in the corresponding region. Additionally, it was clearly confirmed and inferred from the image that the other regions of the cross-section of the lemma shows very little or almost no brightness.[12] This is a clear evident reported for the microstructure characteristics of rice hull and its silica distribution. Reaming regions was found to have less silica concentration as well as considerable reduction in brightness compared to silica rich external region of epidermal cells.



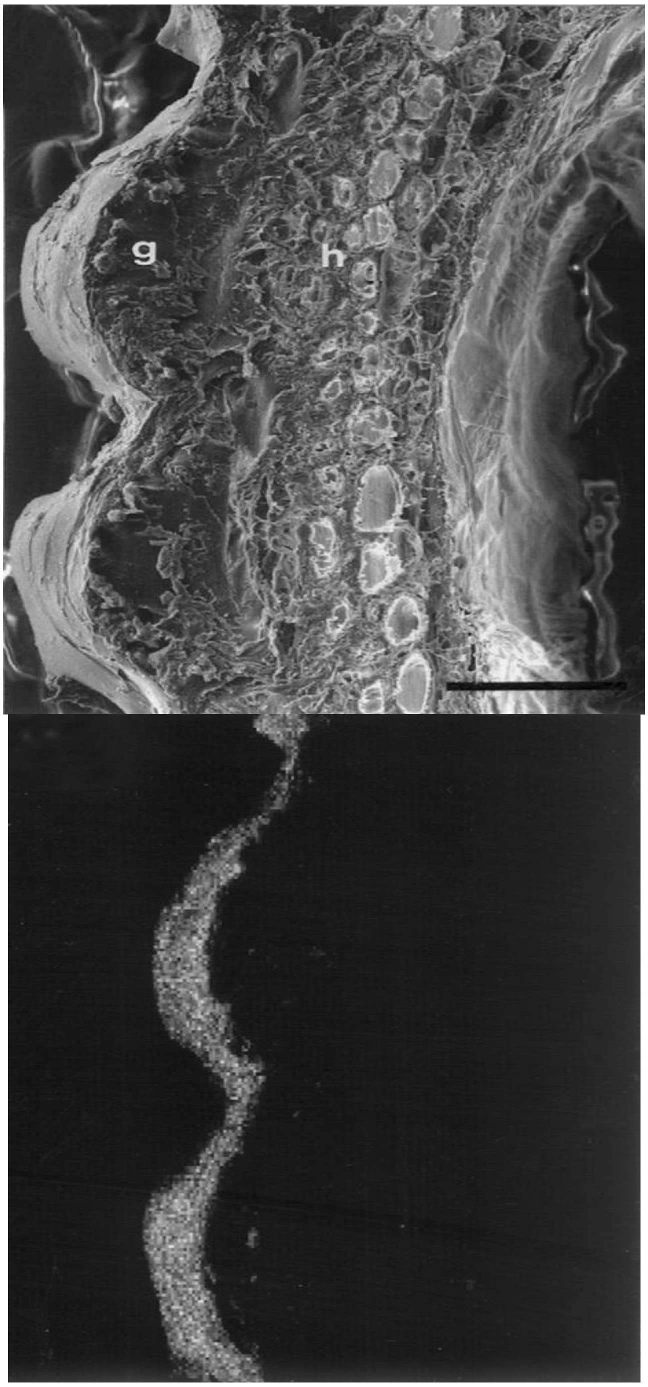


Fig 7: FE-SEM image and elemental mapping of silica of the outer surface of lemma (above); Elemental mapping of lemma across its thickness[12]

It was further confirmed that silica was found to present in abundance in the outer tissues of its lemma as well as in the region of internal tissues that corresponds to the sub-epidermal fibers. SEM Micrograph was observed for outer epidermal cells and images from fractured surfaces were investigated. It is interested to note that abundant silica was found to be present over small particles of silica (see Figure 8) were easily distinguishable. Size of these particles varied from less than 100 nm to 1µm. Large size particles were clearly observed and it was further confirmed as a results of the accumulation of smaller particles.[12]

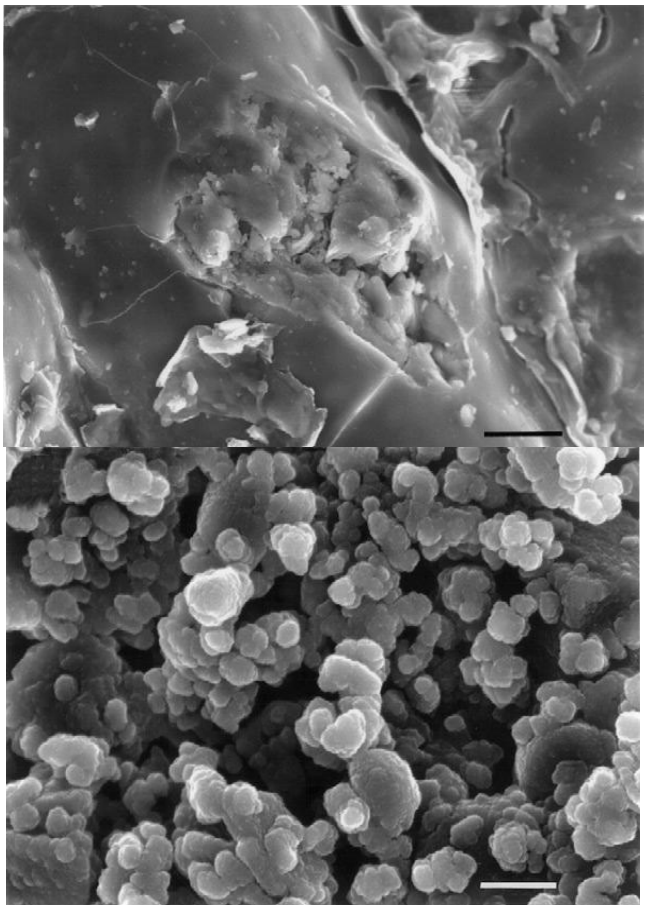


Fig 8: FE-SEM micrograph showing the presence of silica just underneath the outer epidermal cell surface fractured (left); SEM micrograph showing silica grains [12]

3 EXPERIMENTAL EVALUATION OF POZZOLANIC PERFORMANCE OF RHA

Based on the above discussion, rice husk was collected from a sugar mill, India and burnt at 700 °C and an accelerated pozzolanic strength activity index test was performed for the processed rice husk ash (as per ASTM C1240)[13]. The replacement level of rice husk ash was adopted as 10% and standard water-binder ratio was maintained for control specimens as well as rice husk ash blended specimens. Specimens were cast as described in standard and initially cured for 24 hours in the moist room. Subsequently, the specimens were stored at 65±2 °C for additional six days as specified in ASTM C1240. However, the standard suggests addition of water reducer or superplasticizer, sufficient workability was observed in the study without superplasticizer. This is an important feature in ASTM standard, because a replacement level 10 % and the use of superplasticizer are suggested in the evaluation of pozzolanic performance of high pozzolan such as silica fume, rice husk ash based on real concrete applications where usually equivalent levels of replacement and use of superplasticizers are accepted. After 7 days and 28 days of curing, compressive strength of control and rice husk ash blended mortar specimens was tested to evaluate the pozzolanic performance of rice husk ash and results are shown in Figure 9.

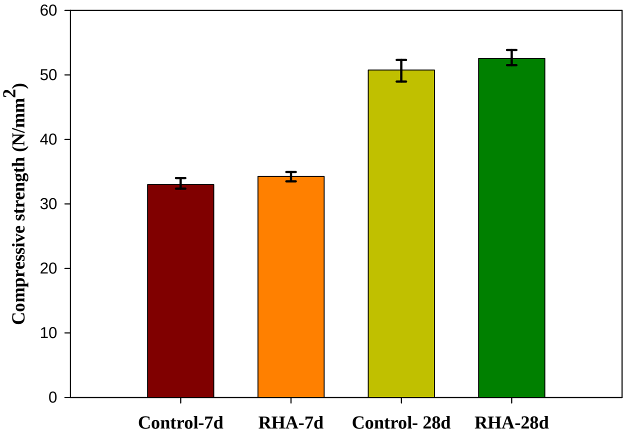


Fig 9: Compressive strength after 7 and 28 days of curing.

Compressive strength of RHA blended mortar was found to be high compared to control mortar specimens. Moreover, significant increase in compressive strength was observed for RHA blended mortar specimens after 28 days curing in the present study. It is a clear evident for pozzolanic performance and additional strength gain is as results of CSH formation from pozzolanic reaction.

4 CONCLUSIONS

- The available information on microstructural characterization of rice husk as well as its residual ashed, its influence on the pozzolanic performance for use as a supplementary cementitious material in concrete has been reviewed. It will throw light on an improved understanding of rice husk ash and leads to achieve its effective utilization in concrete.
- Rice husk ash processed based on suitable methodology and proper assessment of pozzolanic performance of rice husk ash blended cement mortar was performed. Rice hush ash blended specimen showed higher strength after 7 and 28 days of curing compared to concrete specimens due to excellent pozzolanic performance of rice husk ash.

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