

DEVELOPMENT OF BLENDED CEMENT UTILIZING THE POZZOLANIC AMORPHOUS SILICA COMPONENT OF RICE HUSK ASH

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Abstract: Ordinary Portland cement (OPC) manufactured in Sri Lanka, is expensive and in short supply. The use of OPC for low strength masonry applications is also uneconomical. The pozzolanic activity of natural agricultural wastes like rice husk can be used to produce blended cements to overcome this problem. In the present study, rice husk was fired separately in a laboratory electric furnace and in an experimental incinerator set up in the field. The amount of active silica present in the resulting ash from two known varieties of rice (BG 300 and BG 400) fired in the laboratory furnace was found to be maximal (around 45%) at a firing temperature of 700^o-725^oC. However, rice husk fired in the experimental kiln exhibited variable active silica content (24%-45%). Ash obtained from experimental kiln was ground to a fine powder and blended with OPC in the proportions of 70/30, 60/40, 50/50, 40/60, and 35/65 ASH/OPC. Mortar strength of the above compositions showed low early strength development. However, the strength development at 28 days was appreciable. The compositions 40/60, and 35/65 were found to be optimal.

Key words: Agricultural waste, cement, rice husk ash

INTRODUCTION

The cost of construction using cement produced in Sri Lanka is high to the local house builder. This is mainly due to the high cost of fuel used in cement production. The troubled situation prevailing in the North and East also results in reduced production of cement. This necessitates the importation of either clinker, cement or both. Portland cement is uneconomically and unnecessarily used for low-strength applications such as foundation mass concrete, plasters, mortars and soil stabilization.

In most developing countries there exist several types of cement for different applications. But Sri Lanka and most African countries have only one type of cement. This gap can be bridged by exploiting the pozzolanic activity of natural agricultural wastes such as rice husk for the production of pozzolanic cements. Every paddy harvesting season leaves an abundance of paddy husks in many parts of the country. Areas where average rice milling capacity exceeds 500MT per annum from which rice husk could be exploited as a raw material for the production of pozzolanic cements include Kurunegala, Polonnaruwa, Ampara, Anuradhapura, Hambantota, Jaffna, Matara, Batticaloa, Trincomalee and Kandy.

When rice husk is burnt, the resulting ash has a good binding action in conjunction with hydrated lime in the presence of water. Previous research¹ has shown that when rice husk is burnt in a furnace or an incinerator where the temperature and firing times are well controlled, the resulting ash has good pozzolanic properties.

Pozzolanic activity of rice husk can be utilized to produce blended cements by intergrinding or blending ash with Portland cement clinker or OPC in definite proportions. The resulting Portland pozzolanic cements have a range of properties according to the degree of activity of ash, the proportions used and the method of processing employed.² Previous research with such pozzolanic cements as the main binder has indicated strength comparable with OPC mortar and concrete. Other properties such as soundness, fineness and setting times of pozzolanic cements are also within acceptable limits. However, the rate of gain in strength of pozzolanic cements is low compared to OPC.¹

Generally, the pozzolanic materials, including rice husk ash (RHA), improve the durability of concrete. RHA cement compared to OPC is observed to be highly resistant to the attack of acids.^{1,3} This is due to RHA's low lime content. Tests carried out on durability showed that lime-RHA cements stored in 1% acetic acid solution remained in excellent condition for five years while Portland cement deteriorated substantially within one year.¹ It has been observed that the reactivity of silica in rice husk is influenced by the firing temperature and duration of firing. The studies also established that the maximal temperature of firing is 750°C. Higher temperature combined with increase in firing time leads to growth of silica crystals resulting in poor reactivity.¹

The objective envisaged in this study was to investigate the following: (i) the critical firing temperature and the isothermal firing time (soaking period) for different varieties of rice husk available locally; (ii) the factors favourable for the formation of active silica present in the ash fired in the laboratory furnace and relate these conditions with the field trials in order to improve the efficiency of the production of ash under field conditions.

METHODS AND MATERIALS

Rice husk: The husks from the following varieties of rice were used to produce ash (i) BG 300 (ii) BG 400 and (iii) two unknown varieties from Kurunegala and Galle districts.

Optimization of firing conditions: Previous research with rice husk available in other countries indicates an optimum firing temperature range of 600° to 700°C and isothermal firing time of 30 min. Based on these results, an attempt was made to optimize the conditions for local rice husk by firing in an electric muffle furnace at temperatures varying from 500°C to 800°C for durations of 30 min and 1h.

Determination of amorphousness of silica present in rice husk ash: A relative indication of the maximum amount of amorphous silica produced under different thermal conditions, was determined by a solubility test.⁴ 3g of the prepared rice husk ash sample (material passing 63 μ m test sieve) was digested with 100 ml of 0.2 N NaOH at 100°C and continuously stirred for 3 min in order to ensure that the amorphous silica mass is dissolved in NaOH. After this treatment, the contents were centrifuged and the residue washed with distilled water. Finally,

the sample was dried to a constant weight. The difference in weight expressed as a percentage of the original weight of the sample was recorded as the degree of amorphousness of silica contained in ash.

Field trials on calcination of rice husk

Features of the experimental kiln: An experimental firing unit was constructed at the Materials Laboratory of the National Building Research Organization. The important features of two similar kilns (i.e. i. square type experimental incinerator and ii. circular type incinerator) developed in India⁵ were incorporated into this kiln and the cost of construction minimized.

A square type incinerator with open brickwork was set up due to the ease of construction using brick wastes. The capital investment for a circular type ferrocement incinerator (developed at the Asian Institute of Technology) and Tube-in-Basket type incinerator (developed at the Indian Institute of Technology - Kanpur) is higher compared to the brickwork construction used in the experimental kiln.

This experimental unit (Fig. 1) includes the following features favourable for the formation of active material:

- (a) Open brickwork construction
- (b) Greater access of air
- (c) Metal netting at the centre, connected to the metal chimney to retain the material and to facilitate adequate ventilation
- (d) Damper to control the draught
- (e) Feeding hole for continuous operation of the kiln.

Calcination schedule: The kiln had a capacity of burning 150 kg of rice husk in a single firing. The temperature of burning rice husk was measured by using thermocouples at different heights and across different sections of the kiln.

Grinding of ash and formulation of suitable compositions involving OPC and active material: The ash produced from the experimental kiln was ground in a laboratory pot mill to a fine powder (fineness 425 m²/kg) for 6h. Powdered ash was blended with OPC for 1h for making mixes having proportions of OPC to ash 30/70, 40/60, 50/50, 60/40 and 65/35 by weight.

Fineness test for 60/40 and 65/35 blended mixes was carried out using Rigden's Air Permeability apparatus in accordance with the method given in IS 4031.⁶

Properties of blended mixes

Strength development: From the experimental cement prepared, a set of 9 standard mortar cubes (70.7 mm) was cast from each blended mix. A set of control cubes was also cast using OPC. These cubes were cured and tested according to the test methods given in IS 4031.⁶ Test samples were cured as follows.

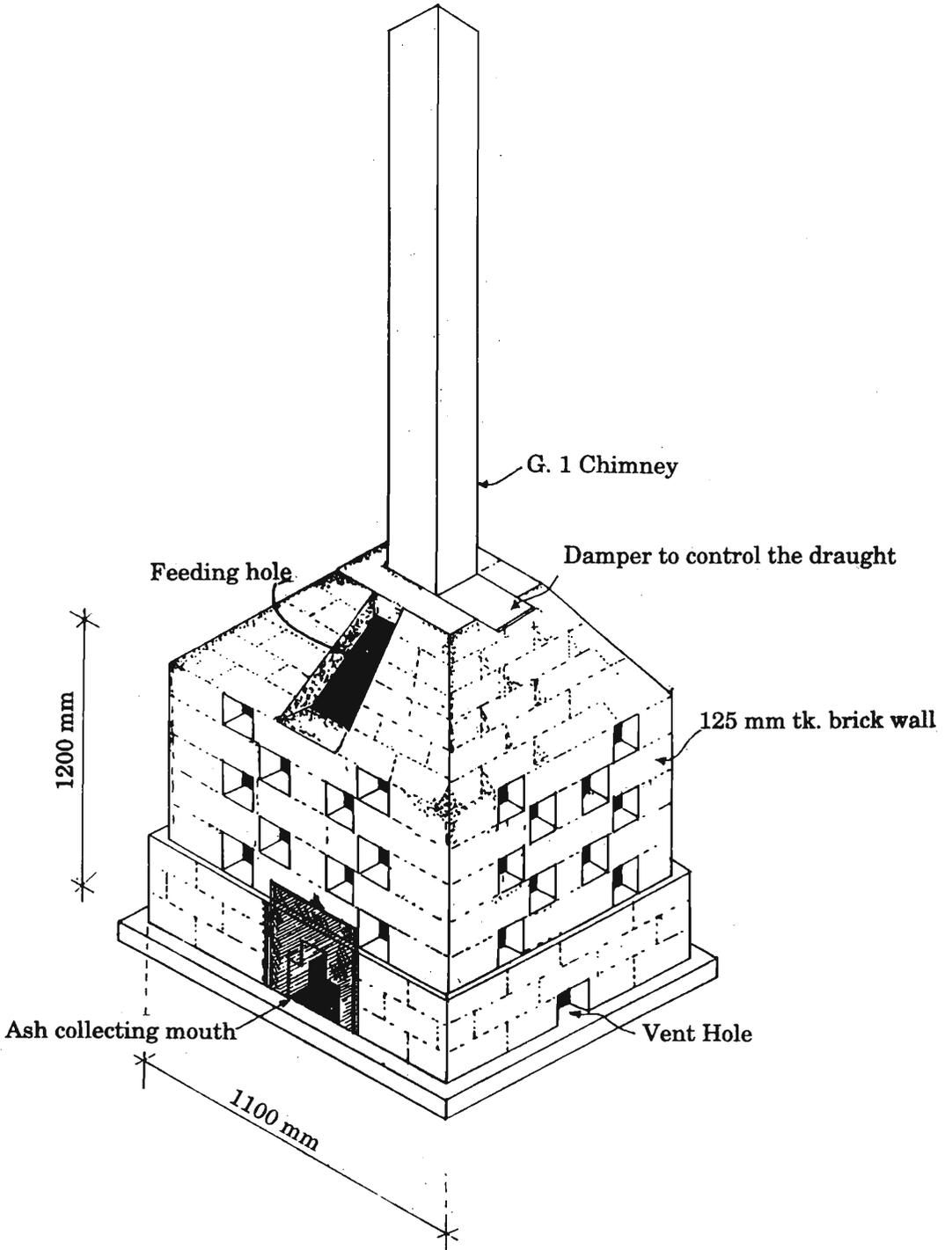


Figure 1: A schematic diagram of the experimental incinerator.

Filled moulds were kept under ambient conditions for 24h after completion of vibration. The cubes were then removed from the mould and immediately submerged in clean water until testing. Three cubes were tested after 3d and the balance cubes were tested (three cubes at a time) after 7d and 28d respectively. Compressive strengths were calculated from the crushing load and the average area over which the load was applied. Strength development in relation to the requirements specified in IS 1489 for Portland pozzolana cement⁷ were evaluated (Fig. 2).

Determination of pozzolanic activity index (PAI): Two blended cement compositions which showed significantly high strength development (i.e. 40/60 and 35/65 of Ash/OPC) were subjected to further tests. The reasoning behind the test is given in (a) and (b) below.

a. Pozzolanic activity

The ability of silica in rice husk ash to combine with lime to form cementitious compounds could be estimated in terms of the pozzolanic activity index (PAI).⁸ The PAI values for experimental cements were determined by comparing the compressive strength of test mortar (prepared with the above blended mixes) with the OPC control mortar prepared and tested under similar conditions.

b. Strength development under the accelerated curing conditions

It has been found⁸ that in concrete made with the pozzolanic cements such as those based on pulverized fuel ash, and cured under accelerated curing conditions, a strength equivalent to OPC concrete at 28 days could be expected. Based on the above, the following tests were carried out according to BS 3892.⁸

i. The flow of OPC control cement mortar (for a water : cement ratio of 0.5 and cement : sand ratio of 1:3) was determined by the method given in BS 3892.⁸ Test mortar was prepared with a water content which gave the same flow value (a flow value of 8) as the control cement mortar (refer Table 1 for actual water: cement and water: blend ratios). Three sets (a set comprised 3 cubes of 50 mm standard mortar) were cast along with control mortar cubes.

ii. The cubes were subjected to the following curing regime: 24h in moist air, followed by four days in water at ambient temperature, 46h in water at 50°C and finally 2h in water at ambient temperature.

iii. Immediately after the final stage of curing, the cubes were tested for compressive strength using AP 1000 series Versa Tester using a rate of loading of 60 N/mm² per minute.

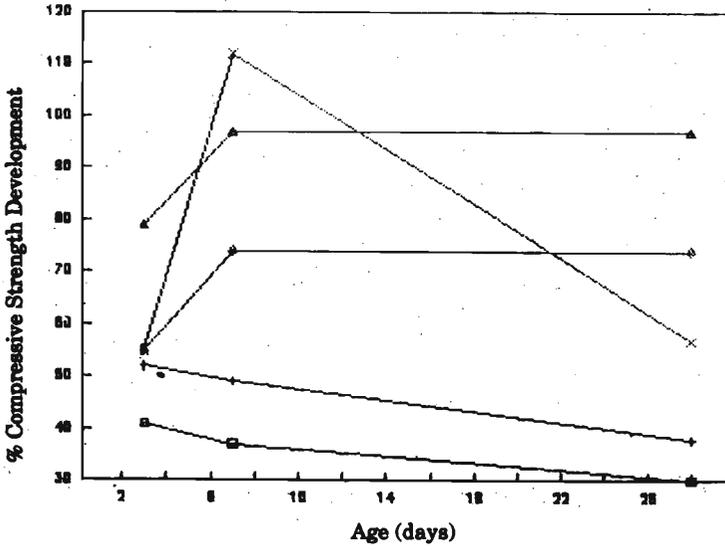


Figure 2: Strength development of mortars made from different compositions of RHA/OPC.
 RHA/OPC □ 70/30 + 60/40 ◇ 50/50 △ 40/60 × 35/65

$$\% \text{ strength development} = \frac{\text{Observed strength}}{\text{Expected strength}} \times 100$$

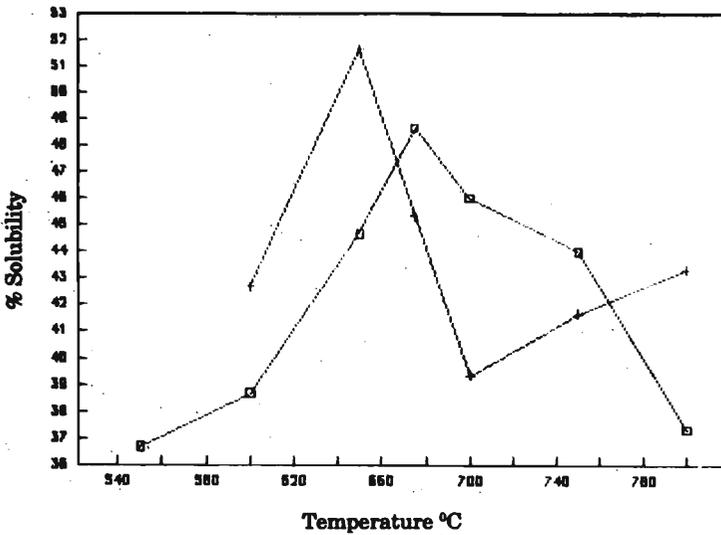


Figure 3: Relationship between solubility of amorphous ash and firing temperature for an unknown variety of rice husk.
 source of rice husk : par boiled rice
 □ 30 min soaking
 + 60 min soaking

RESULTS

Solubility test for rice husk ash obtained from electric muffle furnace

The variation of % solubility with temperature of calcination under different isothermal firing times for different varieties of rice husk calcined in the electric furnace is given in Figures 3-5.

Calcination of rice husk in the experimental field kiln

The maximum temperature observed in the trial firing was approximately 900°C in the immediate vicinity of the metal netting at the central section of the kiln. It dropped rapidly with increasing distance from the centre. The temperature of the bed of rice husk varied from 400° to 900°C. The maximum temperature was attained 2 1/2h after the commencement of fire. Complete firing cycle was 10 to 12h while the maximum temperature persisted for approximately 50 min.

The rice husk ash fired in this experimental kiln exhibited a wide variation in reactive silica from 24% to 45%. This is evident from the values of solubility of different varieties of rice husk (which in turn is related to reactive silica content) fired in electric furnace, which vary within the limits 41.6% - 51.6% and the corresponding values 24% to 45% obtained when rice husk was calcined in the experimental kiln.

Grinding of ash and the determination of fineness of blended mixes:

It was observed that the well burnt grey ash could be ground to the required fineness within a period of about 4h. The resulting ash had an average fineness of 425 m²/kg. Blended ash/OPC optimized had an average fineness of 350 m²/kg.

Properties of blended mixes

Strength development of mortar

(a) Under conditions of normal curing

Strength development of mortar cubes in relation to the requirement specified in IS 1489 for Portland pozzolanic cement is given in Figure 2.

(b) Under conditions of accelerated curing

Strength of mortar cubes in relation to those made with OPC control mixes is given in Table 1, which also shows Pozzolanic Activity Index of the two blended formulations of rice husk ash calcined in both the experimental incinerator and laboratory electric furnace.

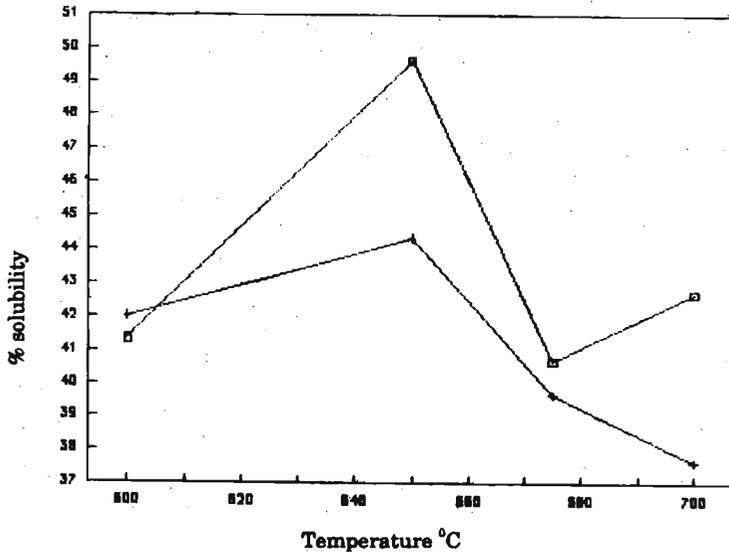


Figure 4: Relationship between solubility of amorphous ash and firing temperature for an unknown variety of rice husk.

source of rice husk : Raw rice

□ 30 min soaking

+ 60 min soaking

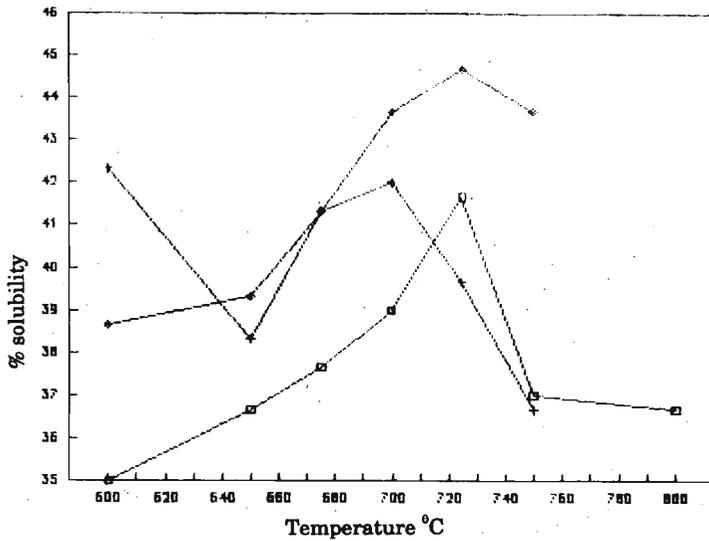


Figure 5: Relationship between solubility of amorphous ash and firing temperature for various varieties of rice husk.

source of rice husk : par boiled rice

□ BG 300, 30 min soaking

◇ BG 400, 30 min soaking

+ BG 300, 60 min soaking

Table 1: Pozzolanic Activity Index (PAI) of two blended formulations of rice husk ash (RHA)

RHA Source	Composition RHA/OPC	Average Compressive Strength N/mm ²	PAI	w/c or w/blend ratio
Experimental Incinerator	RHA	24.0	120	0.69
	40/60	13.0	65	0.67
	35/65	15.0	75	0.62
Laboratory electric furnace*	RHA	32.0	160	0.88
	40/60	21.5	107	0.75
	35/65	30.5	152	0.77
	OPC control	20.0		0.50

* Ash from laboratory electric furnace fired at 675°C and 30 minutes isothermal firing.

DISCUSSION

The amount of amorphous silica in rice husk ash from two known varieties of rice husk (BG 300 and BG 400) fired in the laboratory electric furnace was found to be maximum (around 45%) when the firing temperature was between 700° to 725°C (Fig. 5). For one of these varieties (BG 300) it was found from solubility test results that the optimum isothermal firing time was 60 min. For other variety (BG 400) it was not possible to state which isothermal firing time (30 min or 60 min) was best, due to insufficient data. However, for BG 400, the percentage solubility (amount of amorphous silica present in the ash) was higher under 30 min isothermal firing than that for BG 300 observed under 60 min isothermal firing. Similar calcining trials performed with the two unknown varieties of rice husk indicated an optimum firing range between 650° to 675°C (Figs. 3 and 4). For one of these unknown varieties (Fig. 3) an isothermal firing time of 60 min was found to be the optimum and for other variety (Fig. 4) 30 min was the optimum.

Thus, the present study indicates that: (i) Both the firing temperature and isothermal firing time for attaining the highest amount of amorphous silica in the fired ash depend on the variety of rice husk. (ii) As an approximation, a firing temperature of 700°C seems to be the optimum for the four Sri Lankan varieties of rice husk examined. (iii) An isothermal firing time of 30 to 60 min is to be employed based on the prior knowledge of the variety of rice husk.

Compressive strength of mortar cubes of five blended cement compositions showed low early strength development as compared to OPC control mortar. Strength development at 28d however, was appreciable. The compositions 40/60 and 35/65 were the most favourable in terms of strength development. However,

the 28d compressive strength for 35/65 mix was anomalous and shows a decrease in absolute terms from the 7d strength to the 28d strength. This could be due to the non-homogeneity of ash obtained in different trials of the experimental kiln.

The Pozzolanic Activity Index (PAI) determined for the 35/65 mix was 75 whereas that for the mix 40/60 was 65, for RHA obtained from the experimental incinerator. (A minimum PAI value of 85 is recommended for pulverized fuel ash based pozzolanic cements).⁸ These observations indicate that the efficiency of calcination in the experimental kiln requires further improvement. PAI determined for RHA obtained from the experimental kiln is low as compared to the PAI determined for RHA obtained from laboratory electric furnace. But the PAI determined for the optimized blended mixes (OPC with ash from experimental kiln) were at reasonable levels (75) when compared with the PAI recommended (85) for pulverized fuel ash based pozzolanic cement.

Mortars made from five different compositions of RHA/OPC showed low early strength development as compared to OPC mortars. However, the strength development at 28d was appreciable. The development of strength increases with the level of replacement of OPC and the composition of 40/60 (Ash/OPC) was the most favourable. Composition of 35/65 (Ash/OPC) gave anomalous results at 28d probably due to the variation of uniformity of rice husk ash produced from experimental kiln in different trials. Mortar strengths comparable to OPC mortars could be achieved by changing the W/blend ratio by adding some plasticising agent with water reducing properties.

Conditions adopted for the formation of active silica present in the ash fired in the experimental kiln were not completely satisfactory. Rice husk fired in the experimental kiln exhibited a wide variation in amorphous silica from 24% - 45% based on the solubility test. Considering the above, following modification to the experimental kiln are recommended: (i) In order to reduce the temperature difference observed between the centre of the kiln and kiln walls, a metal netting to be fixed at the periphery of the kiln (keeping a 75mm gap between the netting and the inner faces of the kiln walls). This would facilitate greater access of air for improved burning of rice husk near the kiln walls. (ii) A metal netting to be fixed at the bottom of the kiln to maintain efficient contact of air with the rice husk at the bottom of the kiln and to effect easy removal of rice husk ash from the kiln. (iii) A reduction in the capacity of the kiln (to ensure consistency of product).

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