The Potential of Arecaanut Husk Ash as Supplementary Cementitious Material

Neelu Das\textsuperscript{1c} and Shashikant Singh\textsuperscript{2}

\textsuperscript{1} Department of Civil Engineering, Assistant Professor, CIT Kokrajhar, Kokrajhar, Assam 783370, INDIA
\textsuperscript{2} Department of Civil Engineering, Professor, NERIST, Nirjuli, Arunachal Pradesh 791109, INDIA

Received: 15/06/2015 – Revised 01/08/2015 – Accepted 15/08/2015

Abstract

This paper reports the recycling of areca nut husk ash as supplementary cementitious material in respect of cement mortars. The mixes are prepared with five percentages (0, 10, 15, 20, and 25) of areca nut husk ash as partial replacement of portland cement. The properties investigated are; chemical composition, particle size, presence of crystalline matter, compressive strength, water absorption and sorption. Mortar cubes are tested for compressive strength up to the age of 56 days, whereas water absorption and sorption tests are carried out at the age of 28 days. Test results have shown that areca nut husk ash is an effective pozzolan up to the optimal replacement ratio of 20% cement with better water absorption characteristics.

Keywords: Areca nut husk ash; compressive strength; sorptivity; water absorption; x-ray diffraction

List of abbreviations used

\begin{tabular}{|c|p{8cm}|}
\hline
ANA & Areca nut husk ash \\
IS & Indian Standard \\
OPC & Ordinary portland cement \\
\hline
\end{tabular}

1. Introduction

Ordinary Portland cement is the most common type of cement in general use around the world because of its high compressive strength. The current production rate of OPC cement of the world is nearly 3.4 billion metric tons per year [1]. The continuously increasing demand of concrete will raise this figure to more than 5 billion metric tons by 2030 [2]. Although the raw materials for making OPC are readily available in most countries, search for new and viable alternative is important for conservation of natural resources, reduction in the manufacturing cost and environmental burden as OPC production is still responsible for 7-10\% of global CO\textsubscript{2} emission.
These alternative materials are generally selected on the basis of additional functionality that they offer and their cost effectiveness [3]. Typical examples are fly ash [4], slag cement (formerly called ground, granulated blast furnace slag) [5], silica fume [6], rice husk and egg shell. According to Ganesan et al. [7] up to 30% of rice husk ash could be advantageously blended with cement without adversely affecting the strength and permeability properties of concrete. Gowsika et al. [8] reported that replacement of 5% eggshell powder with 20% microsilica can be added without any reduction in compressive strength properties of conventional cement. A similar conclusion was reported by Yerramala et al. [9] where it was found that when eggshell powder are added in 5% partial replacement of cement, the compressive strength increases than control concrete at 7 and 28 days of curing ages.

Figure 1: Photograph of (a) green arecanut, (b) ripe arecanut and (c) dried arecanut

The areca nut (Figure 1) is the seed of the areca palm (Areca catechu) and extensively cultivated in India. The nut is used mainly for masticatory purpose with betel leaf (piper betle) and its husk has no conventional use. The quantity of areca nut husk obtained from an areca nut garden is approximately 5.5 - 6 metric tonnes per hectares per year, and this creates a major problem in disposal. India ranks first in the areca nut production in the world having around 4 hundred thousand hectares area with 4.78 hundred thousand tonnes production [10]. The husk of the areca nut is removed before it is sold (in dry form) and consumed, which leads to a large quantity of this bio-waste unutilized. The areca nut husk is fibrous and constitutes 50-75% of the total volume and weight of the fruit. The husk fibers are composed of cellulose with varying proportions of hemicellulose, lignin, fat and wax [11] and the average fiber length is 4 cm and is too short compared to other biofibers. Non-wood biofibers are classified into three categories according to their origin as: bast (jute, hemp, vine), leaf (pineapple, banana, sisal) and seed/fruit (coconut, cotton, palm). The advantage of biofibers over synthetic fibers (glass, carbon, polymer and aramid fibres) are the acceptable specific strength and other mechanical properties, low cost, low density, recyclability and biodegradability [12]. Fiber reinforced cement composite materials have found increasing applications in building, road, dam, bridge etc [13]. Now a day, biofibers are utilized as modifier in bituminous mixes.

Previous research efforts on the use of areca nut coir on lateritic soil stabilization for low volume pavements, resulted in medium improvement in the soil properties and the optimal content was found to be 0.6% by weight of soil [14]. To this date, no study has been reported on the usage of areca nut husk ash as supplementary cementitious material. The objective of this investigation is to evaluate areca nut husk ash as supplementary cementitious material and to identify the optimal level of replacement.
2. Materials and Method

2.1. Materials used

Ordinary Portland cement of 43 grade manufactured by Dalmia Cement (Bharat) Ltd conforming to IS: 8112-2013 [15] was used. Local clean river sand passing through 1.18 mm sieve conforming to grading zone III of IS: 383-1970 [16] was used as fine aggregate.

Areca nut husk were collected from areca nut gardens of Kokrajhar District of Assam, India (latitude 24.60°N and longitude 90.27°E). The husk were cleaned with water, dried in sun and incinerated in electric muffle furnace at a rate of 10°C per min up to 700°C for 6 hours to remove volatiles and residue carbon. After the burning process is completed, the ash was left to cool down to room temperature (Figure 2). In order to achieve fineness comparable to OPC, the burned ANA was grounded in a ball mill for 30 minutes and screened through 150µ sieve (as per IS: 1727-1967 [17]). The sieved ash was stored immediately in air tight containers to avoid pre-hydration.

![Figure 2: Steps of areca nut husk ash preparation (a) initial dry form, (b) after muffle furnace and (c) after grinding and sieving](image)

2.2. Physical and chemical analysis of ANA

The mineralogical composition of ANA was determined by X-ray diffraction analysis (Rigaku, Ultima IV, Japan). Chemical composition of the ash were determined as per IS: 1350(Part III)-1969 [18], IS: 1355-1984 [19] and Vogel’s text book of quantitative inorganic analysis for the preparation and storage of analysis solution [20].

Physical properties such as specific gravity, soundness, fineness by sieving were determined as per IS: 1727-1967 [17].

2.3. Blended cement

ANA blended cement were prepared by replacing OPC with 10, 15, 20, 25 wt % of ANA in dry condition along with control (Table 1). Mix proportion used for preparation of mortar cubes was composed of one part of cement plus ANA, three parts of standard sand by mass and (P/4 + 3.0) percent water of combined mass of ANA, cement and sand.(P is the percentage of water required to produce a paste of standard consistency as per IS:1489(Part-1)-1991 [21].
Table 1: Blended ratios of mortars

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Blending ratio (By weight %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A0</td>
<td>100% OPC + 0% ANA</td>
</tr>
<tr>
<td>A10</td>
<td>90% OPC + 10% ANA</td>
</tr>
<tr>
<td>A15</td>
<td>85% OPC + 15% ANA</td>
</tr>
<tr>
<td>A20</td>
<td>80% OPC + 20% ANA</td>
</tr>
<tr>
<td>A25</td>
<td>75% OPC + 25% ANA</td>
</tr>
</tbody>
</table>

2.4. Consistency and setting time of blended cement

The water consistency of ANA pastes and control were worked out by the Vicat apparatus method in accordance with IS: 4031 (Part-4)-1988 [22]. Then the pastes having normal consistency were used to determine initial and final setting time in accordance with IS: 4031 (Part 5)-1988 [23].

2.5. Compressive strength of blended cement

Mortar cubes (area of face 50 cm²) were prepared and cured as per IS:1489(Part-1)-1991 [21]. After removal from moulds at the age of 24 hours, mortar cubes were submerged immediately in clean fresh water and kept there until the testing is undertaken. The water in which the cubes were submerged was maintained at a temperature of 27°C and renewed every seventh day. For each substitution ratio 18 mortar cubes were prepared for compressive strength testing, conducted at the age of 7, 14, 28 and 56 days. Compressive loading tests on concretes were conducted on a compression testing machine of capacity 500 kN with a resolution of 2kN. For the compressive strength test, a loading rate of 140 kg/cm²/min was applied as per IS: 1727 – 1967 [17].

2.6. Water absorption

To determine the water absorption of the specimens, three cubes from each series after 28 days of curing were dried in hot air oven at 105°C for 24 hours. After mass stabilization its weight was taken as dry weight (W₁). Then the cubes were again submerged in water for another 24 hours and this weight was taken as wet weight (W₂) of the specimen. The water absorption was obtained as a percentage of initial mass as below:

\[
\text{Water absorption (\%)} = \frac{W₂ - W₁}{W₁} \times 100
\]

2.7. Sorptivity

Sorptivity was also measured on each series of specimens after 28 days of curing. Water was used as the test fluid. After being dried three cubes per specimen were drowned in a capillary chamber with water level not more than 5 mm above the base of specimen. They were coated with waterproof enamel paint on their lateral surfaces in order to ensure water absorption from its base only. The masses of the specimens were measured by weighing at regular intervals up to 120 minutes on a balance weighting up to 0.1 mg. A schematic diagram of the test is shown in Figure 3.
The Potential of arecanut husk ash as supplementary cementitious material

Figure 3: Schematic diagram of water absorption test

The sorptivity coefficient was obtained using the following expression:

\[
\frac{Q}{A \rho} = S \sqrt{t}
\]

Where \( Q \) is the amount of water absorbed, \( A \) is the surface area of the specimen through which water penetrated, \( t \) is the elapsed time, \( \rho \) is the density of water and \( S \) is the sorptivity coefficient of the specimen (mm/min\(^{0.5}\)).

3. Results and discussion

3.1. Chemical and mineralogical composition of material

Table 2: Physical and Chemical properties of cement and ANA

<table>
<thead>
<tr>
<th>Properties</th>
<th>Cement</th>
<th>ANA</th>
</tr>
</thead>
<tbody>
<tr>
<td>A) Physical Properties</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Specific Gravity</td>
<td>3.15</td>
<td>2.97</td>
</tr>
<tr>
<td>Fineness</td>
<td>300.6 m(^2)/kg</td>
<td>79%</td>
</tr>
<tr>
<td>Le Chatelier Expansion</td>
<td>0.5 mm</td>
<td>0.5 mm</td>
</tr>
<tr>
<td>(Residue on 75µm sieve)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>B) Chemical Composition</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SiO(_2) (Silica)</td>
<td>19.62%</td>
<td>28.44%</td>
</tr>
<tr>
<td>Al(_2)O(_3) (Alumina)</td>
<td>5.62%</td>
<td>3.64%</td>
</tr>
<tr>
<td>Fe(_2)O(_3) (Iron Oxide)</td>
<td>5.33%</td>
<td>1.91%</td>
</tr>
<tr>
<td>CaO (Calcium Oxide)</td>
<td>61.24%</td>
<td>2.71%</td>
</tr>
<tr>
<td>MgO (Magnesia)</td>
<td>0.88%</td>
<td>3.87%</td>
</tr>
<tr>
<td>SO(_3) (Sulphur Trioxide)</td>
<td>2.60%</td>
<td>7.80%</td>
</tr>
<tr>
<td>Na(_2)O (Sodium Oxide)</td>
<td>--</td>
<td>0.24%</td>
</tr>
<tr>
<td>K(_2)O (Potassium Oxide)</td>
<td>--</td>
<td>26.52%</td>
</tr>
<tr>
<td>Lime saturation factor</td>
<td></td>
<td></td>
</tr>
<tr>
<td>(CaO - 0.7SO(_3))/ (2.8SiO(_2)+1.2Al(_2)O(_3)+0.65Fe(_2)O)</td>
<td>0.91</td>
<td>-0.032</td>
</tr>
<tr>
<td>Ratio of Alumina/Iron Oxide</td>
<td>1.05</td>
<td>1.90</td>
</tr>
<tr>
<td>Total Loss on Ignition</td>
<td>2.06%</td>
<td>23.50%</td>
</tr>
</tbody>
</table>

The results of tests on chemical composition of the ANA are reported in Table 2. It can be seen that the ash contains silica as primary compound. In addition it also contains alumina (Al\(_2\)O\(_3\)),...
iron oxide (Fe₂O₃), calcium oxide (CaO) and significant amount of alkali (K₂O and Na₂O). The sum of SiO₂, Al₂O₃ and Fe₂O₃ is more than 45% of the overall material composition.

Figure 4 shows the spectrum of ANA which was analyzed by X-ray diffraction test. ANA has a highly crystalline structure with silica in the form of quartz, potassium oxide and calcite. Silica and potassium oxide are present as major constituent with lesser amount of calcite. Diffraction peaks of quartz (silica) appeared at 21.040°, 26.720°, 60.120°, 66.500° and 73.780°, peaks of potassium oxide were 28.460°, 30.960°, 32.400°, 34.180° and 40.620°, whereas that of calcite were 43.460°, 50.300° and 58.760°.

Figure 4: X-ray diffractogram of ANA3.2. Consistency and setting time of blended cement

The graph of cement replacement level versus consistency (Figure 6) indicates that the water required for normal consistency increased with the increase of cement replacement level. It is due to the high hygroscopicity areca nut husk ash. The various oxides of ANA including calcium (Ca), potassium (K) and silicon (Si) are basic or amphoteric and produce hydroxides upon their reaction with water. Amongst these, potassium hydroxide (KOH) is known to be highly hygroscopic compound and composition of potassium oxide (K₂O) in ANA is 26.52%.

From the chart of initial and final setting time (Figure 7) it is seen that addition of ANA retarded the setting and this is due to the absorption of water at the surface of ANA. Increase in the proportion of ANA, increased the absorption of water and therefore higher amount of water retarded the setting time. The values are found to be well within the permissible limits as per IS: 8112-1989 [15].
The Potential of arecanut husk ash as supplementary cementitious material

Figure 5: Particle size distribution of ANA with dry sieving

Figure 6: Water consistency of ANA blended cement

Figure 8 shows change in compression strengths for control and ANA blended cement mortars with age. It can be seen that strength increased with the curing age for all the samples. Control mortar gained 49% at day 7 and 71% after 14 days, 92% after 28 days of curing over its 56 days compressive strength. The ANA blended mortars gained 49-55% at day 7, 73-77% after 14 days, and 90-93% after 28 days of curing than their corresponding 56 days strength. It is clear from the observation that strength enhancement in 10% replacement of ANA is higher than the cement mortar between 7 and 56 days and the optimal level can be considered up to 20% replacement of ANA to OPC.
3.4. Water absorption and sorptivity

Results of water absorption and sorptivity are reported in Table 3. It is seen that mortar mixes containing ANA produces lower absorption and sorptivity compared to the mixture containing no ANA (control). This may be due to because ample water was available for hydration and particle surface area became less critical.

Table 3: Water absorption and sorptivity test results of specimen

<table>
<thead>
<tr>
<th>Specimen ID</th>
<th>Water absorption (%)</th>
<th>Water sorptivity (mm/min\textsuperscript{0.5})</th>
</tr>
</thead>
<tbody>
<tr>
<td>A0</td>
<td>7.19</td>
<td>0.143</td>
</tr>
<tr>
<td>A10</td>
<td>7.02</td>
<td>0.139</td>
</tr>
<tr>
<td>A15</td>
<td>6.94</td>
<td>0.131</td>
</tr>
<tr>
<td>A20</td>
<td>6.78</td>
<td>0.122</td>
</tr>
<tr>
<td>A25</td>
<td>6.47</td>
<td>0.116</td>
</tr>
</tbody>
</table>
4. **Conclusions**

Based on the results of this study, the following conclusions can be drawn:

- 7-, 14-, 28- and 56-day compressive strength obtained for control mortar cubes were 23.2-, 33.6-, 43.2- and 47.2-MPa respectively. A significant increase in compressive strength of 3.44%, 7.14%, 1.85% and 3.38% at 7-, 14-, 28- and 56-day respectively was observed for 10% replacement of cement (ANA 10) than the control samples. However replacement with 15% and 20% ANA resulted in close comparable compressive strength. This increase in compressive strength is due to the excess amount of silica in ANA.
- Setting times of the blended pastes were extended by incorporation of ANA and this will be helpful for concrete that has to be transported to long distance and to offset the effect of high temperatures.
- The soundness of the blended pastes were at 0.50 mm for all replacement level of ANA, which indicates no excessive amounts of free lime or magnesia.
- ANA mortar resulted in lower water absorption and sorptivity than that containing no ANA (control), which are important features of mortar resistance to exposure in aggressive environment.
- ANA has great potential to improve mechanical and durability properties as an innovative supplementary cementitious construction material that will reduce the cement cost on the construction of massive structures, but judicious decisions are to be taken by structural engineers evaluating the bond strength in terms of pull-out test, beam bending test and collapse test.

**References**


[17] IS: 1727-1967, Methods of Test for Pozzolanic Materials, Bureau of Indian standards, New Delhi, India.
[18] IS: 1350 (Part III)-1969, Methods for Test of Coal and Coke - Determination of Sulphur, Bureau of Indian standards, New Delhi, India.