Characterisation of prepared rice husk ash and its effects on strength development in recycled aggregate concrete

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Abstract: Waste and by-product materials have a negative impact on the environment due to the pollution associated with them. The conversion of these materials from useless or harmful to valuable substances by, for example, incorporating them into concrete, can thus be considered to be an issue worthy of consideration in the search to reduce this impact. This study aims to prepare and characterise the ash produced from rice husk wastes to discover the ash's effect when used as a cement replacement in recycled aggregate concrete in the presence of styrene butadiene rubber (SBR). The rice husks were burned in the oven at 550 to 650 ºC for two hours. Afterward, the rice husk ash (RHA) was characterised using X-rays, FT-IR, and grain size analysis tests. Thereafter, four concrete mixes, 0% RHA + 0% SBR, 1% RHA + 1% SBR, 3% RHA + 1% SBR, and 0% RHA + 1% SBR were made. The RHA was used as cement replacement, while the SBR was used as mixing water replacement, with percentages measured by weight for both materials. Crushed clay bricks were employed as coarse aggregate for all mixes. Compressive strength tests were carried out at 7 and 28 days. The X-ray and FT-IR results demonstrate that an amorphous form of silica with good purity was produced from the prepared RHA. For concrete mixes, the results indicate an important enhancement in compressive strength obtained by using RHA.

Keywords: rice husk ash, characterisation, recycled aggregate concrete, compressive strength

1. Introduction
Pollution is a huge problem in the modern world, and many researchers and environmentally concerned people have thus focused their attention on ways to reduce it. Recycling has become required in human culture as a result of the accumulation of various wastes, such as metallic materials, glass, papers, and plastics [1,2]. By-products of many processes also present accumulated wastes that can cause environmental pollution, such as rice husks, sawdust, date stones, and crushed bricks [3–5]. Recycling of by-products is thus a smart attempt to change valueless waste materials into valuable renewable materials. Rice husk is a by-product of rice milling and it is considered to be an agricultural waste. The large volume of production of rice, approximately 600 million tons/year, means that there is a large amount of rice husk waste produced, representing about 20 wt.% of total rice production. Rice husk is composed of roughly 32% cellulose, 21% hemicelluloses, 22% lignin, and 15% mineral ash. The mineral ash is composed of 55 to 97 % SiO2, with several other constituents such as K2O, MgO, Fe2O3, CaO, and Al2O3 [6–9]. The burning of rice husk has specific benefits, changing rice husk from a valueless by-product accumulated by the millions of tons, which presents an environmental pollution problem in rice-producing countries to an important source of valuable materials such as silica [10,11]. As silica is the main component of rice husks ash, the ash can be used as a pozzolanic material in cement and as an aggregate in the production of bricks and blocks [12–14]. Crushed clay brick (CCB) is considered to be a waste material of the clay brick industry as well as construction and demolition; several researchers have thus focused their attention on converting this waste to a useful renewable material that can be used in concrete production. CCB can thus be used as an alternative aggregate for producing concrete [15,16].
Tangchirapat studied the influence of ground rice husk-bark ash (RHBA) on the mechanical properties of recycled aggregate concrete [17]. He reported that using 100% recycled aggregate as coarse aggregate replacement decreased the compressive strength in comparison to normal aggregate concrete. Additionally, it was revealed that replacing cement with RHBA enhanced the compressive strength compared to concrete without RHBA.

Alwan studied the effects of RHA on the compressive, splitting, and flexure properties of cement mortar [12]. The proportions investigated were 3%, 4%, 5%, and 6%, used as replacement of cement weight. The maximum enhancement ratio (about 86%) in compressive strength at 7 days as related to the control sample was found by substituting in 4% RHA, while replacing cement with 3% RHA achieved a 64% enhancement in compressive strength for the same test age.

The present paper focuses on recycling agricultural wastes, such as rice husks, and industrial wastes, such as broken bricks. The preparation and characterisation of the RHA and investigation into the effects of adding it as a cement replacement for recycled aggregate concrete are the main aims of this work. The purpose of using these recycled materials is to produce concrete with a lower environmental impact.

2. Experimental work

2.1 Rice husk ash (RHA) characterisation details

Rice husk ash was prepared by putting rice husks in the oven at 550 to 650 °C for two hours, then leaving it for about 24 hours to cool. Many researchers [18,19] have reported that burning the rice husks within the temperature range 500 to 650 °C is preferred to avoid the transformation of silica from its amorphous (active) state to its crystalline form. After gradual cooling, the ash was ground and prepared for the characterisation tests.

The rice husks ash was characterised using X-rays, FT-IR, and grain size analyses tests. The X-ray was performed using a Shimadzu XRD-6000, Germany. The measured conditions were target Cu, voltage of 40 kV, speed of 6 deg/min), and range of 20 to 70 deg. The Bruker Optik GMBH and Bettersize2000 Laser Particle Size Analyzer instruments were used for the FT-IR and grain size analyses tests, respectively.

2.2 Concrete work details

Sulphate-resistant cement (Type V) was used as a binding material for all concrete mixes. The chemical properties of the cement were in agreement with Iraqi specifications I.Q.S. 5/1984 [20], and are illustrated in table 1. Natural sand conforming to Iraqi specifications I.Q.S. 45/1980 [21] (see table 2) was used as a fine aggregate in this study. Clay bricks were crushed and graduated to be used as coarse aggregate for the concrete mixes; the grading of this aggregate is shown in table 3. The prepared RHA was used as a cement replacement. Additionally, Styrene butadiene rubber (SBR) was employed as a replacement (by weight) for mixing water. Four concrete mixes were made for this study in weighted proportions of 1:1.5:3 (binder: fine aggregate: coarse aggregate), as detailed in table 4.

In order to prevent the absorption of mixing water by the crushed clay brick, the latter was submerged in water for 24 hours before mechanical mixing began. The mixing procedures were as follows: the dried materials were fed into the mixer and it was allowed to operate for a few revolutions before the water (and SBR, if any) was added. The mixing process was continued until a homogeneous consistency was reached. The total mixing time was generally about 5 minutes. After that, the fresh concrete was immediately cast in 15 cm standard cubic moulds for the compressive strength test. The fresh concrete was compacted using a damping stick, and the specimens were covered and left to cure in the moulds for 48 hours, before these were lifted and placed in tap water tanks until testing. Two test ages were examined for concrete compressive strength: 7 and 28 days. Three specimens were made for each case.
Table 1. The chemical composition of cement.

| Oxides Composition | Content, % | Iraqi specifications (No. 5/1984), % |
|--------------------|------------|-------------------------------------|
| CaO                | 63.67      | -                                   |
| SiO₂               | 22.15      | -                                   |
| AL₂O₃              | 4.09       | -                                   |
| Fe₂O₃              | 4.95       | -                                   |
| SO₃                | 2.47       | ≤ 2.5                               |
| MgO                | 1.93       | ≤ 5                                 |
| L.O.I              | 1.97       | ≤ 4                                 |
| L.S.F              | 0.88       | 0.66 – 1.02                         |
| I.R                | 0.82       | ≤ 1.5                               |

Table 2. Grading of the sand used.

| Sieve opening (mm) | Accumulative passing, % | Accumulative passing according to Iraqi specifications (No. 45/1984), % |
|--------------------|--------------------------|---------------------------------------------------------------------|
| 10                 | 100                      | 100                                                                 |
| 4.75               | 98                       | 90 – 100                                                            |
| 2.36               | 86                       | 75 – 100                                                            |
| 1.18               | 71                       | 55 – 90                                                             |
| 0.60               | 49                       | 35 – 59                                                             |
| 0.3                | 19                       | 8 – 30                                                              |
| 0.15               | 2.5                      | 0 – 10                                                              |
Table 3. Grading of the crushed clay brick.

| Sieve opening (mm) | Accumulative passing, % |
|--------------------|-------------------------|
| 37.5               | 100                     |
| 20                 | 86                      |
| 10                 | 51                      |
| 5                  | 29                      |
| 0.075              | 0                       |

Table 4. Mixes details.

| Mix symbol | Water/binder | Cement, kg/m³ | RHA, kg/m³ (% by wt. of cement) | Sand, kg/m³ | Crushed clay brick, kg/m³ | SBR, kg/m³ (% of mixing water) |
|------------|--------------|---------------|-------------------------------|-------------|---------------------------|-------------------------------|
| A          | 0.4          | 400           | 0                             | 600         | 1200                      | 0                             |
| B          | 396          | 4 (1%)        | 600                           | 1.6 (1%)    |
| C          | 388          | 12 (3%)       | 600                           | 1.6 (1%)    |
| D          | 400          | 0             | 600                           | 1.6 (1%)    |

3. Results and discussions

3.1 Rice husk ash characterisation

3.1.1 X-ray diffraction.

The X-ray pattern of the prepared ash is shown in figure 1. The test data indicates that the main compound sensed by the device is silicon dioxide. The X-ray diffraction also shows an amorphous peak at the Bragg angle (2θ = 22). These observations suggest that a high purity amorphous silica was prepared. Similar results were recorded by [22].

3.1.2 FT-IR.

Figure 2 shows the FT-IR spectrum of the RHA. The red line refers to the prepared ash while the blue line refers to the standard spectrum of the amorphous silica provided by the device. It is clear that there is a good match between the two spectra, which means that amorphous silica was obtained. Different IR bands can be seen along the spectrum of the prepared RHA, and the IR band at 2,375 cm⁻¹ is a result of the stretching vibration of the O-H and adsorbed water in the amorphous silica, while the IR band at 1,577 cm⁻¹ is due to the H₂O bending vibrations. The wide and very strong band at 1,049.5 cm⁻¹ is due to the stretching vibration of the Si-O-Si [23,24]. These results agree with [22] and [25].

3.1.3 Grain size analyses.

The grain size analyses of the RHA are illustrated in table 5. It is obvious that more than 60% of the particles are less than 20 μm in size and about one-half of the particles are smaller than 10 μm in size. This characteristic is important when the RHA is used as an admixture in concrete, as planned in the study. The small size gives these particles (when present in a sufficient amount) the ability to fill the pores between cement particles leading to densifying and strengthening of the microstructure, which increases the concrete strength.
3.2 Concrete results

3.2.1 Compressive strength.
Compressive strength results for ages 7 and 28 days are shown in figure 3. It can be seen that replacing cement with rice husk ash and the mixing water with SBR, as in the B and C mixes, leads to significant increments in compressive strength for both considered ages (7 and 28 days). However, the enhancement is more pronounced at the higher levels of replacement of ash (3%) than at lower replacement levels (1%) in comparison to the control mixture. The chemical activity of the rice husk ash that is represented by the reaction of the amorphous SiO$_2$ with the Ca(OH)$_2$ released from cement hydration plays an important role in increasing the compressive strength of the concrete. In addition to the good packing of the small particles (as proved by the grain size analyses test) which densifies the microstructure of mortar, it has been reported previously that the presence of amorphous silica is accompanied by an enhancement of the interface transition area leading to the production of a microstructure with a higher density [26]. The maximum improvement was found after replacing 3% cement with rice husks ash, with improvements of about 45% and 53% for 7 and 28 days ages respectively in comparison to the control mix.

On the other hand, replacing the mixing water with SBR for the D mix had a negative effect on compressive strength, with a loss of about 3% at the later age (28 days), and only comparable values at the earlier age (7 days) in relation to control mix. This may be attributed to the relatively low replacement percentage of water with SBR in this study.
Table 5. Particle size analysis of the rice husk ash.

| Size (µm) | Passing, % |
|-----------|------------|
| 200       | 100        |
| 100       | 99.75      |
| 75        | 97.67      |
| 45        | 90.19      |
| 20        | 62.42      |
| 10        | 48.05      |
| 5         | 36.77      |
| 2         | 7.5        |
| 1         | 3.96       |
| 0.5       | 0.32       |

Figure 3: Compressive strength results at 7 and 28 days.

4. Conclusions
The following conclusions can be drawn from the study:

1. The FT-IR test shows high correspondence levels between the produced silica and the standard spectrum of amorphous silica.
2. The X-ray pattern supports the FT-IR findings by highlighting the presence of amorphous silica. The X-ray pattern indicates that high purity silica is obtained in the prepared ash.
3. Under the specified preparation conditions, it is possible to produce RHA with an acceptable quality and amorphous (active) properties.
4. Though used only in relatively small proportions (3%), rice husk ash can improve the strength of recycled aggregate concrete.

5. References
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