Effect of Carbonization Temperature on Wear Rate Behaviour of Rice Husk Ash Reinforced Epoxy Composites

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Abstract
The effect of carbonization temperatures on wear rate behaviours of different volume fractions of rice husk ash epoxy composite was studied. Rice husk ash carbonized at 850, 900, and 950°C were reinforced in epoxy resin. Rice husk ash epoxy composite containing 10%, 20%, 30% and 40% rice husk ash was synthesized using manual stirring method and specimens were prepared for wear studies. Dry wear behaviour of sample was studied against abrasive paper using a pin-on-disc machine. Wear rate and specific wear rate behaviours of different samples have shown near uniform behaviour. The 950°C carbonized ash showed steady wear rate and specific wear rate behavior for all ash contents. The composites with 30% and 40% ash carbonized at 950°C showed minimum wear rates of $1.03 \times 10^{-5}$ N/m and $0.98 \times 10^{-5}$ N/m at 5N load respectively. The wear behaviours of 950°C carbonized ash were better than those of 850°C and 900°C carbonized ash due to the degree of alteration in the structure of the silica.

Keywords
Rice husk; Carbonization temperature; Wear rate; Specific wear rate.
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Introduction

In recent time there is growing interest in developing composites using various recycled wastes [1], especially in developing composites using most environmentally friendly agro-wastes as reinforcing fillers and thermosetting polymers as matrixes. Recent investigations of polymer-based composite materials have opened new routes for polymer formulations and have allowed the manufacture of new products with optimal properties for special applications [2, 3].

Rice husk (or hulls) is an agricultural residue. In the urban and rural areas of Nigeria, local milling is mostly done by women. The milling generates a waste material - the husk surrounding the dried rice paddy. They mainly use firewood as heat source and as such one hundred percent of the rice husk from the mill is a waste. About 20% of a dried rice paddy is made up of the rice husks although the percentage differs by variety. About $10^8$ tons of rice husks are generated annually in the world [4]. That makes rice husk one of the largest readily available, but also one of the most under-utilized resources. Rice husk has a very low nutritional value and as they take very long to decompose are not appropriate for composting or manure. Therefore the $10^8$ million tons of rice husk produced globally constitute environmental pollution if not disposed of properly. The global environmental awareness has led to researching ways of an effective utilization of rice by-product [5, 6]. Various works has been done with regards to the usage of environmental friendly materials as reinforcement in recent years like jute [7], cotton [8, 9], sugarcane [10], oil palm [11], coir [12], kenaf [13], betel-nut [14] and wood flour [15].

In developing countries like Nigeria, proper utilization of rice husk has not been given due attention. The rice husk therefore constitutes an environmental nuisance as they form refuse heaps in the areas where they are disposed. Rice husk is particularly valuable due to its high content of amorphous silica [6]. The RH ceramics has been developed by carbonizing rice husk as the main material at 900°C. It was found that RH ceramics contain amorphous silica in addition to amorphous carbon as the main constituents [16]. The silica in the rice husk undergoes structural transformations depending on the temperature of the heat treatment. By heating at higher temperatures, the unburned carbon can be removed from the ash [17], but this leads to the crystallization of the ash from amorphous silica into cristobalite or tridymite. At lower temperature, the amorphous nature of rice husk ash silica will occur [18].

The most common form of crystalline silica found is quartz [19]. But more research
has been focused on the formation of cristobalite and tridymite [20]. The transformation has been reported that $\alpha$-quartz can be form at below 573°C, $\beta$-quartz at 573-870°C, $\beta$-tridymite at 870-1470°C, and $\beta$-cristobalite at 1470-1710°C [19]. These types of silica have different properties and it is important to produce ash of the acceptable specification for the particular end use. It is possible that such phase transformations could have significant effect on wear rate behaviours of RH ceramics.

The purpose of this investigation was to study the effect of carbonization temperature on the wear rate behaviour against abrasive under dry condition.

**Material and Method**

The Raw materials used in this experimental work are rice husk, epoxy resin 3554A and hardener, Abrasive paper, adhesive tape, jig box, electronic weighing machine, grinding machine and pin-on-disk machine.

**Preparation of Rice Husk Ceramic Ash**

The rice husk was collected from a local rice mill at Bosso Town in Niger State, Nigeria. The rice husk was washed thoroughly with clean water to remove major impurities like clay and rock impurities and subsequently dried in an oven at 120°C for 24hrs to remove water content. The dried RH was taken in crucible and placed in a furnace for 1 h at 500°C to get a black ash. The black ash was ground into powder and only fractions passing BS sieve No. 200 (75μm) was heated again to temperature of 850°C in a step of 5°C each minute for complete combustion to white ash powder. At this temperature one hour soaking time was allowed. After the soaking time 24hours cooling was allowed so that the furnace comes to room temperature. Then the carbonized rice husk white ash was taken out from the furnace and kept in a sealed envelope for further use without additional treatment. The carbonization process was repeated for temperatures of 900°C and 950°C respectively.

**Preparation of Composites**

After the ash material was prepared and the resin to hardener weight ratio carefully
mixed was 100:50, the composites with varying degrees of ash percentage (i.e. 10, 20, 30 and 40) were prepared. The resin and ash was mixed via manual stirring method for five minutes and the mixture was poured into a jig box to form cylindrical pins of 30 mm long and 10 mm diameter. Load was applied upon it and was left for 24 hours to cure in the box jig at room temperature (25°C). After curing the samples were taken out from the box, finished ground to required shape, sizes and placed in a sealed envelope for wear test.

**Determination of Wear Values**

Wear test was carried out on pin-on-disc machine under dry conditions at room temperature (25°C). The sample was mounted vertically on a still vice such that its face pressed against rotating disc. The disc used was carbon steel (Fe-2.3%Cr-0.9%C) hardened to 65 HRC, 120mm diameter and 8mm thick. Abrasive paper of 400 grades (grit-23 μm) was fixed on the rotating disc using double-sided adhesive tape. The holder along with the specimen (pin) was positioned at a particular track diameter. A track radius of 40mm was selected for this experiment and was kept constant for the entire investigation. For each test new abrasive paper was used and sample was abraded under a constant speed and time of 0.633 m/s and 300s.

During experiment the specimen remains fixed and disc rotates. Load was applied through a dead weight loading system to press the pin against the disc. The speed of the disc or motor rpm was varied through the controller and interval of time set by the help of timer provided at the control panel. On completion of each sample testing the specimen was removed, cleaned with acetone, dried and weighed to determine the mass loss due to wear. The difference in the mass measured before and after the test gives wear of the specimen. The mass loss of the pin (specimen) was measured in an electronic weighing machine with a least count of 0.001 g. The ratio of mass losses to sliding distance was defined as wear rate. The wear test was carried out by varying the normal load and keeping sliding velocity and time constant. The volumetric wear rate of the composite was defined as the ratio of mass loss to density ($\rho$) and the abrading time (t). For characterization of the wear behaviour, the specific wear rate was employed. This was defined as the volume loss of the composite per unit sliding distance and per unit applied load.
Results and Discussion

Figures 1-4 shows the variation of wear rate against loads (5, 10, 15 and 20N) for different carbonization temperatures and ash contents. It may be noted in figures 1-4 that the wear rate of all composite samples increased with normal load with the exception of 950°C carbonization temperature in figure 1. The finding is in agreement with that of [21].

The wear rate for 20% and 40% ash contents in figure 2 and 4 showed unsteady behaviour compared to 10 and 30% ash contents in figure 1 and figure 3. In figure 1 and figure 3 the wear rate increased when the carbonization temperature increased from 850°C to 900°C and then decreased for carbonization temperature of 950°C. The 950°C carbonized ash showed lower wear rate. Comparable trend in lower wear rate for all other 950°C carbonized ash were observed. It was seen from figure 2 that with increase in ash content to 20%, the wear rate at 5N load was 0.93N/m for 850°C which was lower than 1.19N/m recorded for 950°C. It thereafter remained above 950°C values for the entire loads. The wear rate of 850°C and 900°C carbonization temperatures follows waveforms, but generally increased at higher loads. The increase in wear rate was because at higher load frictional thrust increased which resulted in increased deboning and fracture. The composites with 30% and 40% reinforcement carbonized at 950°C showed minimum wear rates of $1.03 \times 10^{-5}$ N/m and $0.98 \times 10^{-5}$ N/m at 5N load respectively.

![Figure 1](image-url)  
*Figure 1. Variation of wear rate with load at sliding velocity of 0.633 m/s and time of 300s for 10% ash content*
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Figure 2. Variation of wear rate with load at sliding velocity of 0.633 m/s and time of 300s for 20% ash content

Figure 3. Variation of wear rate with load at sliding velocity of 0.633 m/s and time of 300s for 30% ash content

Figure 4. Variation of wear rate with load at sliding velocity of 0.633 m/s and time of 300s for 40% ash content
Figures 5-8 shows the specific wear rate with 10-40% ash contents as a function of normal load for all the composites. It was seen from the plots that specific wear rate of the composite containing ash carbonized at 950°C was lower for all ash contents and load conditions compared with ash carbonized at 850°C and 900°C. A steady behaviour was observed in composites with ash contents of 10% and 30% (figure 5 and figure 7) compared to 20% and 40% ash contents (figure 6 and figure 8). The 950°C carbonized ash showed steady specific wear rate behaviour for all the ash contents.

![Figure 5](image5.png)

**Figure 5.** Variation of specific wear rate with load at sliding velocity of 0.633 m/s and time of 300s for 10% ash content

![Figure 6](image6.png)

**Figure 6.** Variation of specific wear rate with load at sliding velocity of 0.633 m/s and time of 300s for 20% ash content
The change in wear values could be the consequence of alteration in the structure of the silica. The wear rate increase of RH ceramics from 850°C and 900°C could be as a result of more β-quartz in its transition to relatively harder form β-tridymite silica. Consequently with higher carbonizing temperature of 950°C, the amount of β-tridymite silica is believed to have increased, therefore resulting in decrease of overall wear rate value. This finding is in conformity with [21], that wear resistance improvement is attributed to the combination of hard particles. The steady specific wear rate behaviour for all the ash contents at 950°C could result from the complete transition of β-quartz structure to relatively harder β-tridymite silica form.
Conclusions

From the tests conducted on rice husk ash epoxy composite as presented, the following conclusions were made:

- The rice husk an agro waste from rice mill is available in significant quantities and can successfully be utilized to produce composite by correctly bonding with epoxy resin for value added product. This will go a long way to reduce the quantity of waste in our environment.
- The increase of carbonization temperature to 950°C led to steady wear behaviour due to the structural transformations of the silica to relatively harder form for all the ash contents.
- Wear was observed to increase at higher applied load.
- The wear behaviour of 950°C carbonized ash supersedes those of 850°C and 900°C carbonized ash due to the degree of alteration in the structure of the silica.
- The optimum wear resistance property was obtained at the ash content of 10% volume of reinforcement for 900°C carbonizing temperature with 20N load.
- Minimum wear rate was observed for 40% volume of reinforcement at 950°C carbonizing temperature with 5N load.

References


