Effects of Milling Time on Nano Rice Husk Ash Particle Size

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Abstract. This research focuses on the manufacture of nano Rice Husk Ash (nRHA) by ball milling technique and critically analyzes the effect of milling time on particle size. The process starts with collection of raw rice husk from a local rice mill factory, followed by controlled incineration at a temperature of 700°C for 5 hours to get the amorphous RHA. Finally, the nano RHA is prepared by subjecting the RHA to grinding for the different period like 10, 20, 30, and 40 hrs. The particle size was analyzed with FESEM, and it was found that particles got smaller as grinding proceeded, reaching an optimal size of 28 nm following 30 h grinding. Nonetheless, prolonged grinding resulted into particle agglomeration that was caused by Van der Waal forces. Therefore, these findings are significant in that they help to comprehend the morphology changes and particle size alterations in nRHA that may be applied for different uses such sustainable construction materials.

1 Introduction

Rice husk is a by-product of rice milling, historically regarded as waste material, produced every year in millions of tons [1]. Its abundance in rice-producing regions has made it a topic of interest. RH contains a high percentage of silica, which, when transformed into Rice Husk Ash (RHA), presents an opportunity for multiple industrial applications [2]. RHA, containing amorphous silicon oxide (SiO2) or silica, is highly pozzolanic, making it a suitable material for partial replacement of Portland cement in concrete mixtures. This pozzolanic material has found successful commercial applications in several countries [1]. The composition of RHA closely resembles that of silica fume (SF). Also, RHA has the capability to generate a significant quantity of silica, typically within the range of 90% to 96% of its initial weight, with a minimal 3.8% loss [3]. Combustion of RHA at temperatures below 700 °C generates

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amorphous silica, according to [4]. Similarly, with the findings of other researchers [5][6], the maximum content is obtained between 500 and 700 °C.

In recent years, there has been an increasing research interest in nanomaterials, specifically regarding their capacity for use as ultra-fillers for optimising the compactness of concrete microstructures. especially, the use of nanofillers or ultra-filling can be seen as a technique that acts within the structural components of concrete [7]. As an accelerating additive, nano-silica can facilitate the formation of more compact structures, even during shorter curing periods. The addition of nano-silica particles to high-strength concrete made with large volumes of fly ash can also improve its short-term and long-term strength [8]. The rapid pozzolanic characteristics and notable reactivity of nano-silica improved the microstructure of the concrete, which enabled the development of compact, small-sized C-S-H gel. As a result, the concrete showed enhanced mechanical strength and decreased permeability [8].

While nRHA exhibits potential, there exists a gap in research regarding an accurate control of its particle size [9]. The control of particle size is critical for the functionality and practical uses of nRHA. Although several production methods have been investigated, attaining accurate control continues to be an enormous challenge. The present study investigates the relationship between milling time and the particle size of nRHA. By systematically varying the milling time, ball milling, which is a type of mechanical grinding, is utilised to generate nRHA with different particle sizes. Through a study of this relationship, the study aims to illuminate the variables that determine the optimum size of nRHA particle.

In order to obtain a comprehensive understanding of the impacts of different milling durations, an in-depth analysis of the morphological attributes of the resultant nRHA samples is carried out using Field Emission Scanning Electron Microscopy (FESEM). The insights gained from this analysis regarding the morphology, size, and distribution of the nRHA particles contribute significantly to the field of knowledge concerning the correlation between milling time and particle size.

2 Methodology

2.1 Collection and Preparation of Rice Husk

The primary raw material, rice husk, was diligently sourced from a local rice mill factory located in Muar, Johor, Malaysia as shown in **Fig. 1**. Subsequently, the rice husk was stored in a dry area within the UTHM laboratory. These measures were implemented to guarantee that the rice husk remained in a dry and moisture-free state.

Fig. 1 Rice husk from the factory

2.2 Combustion of Rice Husk

The controlled burning of dry rice husks was conducted in an automated furnace within the laboratory of Universiti Tun Hussein Onn Malaysia (UTHM) as shown in **Fig. 2**. This process resulted in the formation of amorphous ash, which is commonly referred to as rice husk ash (RHA). Previous studies have emphasized the importance of maintaining precise combustion conditions to enhance the amorphous silica content. Typically, the temperature parameters for achieving optimal results are reported to be in the range of 500-700°C [10] [5] [11]. Within the present system, the suggested parameters for the burning of rice husk include maintaining a temperature of 700°C over a duration of 5 hours. This process produces amorphous silica and showing a modest level of energy consumption in comparison to other methods. The graphical representation in **Fig. 3** illustrates the variations in temperature through the duration of the process.

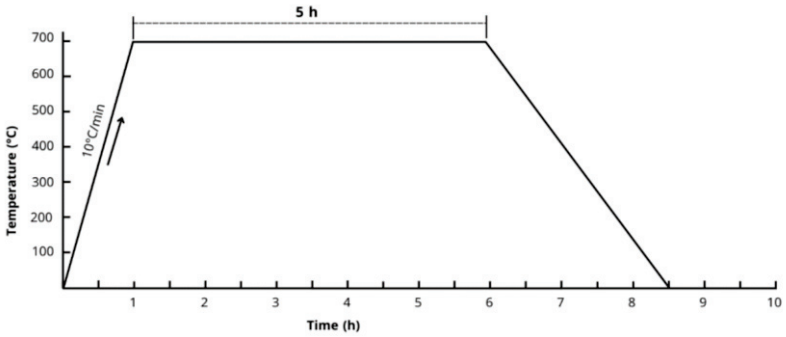

Fig. 2 Burning time and temperature

In order to start the combustion process, the rice husk was precisely weighed and positioned in an appropriate container, as illustrated in **Fig. 3**. The heating rate was continually maintained at a rate of 10°C per minute until it reached a stable condition at a temperature of 700°C, in accordance with the recommendations given in the studies by [12][13] [3]. Following this, the combustion process was carried out for a period of 5 hours at the designated temperature of 700°C. It is of interest to mention that the attainment of a target temperature of 700°C required a duration of around 70 minutes, aided by a heating rate of 10°C per minute. In contrast, the subsequent cooling phase endured for a period exceeding 180 minutes.

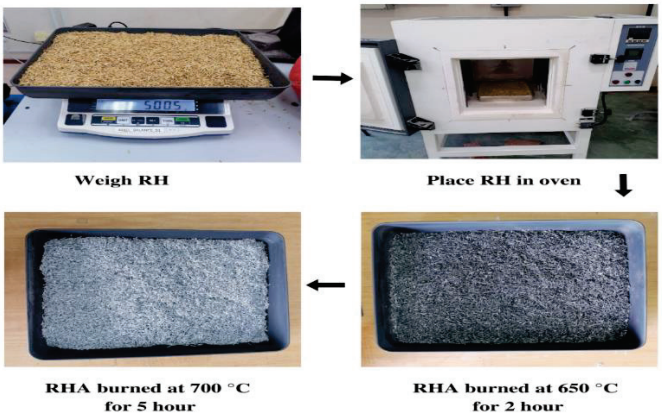

Fig. 3 process of controlled combustion of RH.

2.3 Grinding Process

The process of grinding RHA is a transformative stage that reduces its size to smaller fractions, leading to an improvement in its general properties, save for the ultimate setting time [14] . Four different grinding times were used in order to attain a smaller particle size of nano Rice Husk Ash (nRHA). The previous process was conducted within the laboratory of UTHM, using a laboratory mill grinder, as shown in **Fig. 4**. In order to ensure uniformity in the characteristics of the ground RHA, it was ensured that each grinding drum had 500 grammes of RHA for every grinding interval, in accordance with the guidelines provided by [15]. The durations of grinding covered four different periods of time, namely 10, 20, 30, and 40 hours. A thorough examination was carried out on all samples, for the determination of the most suitable duration for grinding. **Table. 1** illustrates the grinding media, which consists of drums and balls made from steel materials, used in the grinding process. The milling operation was carried out at a constant speed of 60 revolutions per minute.

Fig. 4 The process of grinding RHA in a UTHM laboratory ball mill.

Table 1 Grinding media of RHA

Drum (Steel)	200 mm Dia. x 230 mm Height	
Balls (Steel)	Diameter (mm)	Number
	25	7
	20	17
	16	18
	12	18
Milling speed	60 rev/min	

2.4 Field Emission Scanning Electron Microscopy (FESEM)

Field Emission Scanning Electron Microscope (FE-SEM) were used to study nano Rice Husk Ash (nRHA) particle size and morphology. A thin, conductive layer was applied to nano RHA samples on a FE-SEM stub. After preparation, a concentrated narrow electron beam scanned these samples. Where this beam produce X-ray photon when focused, and these photons generate electrical pulses for analysis by interacting with a silicon detector. This analysis was done on RHA milled for 10, 20, 30, and 40 hours.

3 Result and Discussion

3.1 Particle size analysis

Field Emission Scanning Electron Microscopy (FESEM) was employed to evaluate the mean particle size of nano Rice Husk Ash (nRHA) at various grinding durations. The results, presented in **Fig .5** and **Fig .6**, reveal the mean particle sizes of nRHA after 10, 20, 30, and 40 hours of grinding, resulting in sizes of 37 nm, 32 nm, 29 nm, and 35 nm, respectively. The figures illustrates the impact of grinding duration on the average size of nRHA particles. In comparison to NS1, the nano RHA sizes exhibited reductions of 13.5%, 21.6%, and 5.4% for NS2, NS3, and NS4, in agreement with previous research [16]. The FESEM results affirm the effectiveness of the grinding process, with nano particles achieving the optimal size of 28 nanometers after 30 hours of grinding as shown in **Fig. 6**.

The observed pattern reveals a reduction in particle size with prolonged grinding up to a certain threshold, beyond which agglomeration of particles becomes evident. This agglomeration phenomenon is attributed to the Van der Waal forces acting upon the particle surfaces [17]. These results elucidate the dynamic relationship between grinding time and nRHA particle size, shedding light on the optimal grinding duration for achieving desired particle sizes in various applications.

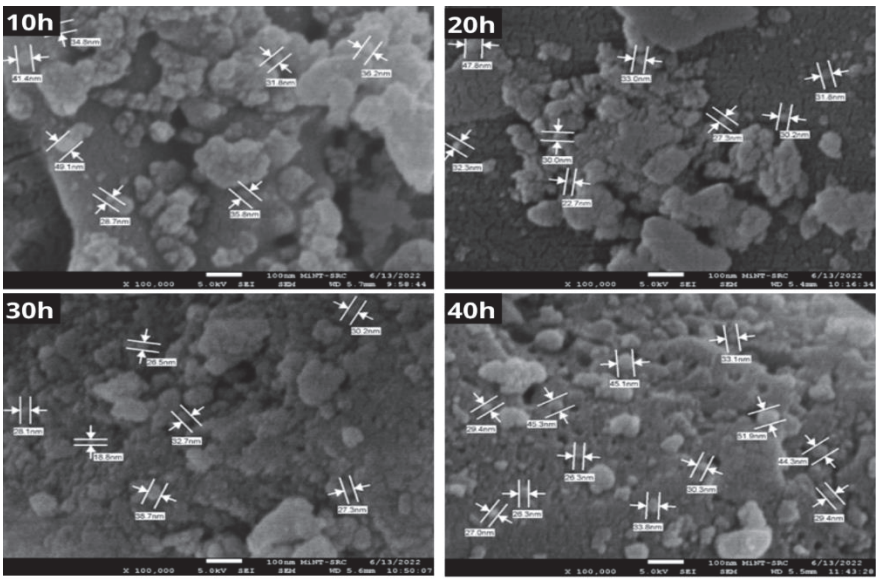

Fig. 5. nRHA after grinding at 10h, 20h, 30h and 40h.

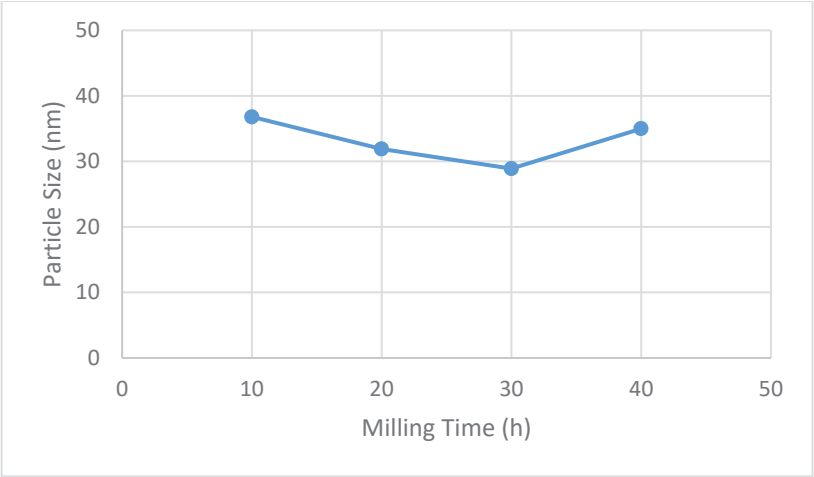

Fig. 6. Effect of grinding time on the mean particle size of nano RHA

3.2 Particle Morphology

Field Emission Scanning Electron Microscopy (FE-SEM) provides significant contributions to the understanding of the morphological properties of nRHA by using particle morphology images. In **Fig. 7**, the FESEM images depict the morphology, size, and distribution of ball-milled nRHA after varying milling durations, ranging from 10 to 40 hours. The images presented illustrate the varied morphologies of nRHA particles, which include irregular and spherical forms. Furthermore, they demonstrate the existence of agglomerates that were generated during the ball-milling process. This agglomeration tendency can be attributable to the Van der Waal forces that operate between them. Additionally, the images illustrate the fusion of certain nRHA particles as a result of the compressive forces that were exerted during the milling process. The results presented in this study are consistent with prior investigations conducted by [16][18], thereby enhancing the comprehension of the morphological alterations in nRHA caused by varying milling times

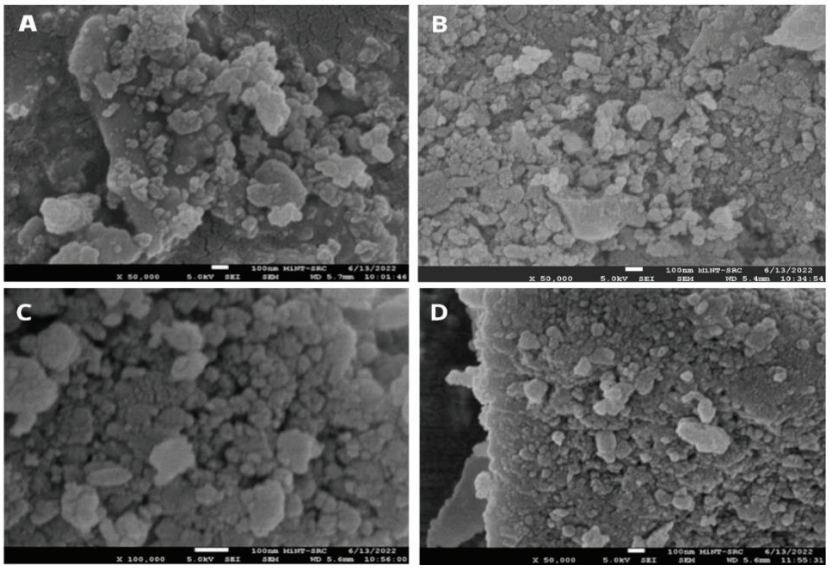

Fig. 7. nano RHA at milling time: (A) NS1, (B) NS2, (C) NS3, (D) NS4

4 Conclusion

1. The duration of grinding affects the particle size of nRHA. An increase in grinding time results in a clear reduction in particle size, but only up to a certain point beyond which particle agglomeration becomes apparent.
2. The research highlights the critical importance of choosing the most effective milling time when utilizing nRHA in a variety of applications. The selection of grinding time has a substantial impact on the particle size.
3. FESEM test provided significant knowledge about the morphological properties of nRHA. The analysis showed a variety of particle arrangements, comprising irregularly shaped and spherical particles, in addition to the existence of agglomerates. The grinding procedure affects these morphological characteristics, which have impacts for the material's properties.
4. The agglomeration of nRHA particles is due to van der Waal forces acting between individual particles. Aggregation of nRHA may have significant influence on performance of nRHA in various applications and hence different materials need to be developed to meet such needs.
5. The issue of agglomeration in nRHA needs to be addressed. Research efforts could involve novel or other additives that minimize agglomeration and provide uniform distribution of nRHA in construction materials.

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