Characterizations of rice husk based silica made from acid leaching extraction method

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Abstract. Rice husk is a material that is greatly acquired through agriculture, which opens up as a source for silica. It available in high quantity that has potential to be used in the concrete. Consequently, this research aims to address this gap by extracting and characterizing the silica from rice husks using the acid leaching method. The different leaching duration of for 6, 12, 18, and 24 hours is the main point of focus in this research. Leaching was done using 0.1 M HCl at room temperature. The XRD, EDX, and SEM are used to characterize the silica. The outcome has been achieved for leaching a period of 24 hour at room temperature was optimal for producing amorphous silica with minimal crystalline and smoothest surface morphology. The study explores an energy efficient and mass production method for silica extraction from rice husks.

1 Introduction

Silica or silicon dioxide (SiO_2), is a prime material in construction industry due to its unique characteristics that include its high thermal stability, mechanical strength, and chemical inertness [1]. In new advance era, the production of silica was increased and traditional method of silica extraction from quartz, caused environmental impact and utilised significant energy [2]. The necessity for sustainable resource, rice husk becomes a source of silica due to content of 15–25% of silica of its weight [3].

Rice husk is the outer layer of the rice grain, whose byproduct is agricultural waste and lacks usage in industry. Rice husk is abundantly available, with around 750 million metric tonnes of rice and 160 million metric tonnes of rice husk produced each year globally [4]. The rice husk composition of 40-50% cellulose, 20-30% hemicellulose and 10-25% of lignin [5]. The silica in the rice husk has the potential to partially replace cement [6]. The extraction of silica from rice husks could be achieved by leaching method to eliminate impurities and maximise the silica yield from rice husks [7,8].

Rice husk contains metallic impurities like aluminum oxide (Al_2O_3), iron (III) oxide (Fe_2O_3), calcium oxide (CaO), magnesium oxide (MgO) and potassium oxide (K_2O) [9]. The technical possibility of using HCl as acid in removing metallic impurities that make easier to breakdown the complex organic structures when compare to nitric acid (HNO_3) and sulphuric

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acid (H_2SO_4) [10]. HCl is a relatively cost low in price and widely available chemical, which making it a viable option for large scale applications.

Based on the conventional method of extracting silica, it involves high temperature during leaching process, this substantial consumes energy, which leads to process by batch operations. This study explores the feasibility of room temperature leaching method that offers sustainable and economically viable alternative, as it can be execute on a mass scale. However, there is limited study of the efficiency and effectiveness of room temperature leaching in producing silica. Recent works have shown that high temperature leaching can achieve high-purity silica but heat required to leach that leads to cost of significant energy input and limited scalability [11]. By comparing the effectiveness of room temperature leaching over different durations to 6, 12, 18, and 24 hours, this study provides a novel perspective on sustainable silica extraction. The techniques like energy dispersive X-ray spectroscopy (EDX), scanning electron microscopy (SEM) and X-ray diffraction (XRD) were used to provide comprehensive insights into the structural, morphological, and chemical properties.

2 Materials and methodology

The rice husk was collected from a rice mill factory located in Langkap, Perak, Malaysia. The sample of rice husk is from MR297 and MR315, which is the type of rice breed created by the Malaysian Agricultural Research and Development Institute (MARDI) [12]. The rice husk was washed at least twice to eliminate contamination and dried in the oven until the rice husks were in a moisture-free state, as shown in Figure 1.

Fig. 1. Rice husk.

The dried rice husks were processed with HCl acid leaching process to leach the rice husks at acid concentration of 0.1 M. The rice husks were completely soak in the solution that diluted acid for a duration of 6, 12, 18, and 24 hours, as shown in Table 1. This is to study the effect of different duration of leaching to extract silica in efficiency and the effective removal of impurities from rice husk. The rice husk was washed thoroughly to achieve a neutral pH level after the specified leaching duration. The washed rice husks were dried in an oven to prepare for the combustion process.

Table 1. Leaching condition.

Sample	Leaching Parameters		
	Acid Concentration	Leaching Temperature	Leaching Duration
RH6	0.1M HCl	Room Temperature	6 hours
RH12			12 hours
RH18			18 hours
RH24			24 hours

The dried rice husk was placed in a claypot to undergo combustion in the electric furnace, as shown in Figure 2. In addition to producing ash, the combustion process would remove the organic matter to obtain pure silica, which would then be converted into crystalline and amorphous silica [13].

Fig. 2. Leached rice husk.

A temperature of 800 °C used by past researchers could achieve 99.28% silica [11]. Therefore, the rice husk was burned at a temperature of 800 °C to achieve the optimal result of amorphous silica. The rice husk was undergoing a combustion process for a duration of 2 hours at increasing rate of 5 °C/min until it reached temperature of 800 °C, as shown in Figure 3.

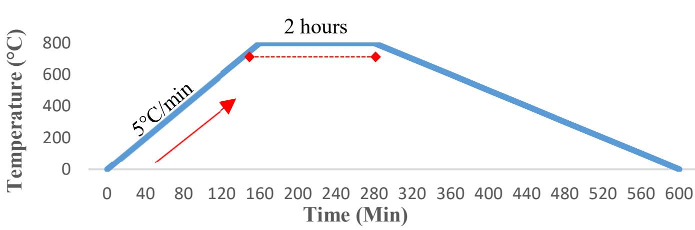

Fig. 3. Burning temperature and time.

To characterize the silica extracted from rice husk, three primary techniques were employed. Siemens D500, which is X-ray diffraction (XRD) machine was used to determine the crystalline structure and phase of silica. Its continuous scanning from 10° to 80° at 0.02° intervals with a fixed X-ray radiation wavelength of 1.54 Å. Energy dispersive X-ray (EDX) analysis was conducted to identify the elemental composition of the silica, where samples were coated with gold with thickness of 15 nm and tested for elements like silicon (Si), oxygen (O) and metallic impurities. At last, Field Emission Scanning Electron Microscopy (FESEM) was used to examine the morphology of surface of the leached rice husk and scanned at 500X magnification with a JEOL JSM-6701F microscope.

3 Results and discussion

As indicated in Table 2, EDX analysis showed that RH24 had the lowest carbon content (41.11%) and silicon concentration (11.22%). This implies that the leaching period of 24 hours was the most effective in eliminating impurities and enhancing the silica concentration compare to Rice Husk. The negligible amounts of sodium, chlorine, and potassium further confirm the purity of the silica extracted. Longer leaching duration result in a decrease in carbon content, which means that organic matter has been effectively removed, while a rise in silicon content indicates that silica has been successfully extracted [14].

Table 2. EDX analysis of RH and RHA.

Element	RH (weight%)	Rice Husk Ash			
		RH6	RH12	RH18	RH24
C	50.32	58.82	48.64	56.20	41.11
O	46.58	38.55	48.52	43.21	47.62
Na	0.00	0.00	0.00	0.00	0.00
Si	3.02	2.13	2.85	0.52	11.22
Cl	0.00	0.50	0.00	0.06	0.05
K	0.09	0.00	0.00	0.00	0.00

As from Table 3 and Figure 4, which the XRD patterns shows that all samples had a low crystalline content in general and a significant amount of amorphous silica. The XRD patterns indicate the presence of amorphous silica as form of broad humps, while the small peaks represent minor crystalline phases. In comparison to RH6 and RH12, the samples that were leached for 18 and 24 hours had a slightly higher amorphous content. This indicates that by eliminating contaminants that may otherwise encourage crystallisation, longer leaching times favour the development of amorphous silica [15]. For construction applications, the high amorphous content is preferred since it increases the pozzolanic activity of the silica.

Table 3. XRD analysis.

Sample	RH6	RH12	RH18	RH24
Amorphous Silica	95.00%	95.00%	95.36%	95.35%
Crystalline	4.00%	4.00%	4.65%	4.65%

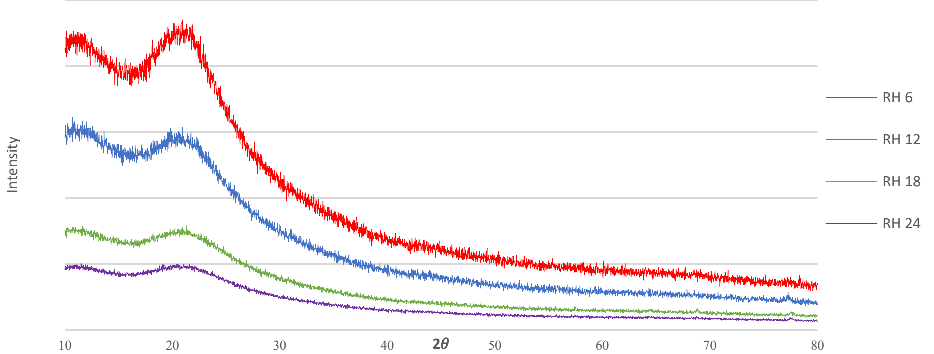

Fig. 4. XRD patterns.

The non-leached rice husk and the RH24 sample had significantly different surface morphologies, as seen from the SEM pictures in Figure 5. The SEM image shows that the surface of the non-leached rice husk was rough and irregular surface with numerous impurities. This shows that fibrous structure is not visible and impurity blocked rice husk, which effect the extraction of the silica. On the other hand, the RH24 image shows a cleaner, smoother and homogeneous surface. This indicating the effective of removal of organic and inorganic contaminants by using HCl acid [15]. The surface morphology of RH24 is attributed to the prolonged duration of leaching as resulted in thorough removal of impurities [15]. The silica's reactivity could increase by cleaner surface, which makes it more appropriate for usage in construction applications.

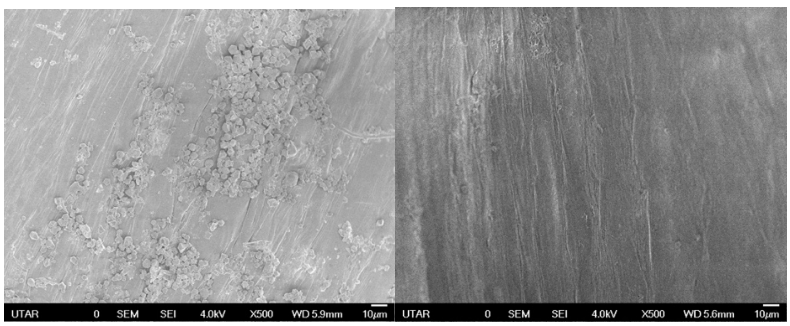

Fig. 5. SEM image of rice husk and RH24.

Collectively, the findings from the XRD, FESEM, and EDX results, show that a RH24 or 24 hours leaching period was the optimal for extracting silica. As a result, the longer leaching durations directly link in increase in silicon content and a drop-in carbon content, indicating effective impurity elimination. The majority of the extracted silica was in an amorphous state, which is desirable for pozzolanic activity [8,16]. Crystalline silica would reduce the pozzolanic reactivity [17]. The surface morphology image was given visual confirmation that extended leaching periods is optimal for producing silica from rice husk. The choice of using HCl as leaching agent was a result of due to its ability to dissolve organic matters and metallic impurities without interacting with silica. The economic feasibility of using HCl lies in due to its readily available and priced at low. The extraction of high-purity silica with low residual impurities is success of extraction in room temperature without any additional heat is indicating the technical feasibility. According to this method, this process of leaching achieved that longer durations enhance the removal of impurity and silica extraction.

4 Conclusions

This study reveals that the sample effected by leaching duration on room temperature and XRD, EDX, and SEM used for characterization. The key findings of study are:

1. The duration of leaching period for 24 hour produced high amount of silica, with the highest silicon content and lowest carbon content, indicating effective impurity removal by HCl acid.
2. The leaching using HCL produced amorphous silica across all samples, with minimal crystalline content. Longer leaching slightly increased the purity of the amorphous silica which showed in XRD result.
3. A slightly increased in silicon content and a decreased in carbon content for a longer leaching duration in EDX results. The RH24 sample had the highest silicon content of 11.22% and the lowest carbon content of 41.11%.

4. SEM images revealed that RH24 sample leaching improved surface morphology which shows a clean, smooth surface and with minimal impurities compared to non-leached samples.

In conclusion, acid leaching for a longer period at room temperature is effective for extracting high purity silica from rice husks that making it a viable and eco-friendly material for the construction industry.

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