

# Improvement in the Extraction of Silica from Rice Husk through Controlling Drying Temperature and Leaching Process

Bahaa F. Shihab<sup>1</sup>, Wesam A A Twej<sup>2</sup>

Department of Physics, College of Science, University of Baghdad, Baghdad, Iraq

**Abstract:** Particular procedure has been adopted in this work using acidic treatment of rice husk and rice husk ash to reduce the impure elements ratio from the final silica product. Hydrochloric acid as a leaching acid was employed before and after combustion of rice husk. Via controlling the drying temperature and the drying sequence, the silica contain percentage, in the final product, was improved from 91.12% to 98.8%, this results was monitored through the change of the ash color from approximately black to obviously white.

**Keywords:** Rice husk, silica extraction, leaching process, drying

## 1. Introduction

Rice husk (RH) can be represented as one of the main agricultural residues initiated as a byproduct during the rice milling process. Most of the rice plant straw is either used for animals bedding material or simply burned in the fields causing air and soil pollution. The expressive content of about 20% silica in RH and, after burning, more than 90% silica in rice husk ash (RHA) have encouraged extensive research. Since biomass is one of the copious resources in the earth, therefore, it is strongly recommended to make use of RHA as inexpensive sources of amorphous porous silica [1-2].

Unfortunately, RHA is, commonly, consists of several undesirable oxides such as  $K_2O$ ,  $CaO$ ,  $MgO$ ,  $MnO$ ,  $Al_2O_3$ ,  $P_2O_5$ , etc., in decreasing concentrations [3-4]. These oxides are hereinafter referred to as impurities because they change the properties of the silica which extracted from the RH. The potential to extract high-quality silica from RHA has involved the attention of several researchers [5-6]. The amorphous and white silica that can be extracted from RH may possess a very high specific surface area, SSA. During combustion, the impure elements, especially alkaline earth oxides, strongly reduce the silica's SSA. Where the impure elements may possibly react at high temperatures and long soaking times with part of the silica to produce a kind of soda lime glass [7-8]. Resulting in reduces the silica SSA to close  $1.0 \text{ m}^2/\text{g}$  [9].

Treatment of the RH with an acid solution as was done by Amick [10] and later extended by Real et al.[8]. Removes a large fraction of the impure elements that would if not contribute to the formation of a glassy phase.

In the third world countries and due to the unwise employing of chemical fertilizers, mainly potash and phosphates, special attention should be utilized in extraction silica from the RH which collected from these countries. In this work particular procedure was followed in the acidic treatment of RH as well as RHA to reduce the impure elements ratio from the final silica product.

## 2. Methods

### 2.1 Raw materials

Rice husk was obtained from a local rice milling company in Almishkab farms in Iraq. Hydrochloric acid (35 %, Merck, India). All chemicals were of AR grade and used directly without purification. Finally, distilled water supplied from our lab.

### 2.2. Procedure

#### 2.2.1. Rice husk ash preparation

The adopted RH was first sieved to free it from dust and foreign material. Thereafter, the RH was washed with water three times, then by distilled water several times reaching notarizes pH. About 30.0 g of clean Rice Rusk (RH) was stirred in 750mL of 1.0M HCl at room temperature for 24 h. Then this RH was washed with copious amount of distilled water to constant pH, this RH was denoted as leaching RH. Leached and no leached RH was dried in an oven at  $150^\circ\text{C}$  for 24 h then burned in a muffle furnace at  $650^\circ\text{C}$  for 6 h so as to obtain white rice husk ash (RHA). The burning process divided into two categories. First drying type utilized by direct rising the temperature to  $600^\circ\text{C}$ , while the second by rises the temperature to  $375^\circ\text{C}$ , keep the sample at this temperature for 30 min then raises the temperature to  $600^\circ\text{C}$  at a rate of  $5^\circ\text{C}$  per min. The two types of drying samples were kept at  $600^\circ\text{C}$  for four h. Figure 1. illustrates the flowchart of the RHA preparation process.

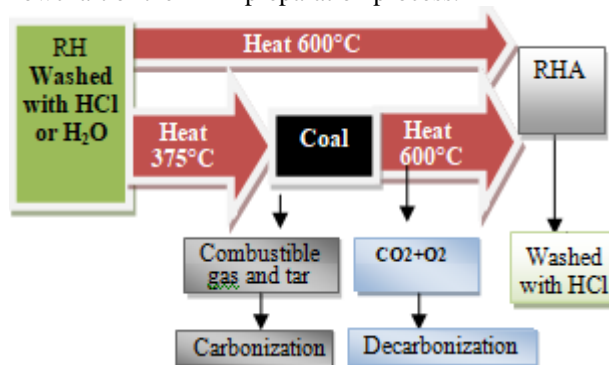


Figure 1: Flowchart of the RHA preparation process

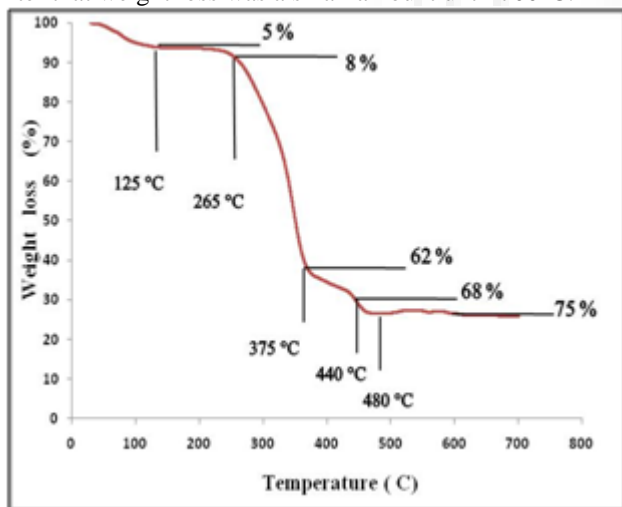
As a summary, five samples were prepared in this work, these samples were described as following; Sample W1 was ash prewash by H<sub>2</sub>O consumption at 600 °C, while sample W2 was ash prewash by H<sub>2</sub>O consumption at 375°C then rise to 600 °C. Whereas, sample H1 was as sample W1 but prewash by HCl as well, and sample H2 was as sample W2 except it prewash by HCl as well. Finally, sample HH2 was like sample H2 but the final dried ash was further washed by HCl [0.5N]. Figure 2 shows the photograph of the resulting RHA samples.



**Figure 2:** Photograph of W1, W2, H1, H2, and HH2 samples

### 3. Result and Discussion

Fig. 3 shows the TG results of RH tested in an atmospheric environment. Noticeably, the figure shows that the moisture had been removed from RH until 125°C as indicated by ~5% RH mass loss at this temperature. The second stage of the thermal decomposition of organic compounds correlated with the removal of volatile matter in RH. It was starting at 265 °C and finishing at 375 °C with the mass loss from 8% to ~62%. Afterward, the third stage can be divided into two parts; first was finished at 440 °C with ~68 % mass loss, and the second was finished at 480 °C with ~75 % mass loss. The first part may correspond to the decomposition of relatively long chain carbonaceous materials, while the second part may correspond to the totally converting of the carbons solid contain to gaseous carbon oxides. Frequently, the zone at around 480-600 °C was referred to as the passive zone where no further decrease in mass was observed [11]. After that weight loss was a small amount until 700°C.



**Figure 3:** Thermogravimetry (TG) curves of rice husk in the natural atmosphere

No appreciable decrease in the mass of sample was observed after this temperature. It is to be mentioned here that based

upon these TGA results, the temperature for thermal treatment of acid-leached RH was chosen to be 600 °C to convert RH into white RH. Moreover, TGA results showed that the organic compounds and carbonaceous materials were degraded before 600 °C.

Mainly, there are two separate stages in the thermal decomposition of RH; carbonization and decarbonization. Carbonization includes the decomposition of volatile matter in RH at a temperature little above 375 °C that releases combustible gas, and tar to form coal. Decarbonization comprises the combustion of carbon in the presence of oxygen.

The compositional analysis results of RH, as performed by XRF, are given in Table 1, while that of RHA are present in table 2. Commonly, RH's are known to have a relatively high content of inorganic compounds. The best result in this work is representing approximately 25 wt% of the dry RH, 98 wt% of which is silica. The percentage of these inorganic compounds depend on several factors, including weather, soil and plant variety.

**Table 1:** XRF compositional analysis of RH

Materials	washed by H <sub>2</sub> O	washed by HCl as well
SiO <sub>2</sub>	22.72 %	24.13%
KO <sub>2</sub>	0.743%	0.0067%
Metal oxides	1.877%	1.4833%
Organic component	74.66%	74.38%

The highest percentage of these groups is potassium oxide K<sub>2</sub>O, that the reason why ash is of gray color instead of white [12]. Basically, white RHA is preferred due to its low carbon level. Even at high burning temperatures, the potassium oxide RH's does not volatilize if the RH's are untreated before burning. This may be due to the strong interaction between potassium and silica which causes a form of potassium poly-silicate (K<sub>2</sub>SiO<sub>3</sub>) on the surface of the RH.

**Table 2:** XRF compositional analysis of RHA samples

Contain	W1 %	W2 %	H1 %	H2 %	HH2 %
SiO <sub>2</sub>	91.12	93.29	97.68	98.02	98.8
MgO	0.354	0.25	0.002	0.002	---
P <sub>2</sub> O <sub>5</sub>	1.15	1.03	0.83	0.78	0.67
K <sub>2</sub> O	3.19	1.67	0.088	0.055	0.0013
CaO	2.12	2.25	0.617	0.512	0.15
Fe <sub>2</sub> O <sub>3</sub>	0.772	0.4	0.32	0.233	0.16
Others	1.294	1.19	0.463	0.398	0.2187

Potassium poly-silicate could form glassy or amorphous phases, and here preventing the conversion of carbon to CO<sub>2</sub> gas [13], resulting in grey-colored ash. Therefore, rapid burning of RH at high temperatures will cause high carbon residual in the ash. Whatever it increased the duration of the burning. The cause for the formation of K<sub>2</sub>SiO<sub>3</sub> from the first hour [12]. The dissociation of potassium oxide at 347°C forms elemental potassium which melts at a very low temperature (melting point of potassium is 64°C). Thus, facilitating its interaction with silica causing production K<sub>2</sub>SiO<sub>3</sub>, thereby entrapping the carbon content and making the carbon unavailable for further conversion even at higher temperatures. Because the carbon is not in direct contact

with the air, and the formation of black particles in RHA [14].

As a general view, the carefully controlling of RHA combustion and leaching can result in high purity silica product about 98.8% in comparison with previously attempted procedures, as mentioned in table 3.

**Table 3:** The composition of rice husk from selected countries

Contain	Malaysia (%) [15]	Taiwan (%) [16]	Thailand (%) [17]	Brazil (%) [18]	Iraq present work (%)
SiO <sub>2</sub>	92.00	95.60	92.80	94.95	98.8
MgO	0.56	-----	-----	0.90	-----
P <sub>2</sub> O <sub>5</sub>	0.77	0.52	1.07	0.74	0.67
K <sub>2</sub> O	1.70	2.66	3.35	0.94	0.0013
CaO	0.83	0.70	0.70	0.54	0.15
Fe <sub>2</sub> O <sub>3</sub>	0.20	0.24	0.17	0.26	0.16
Others	3.04	0.296	1.91	2.57	0.2187

## Conclusions

Rice husk ash, derived from Iraqi Jasmine rice husks through combustion, has a high silica content of more than 98% under suitable treating. Therefore, it exhibits a potentially valuable, cheap alternative to the more expensive natural and synthetic silica for numerous industrial applications. This quest has stimulated a new-found interest in stuff from agro-waste materials having potential 'recyclability'. Their abundant availability, low-cost and recyclability has become a most important powerful factor in their utilization as industrial raw -materials.

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