



ISSN: 2350-0328

**International Journal of Advanced Research in Science,  
Engineering and Technology**

**Vol. 5, Issue 7 , July 2018**

# **Enhancement in extraction silica percentage from Iraqi Jasmine Rice Husk through controlling leaching process**

**Wesam A A Twej\*, Bahaa F. Shihab**

Ph.D., Lecturer of molecular technology Department of Physics, College of Science, University of Baghdad, Baghdad, Iraq.

M.Sc. Student University of Baghdad, College of Science, Department of Physics. Baghdad, Iraq.

**ABSTRACT:** In the present work, we have tried to enhance the extraction rate of impurities from RHA by means of acid leaching process. HCl solution under a different concentration and leaching periods were used. The study of leaching influence is followed by EDS and XRF tests. The leaching solution concentration seemed to be the critical parameter in the purification process. It is found that most of the metal oxides which represent as impurity don't affect under diluted concentrations. The leaching period as well should be sufficient in favor of effective silica extraction. By adopting the leaching solution of a concentration of more than 1[M] and leaching period more than 48h good purification results can be utilized, where the extracted silica percentage will reach 98.8 %.

**KEYWORDS:** Rice husk, Hydrochloric acid, leaching process.

## **I. INTRODUCTION**

Rice husk RH is largely available agricultural waste found in the world. Globally, up to 120 million tons of rice husks are produced annually [1, 2]. Due to the low cellulose and other sugar contents, rice husk is generally not suitable as cattle feed, therefore, industrialists usually used rice husk as fuel in power generation. Commonly, most of the rice husk ends up either burned in open spaces or being dumped, therefore, causing environmental pollution and damage to the land. Rice husk is commonly contained about 20% of ash which can be converted to amorphous silica. The amount of silica in the rice husk ash (RHA) can reach 90–98% [3]. As a result of this high silicon content, rice husk has become a low-cost source for the preparation of silica powder [1].

Sand is the most abundant compound on earth which is mainly contain silica. In practice, all of this silica is in the crystalline form. This form of silica, even as useful in some applications such as construction, is low-value material. For silica to be more useful in a variety of applications it must be in the amorphous, or non-crystalline, form. The current traditional processes for the production of amorphous silica are energy intensive, chemical-intensive and costly methods, that crash and transform the crystalline structure of silica in the sand to the amorphous form. Obviously, these methods are not earth friendly because it has a very large carbon footprint. The nano-silica powder is generally prepared by using vapor-phase reaction, sol-gel, and thermo-decomposition [4-8] methods. In most of the above-mentioned methods, nano silica powder is synthesized using chemicals as a raw material. In chemical methods, it is easy to control size, shape, and purity of the material but the starting reagents are costly. In industrial applications, low costs and large quantities of the initial precursor are needed. Several attempts have been made to produce bulk silica from the most eco-friendly and economical source rice husk [9–14].

The review confirms that rice husk is an excellent source for the production of high-grade amorphous bulk silica powder. In recent years attempts have been made to prepare nano silica powder from rice husk [15–20].

This silica finds broad relevance as an adsorbent, filler, catalyst support, in rubber, plastics, cosmetics, electronics, coating and optics [21]. The pureness and color of the ash usually impacted through a variety of metal ions as well as unburned carbon.

Following the specific procedure of burning and chemical leaching of the husk can remove these ions then produce high purity white silica [22, 23]. In the present work, a simple process has been proposed for the preparation of amorphous silica powder. The effect of leaching process on the silica purity was investigated too.

## II. METHOD

### A. RAW MATERIALS

Jasmine rice husk was obtained from a local rice milling company in Al-Mishkab farms, Iraq. Hydrochloric acid (35 %, Merck, India) of AR grade was used directly without further purification. The distilled water was supplied from our lab. Thermo-gravimetric analysis (TGA) for RH was done in this work using STA PT1600 instruments supplied from Linseis (Germany). The process was achieved in a nitrogen atmosphere. The EDS test was utilized using ZIESS ULTRA 60, USA.

### B. PROCEDURE

#### a. RICE HUSK ASH PREPARATION

Firstly the RH was sieved to remove dust and strange material. Consequently, three times washing the RH by water then twice by distilled water, reaching neutral pH. About 20.0 g of the washed rice husk (RH) was soaking at room temperature in 1L of HCl solution. The leaching process was utilized through two categories. First, five different concentrations of HCl solution were tested; 0.25M, 0.5M, 1M, and 2M, the soaking time was 24 h, these RH samples were denoted as (a), (b), (c), and (d) respectively. Second 1 M HCl solution was used other than the soaking time were varied from 24, 48, 72, and 96 h. The cleaned RH was washed with a plentiful amount of distilled water to constant pH, these RH samples were denoted as (e), (f), and (g) respectively. All samples were dried in an oven at 150 °C for 24 h to remove the moisture. The dry RH was burned in a muffle furnace at 350 °C at a rate of 5 °C / min then kept at this temperature for 30 min. The temperature was raised to 650 °C at a rate of 5 °C per min then kept at this temperature for 6 h so as to obtain white rice husk ash (RHA).

## III. RESULT AND DISCUSSION

Commonly, due to the unwise usage of fertilizers such as potash, different materials are stuck on the RH. These materials are almost not removed through water washing only. The existence of these materials has a serious influence on the silica purity in the RHA. Table 1, illustrates the percentage of RH before and after leaching in 1[M] HCl solution. The results reflect the high removing efficiency of potassium is superior with HCl. Where the removal of up to 99% of the potassium concentration. Treatment of RH with either HCl proved to be effective in removing some metals substantially, where they are not easily soluble in water.

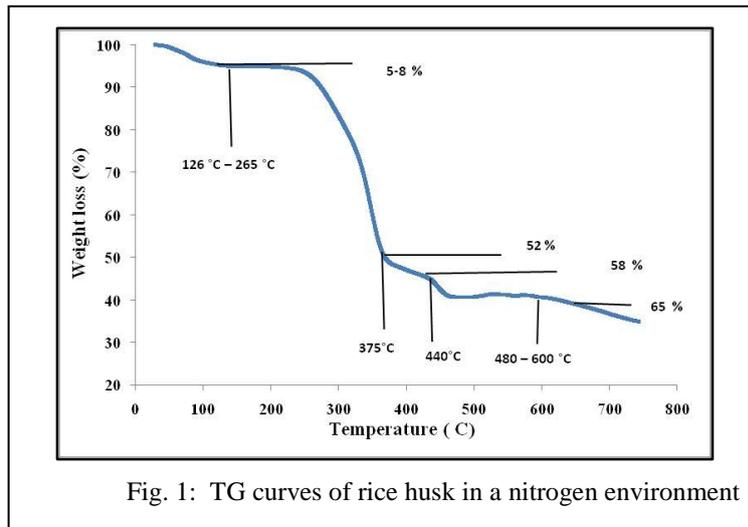
Table 1: RH contains not leached and leached in 1 [M] HCl overnight, examined by XRF

Materials	Silicate %	Potash %	Metal oxides %	Organic component %
Not leached	22.72	0.743	1.877	74.66
Leached	24.13	0.0067	1.4833	74.38

Figure 1 shows the TG results of RH tested in a nitrogen environment. Noticeably, it shows that the moisture had been removed from RH until 126 °C – 265 °C as indicated by ~5% - 8% 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. This stage was starting at 265 °C and finishing at 375 °C with ~52% mass loss. There was a great loss of weight due to oxidation of the volatile products generated, this range is denoted by M. V. Ramiah, as the active pyrolysis zone [24]. Afterward, the third stage can be divided into two parts; first was finished at 440 °C with ~58 % mass loss, and the second was finished at 480 °C with ~65 % 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 content to gaseous carbon oxides. Commonly, the zone at around 480-600 °C was referred to as the passive zone where

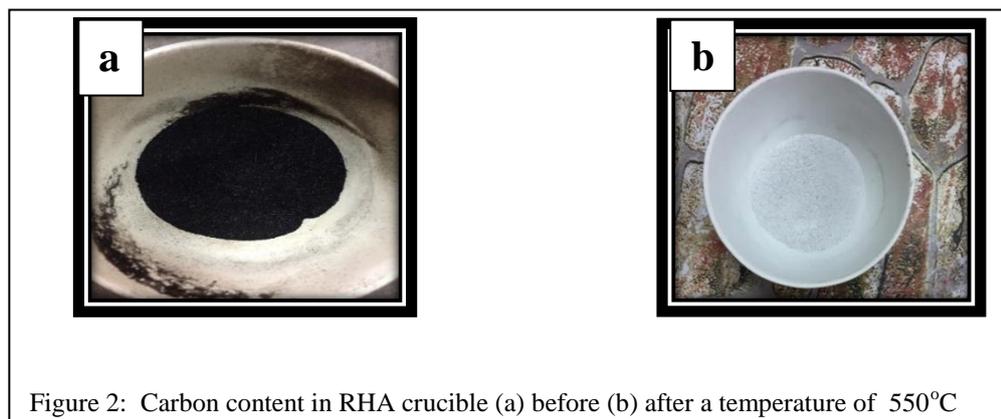
no further decrease in mass was observed [24]. after that weight loss was a small amount until 700°C. The weight loss can be attributed to the inorganic compounds which are converted into vapors, as mentioned by N. B. Singh et. al [25].

It is to be mentioned here that based upon the results of TGA, the temperature for thermal treatment of RH was elected to be 700 °C to convert RH into WRH. Furthermore, TGA results demonstrated that the carbonaceous materials and all organic compounds were degraded before 700 °C.



From the observation of the combustion process for RH, the following notes are seen. In the first stage of the burning process, to 300°C, light brown smoke was slowly emitted from the furnace. As the temperature was rising to 400 °C, the amount of emitted smoke was increased dramatically. By reaching 500 °C, the smoke emission rate starts to decrease gradually and completely stops after half an hour. At this stage, it was observed that a presence of black soot at the door of the furnace as well as at the edges of the crucible, indicated to the finishing of the carbonization stage, as point out by T. H. Liou [26].

As the temperature rises above 550 °C, the carbon begins to volatilize, reflected by converting black RHA gradually to white, as shown in figure 2.



To investigate the influence of leaching processes on the RHA purity, two tests were adopted EDS and XRF with the aim of examining the RHA contains. Figure (3) show the EDS spectra for two samples; (a) that leaching for 24 h in 0.25 [M] HCl and (e) that leaching for 48 h in 1[M] HCl.

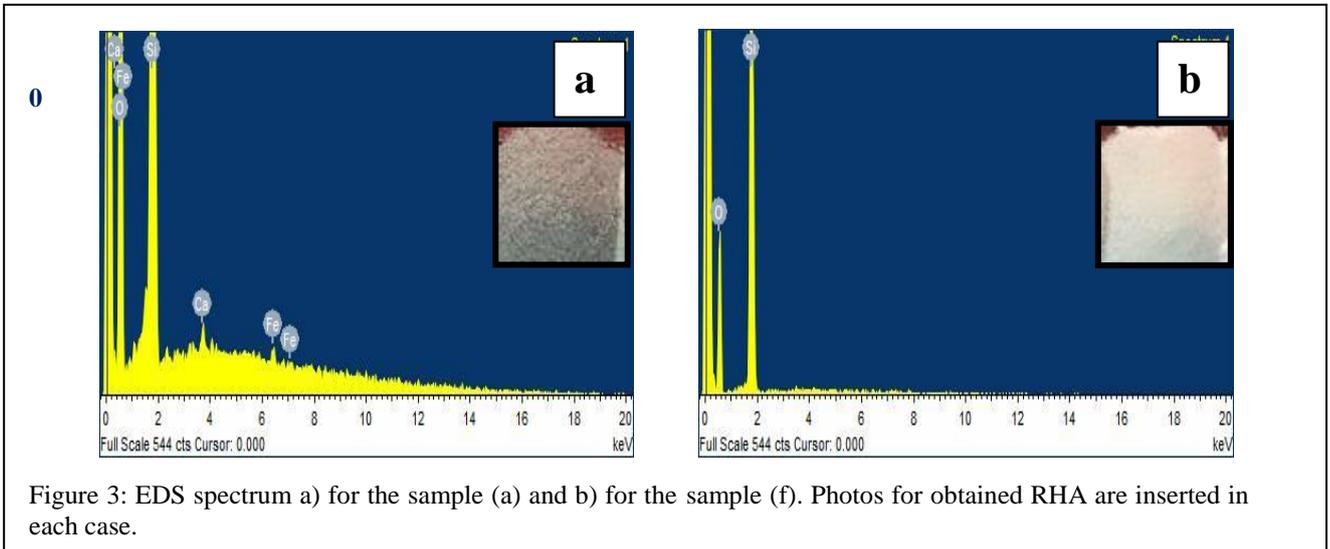


Figure 3: EDS spectrum a) for the sample (a) and b) for the sample (f). Photos for obtained RHA are inserted in each case.

In this figure, two insert photographs for the resulting RHA are present for the two samples. From the change of the color of ash it is clear that, in the case of sample (e), almost carbon and other metal oxides are removed through the leaching process. Table 2, illustrates the EDS contains the result for the prepared samples, however, samples (f) and (g) show the same results as in sample (e) therefore, they were excluded from the table.

Table 2: The contains the result of the RHA samples as examined by EDS

Component	a	b	c	d	e
	Contain %				
<b>O</b>	57.30	56.19	56.19	58.48	56.91
<b>Si</b>	41.93	42.97	42.87	40.76	43.09
<b>Ca</b>	0.40	0.54	0.72	0.75	-
<b>K</b>	0.10	0.09	0.08	0.1	-
<b>Fe</b>	0.23	0.21	0.14	0.11	-
<b>Mg</b>	0.04	-	-	-	-

As presented in table 1, the increasing of the concentration of soaking solution even by soaking the RH can be enhancing the purification of silica ash, reflected by the percentage reduction of foreign materials. However, there is no signification variation in the ash purity at a concentration greater than 2 [M]. While increasing from 24h to 48h has a great influence on the purification process. Where, with respect to the test accuracy, almost impurities material were removed by soaking RH in 1[M] HCl solution for 48h.

#### IV. CONCLUSION

Converting RH to RHA via acidic leaching followed by combustion drying was done in this work successfully. The acidic concentration in the leaching process was an important parameter in resulting product quality. The acid concentration should be not less than 1 M, and the leaching period should be not less than two days, in order to obtain high purity silicate in the extraction of silica from RHA. The heat treating temperature should not rises than 700 °C for achieving amorphous silica which was more preferable in a different application. in order to obtain high purity silicate in the extraction of silica from RHA.

#### REFERENCES

[1] Gidde M. R., Jivani A.P. "Waste To Wealth - Potential Of Rice Husk In India A Literature Review" Proceedings Of The International Conference On Cleaner Technologies And Environmental Management PEC, Pondicherry, India. 4-6, pp.586-590, 2007.  
 [2] Ludueña L., Fasce D., Alvarez V. A., Stefani P. M. "Nano-cellulose from rice husk following alkaline treatment to remove silica," Bioresources. vol.6, pp.1440-1454. 2011.



ISSN: 2350-0328

# International Journal of Advanced Research in Science, Engineering and Technology

Vol. 5, Issue 7, July 2018

- [3] Rosario M., Nogueira C. A., Margarido F. "Production And Characterization Of Amorphous Silica From Rice Husk Waste" 4th International Conference On Engineering For Waste And Biomass Valorisation, September 10-13 2012.
- [5] Tomozawa M., Kim D.L., Lou V. "Preparation of high purity, low water content fused silica glass" J. Non-Cryst. Solids 296, pp. 102–106, 2001.
- [6] Tanner P.A., Yan B., Zhang H. "Preparation and luminescence properties of sol-gel hybrid materials incorporated with europium complexes" J. Mater. Sci. 35, pp. 4325–4328, 2000.
- [7] Wu G., Wang J., Shen J., Yang T., Zhang Q., Zhou B., Deng Z., Bin F., Zhou D., Zhang F. "Properties of sol-gel derived scratch-resistant nanoporous silica films by a mixed atmosphere treatment" J. Non-Cryst. Solids 275, pp. 169–174, 2000.
- [8] Sadasivan S., Rasmussen D.H., Chen F.P., Kannabiran R.K. "Preparation and characterization of ultrafine silica" Colloids Surf. A: Physicochem. Eng. Aspects 132, pp. 45–52, 1998.
- [9] Bogush G.H., Tracy M.A., Zukoski C.F. "Preparation of monodisperse silica particles: Control of size and a mass fraction" J. Non-Cryst. Solids 104, pp. 95–106, 1988.
- [10] Mishra P., Chakraverty A., Banerjee H. D. "Production and purification of silicon by calcium reduction of rice husk white ash" J. Mater. Sci. 20, pp. 4387–4391, 1985.
- [11] Chakraverty A., Mishra P., Banerjee H. D. "Investigation of combustion of raw and acid-leached rice husk for production of pure amorphous white silica" J. Mater. Sci. 23, pp. 21–24, 1988.
- [12] James J., Rao S. M. "Silica from rice husk through thermal decomposition" Thermochim. Acta 97, pp. 329–336, 1986.
- [13] Kamiya K., Oka A., Nasu H. "Comparative study of structure of silica gels from different sources" J. Sol-Gel Sci. Technol. 19, pp. 495–499, 2000.
- [14] Mansaray K.G., Ghaly A.E. "Determination of kinetic parameters of rice husks in oxygen using thermogravimetric analysis" Biomass Bioenergy 17, pp. 19–31, 1999.
- [15] Yalcin N., Sevinc V. "Studies on silica obtained from rice husk" Ceram. Int. 27, pp. 219–224, 2001.
- [16] Liou T.H. "Preparation and characterization of nanostructured silica from rice husk" Mater. Sci. Eng.: A 364, pp. 313–323, 2004.
- [17] Zaky R. R., Hessien M.M., El-Midany A.A. "Preparation of silica nanoparticles from semi-burned rice straw ash" Powder Technol. 185, pp. 31–35, 2008.
- [18] Real C., Alcalá M.D., Criado J.M. "Preparation of silica from rice husks" J. Am. Ceram. Soc. 79, pp. 2012–2016, 1996.
- [19] Conradt R., Pimkhaokham P., Leela-Addison U. "Nano-structured silica from rice husk" J. Non-Cryst. Solids 145, pp. 75–79, 1992.
- [20] Li D., Chen D., Zhu X. "Reduction in time required for synthesis of high specific surface area silica from pyrolyzed rice husk by precipitation at low pH" Biores. Technol. 102, pp. 7001–7003, 2011.
- [21] Liou T.H., Yang C.C. "Synthesis and surface characteristics of nano-silica produced from alkali-extracted rice husk ash" Mater. Sci. Eng. B 176, pp. 521–529, 2011.
- [22] Singh L. P., Bhattacharyya S. K., Ahalawat S, Kumar R., Mishra G., Sharma U., Singh G. "Sol-Gel processing of silica nanoparticles and their applications," *Advances in Colloid and Interface Science*, 2014.
- [23] Deschamps G., Caruel H., Borredon M.E., Bonnin C., Vignoles C. "Oil Removal from water by selective sorption on hydrophobic cotton fibers. 1. Study of sorption properties and comparison with other cotton fiber-based sorbents." Environ. Sci. Technol, vol.37, pp.1013–1015, 2003.
- [24] Draman S.F.S., Daik R., Latif F.A., El-Sheikh S.M., "Characterization and thermal decomposition kinetics of kapok (Ceiba pentandra L.)-Based Cellulose," Bioresources. vol.9, pp.8-23, 2014.