

# Preparation and Characterization of Egyptian Rice Husk Ash

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**Abstract:** Solid waste utilization and cost reduction in industrial processing has attracted the use of Egyptian Rice Husk (RH) as added value materials. Rice Husk Ash (RHA) has been found suitable for wide range of domestic and industrial applications. The present paper highlighted and discussed preparation and characterization of burning RH (rice husk) and LRH (leached rice husk) respectively in muffle furnace, at 10 °C rate of heating, in presence of air flow for different temperatures (450 -700 °C) and time (1 – 3 hours). Leaching is carried out by using different inorganic and organic acids, i.e., hydrochloric acid (HCl), sulfuric acid (H<sub>2</sub>SO<sub>4</sub>), nitric acid (HNO<sub>3</sub>) and acetic acid (CH<sub>3</sub>COOH) of different concentrations (0.1N - 4.0N). X-ray fluorescence (XRF) is used to determine the chemical composition of RH, CRH and CLRH and X-ray diffraction (XRD) is used to identify the amorphous silica in RH and both calcined RH. The structure and morphology of silica content was tested by electron scanning microscopy (SEM). Amorphous silica was obtained in the range of 450 - 600 °C High amorphous silica content of 92% and 95.6% were achieved for CRH at 500 °C after 2 hours and for CLRH at 550 °C after 3hours respectively. The investigation of adsorption capacity for the native RH, CRH, and CLRH at pH 11 were found to be 90.9, 98.16 and 96.29 mg/g respectively.

**Keywords:** Rice husk; Ash; Silica; Calcination; Leaching; Analyses.

## 1 Introduction

Rice Husk (RH) is one of the most widely available agricultural solid waste residue in many rice producing countries such as Egypt. The annual production of the Egyptian Rice is nearly four million ton, where RH, as a by-product waste, represents 22% of rice production. The chemical composition of RH is found to vary from sample to sample due to the differences in type of the paddy, crop year, climatic and geographical conditions, in addition to sample preparation and methods of analyses [1]. It consists of 75-80% of cellulose, hemicellulose and lignin and the rest is silica (SiO<sub>2</sub>) [2,3]. The RH is usually burned in the field, causing serious environmental problems for generation of rice husk ash (RHA) [4,5,6]. RH when burned gives RHA with 85-95% silica content, and the rest is some carbon and other nonmetallic and metallic impurities. Controlled burning of the husk after removing these ions can produce silica in white amorphous or crystalline silica form of high purity [7,8]. Silica or silicon dioxide (SiO<sub>2</sub>) exists in two forms, amorphous and crystalline. Processing of silica of specific quality results in several types of specialty silicas, such as colloidal silica, fumed silica, fused silica, high-purity ground silica, silica gel and precipitated silica. The global demand for specialty silicas is growing at an annual rate of 3% with revenue generation of more than USD 2.5 billion [9]. SiO<sub>2</sub> is one of the valuable inorganic multipurpose chemical compounds. Silica has many applications such as in electronics, ceramic and polymer industries, based on its form and by controlling the heating conditions [10,11,12,13]. It was reported by many authors [14,15,7,16], that at calcination of RH at low temperature 400 – 600 °C, pure amorphous silica with high surface area has no spatial order of silicon and oxygen atoms is formed. In contrast, at high temperature 700 -1100 °C crystalline silica with carbon impurities are formed and its atoms are arranged in a fixed geometric pattern. In addition, amorphous silica differs from crystalline SiO<sub>2</sub> not only in physical terms but also as regards its toxicological properties and there are no hazards associated with amorphous silica. Although of these various studies, there are still gaps regarding the optimization of treatment conditions and its relationship with the properties of the final RHA [17,18,14]. In this

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sense, many researches were investigated for producing of high reactive and pure silica from RH, some conditions are crucial in order to obtain an amorphous structure with absence of unreacted carbon [19,20,17,15].

For that, In the present work an attempt therefore has been made to study the properties of Egyptian RH and RHA, and to investigate the thermal behavior of RHA aiming to obtain a better knowledge about the appropriate conditions to achieve a good quality RHA of high amorphous silica content which will be extracted and used in environmental purification and to assess their efficiency in removal of color and heavy metals from waste water.

## 2 Materials and experimental procedure

The experimental plan is subdivided into three sets of experiments with 22 runs. The first set is dedicated to the characterization of raw RH, the second set is devoted to the calcination of RH (CRH) and the third set is covering the calcination of leached RH (CLRH) at different parameters. The aim of the second and the third sets are to identify optimum conditions for RHA acquired with high silica content to be used as adsorbent material. Each experiment was conducted two times for reproducibility check.

### 2.1 Materials and equipment

RH from El-Sharqia Governorate in Egypt is selected for the present investigation. Inorganic acids covering hydrochloric (HCl), sulfuric (H<sub>2</sub>SO<sub>4</sub>), nitric acid (HNO<sub>3</sub>) and organic acid, namely acetic acid (CH<sub>3</sub>COOH) of analytical grades are used for leaching of RH. Programmable muffle furnace controlled by high precision temperature controller with +/- 1 °C accuracy and 40 programmable segments and furnace housing is built with double-layer steel is used for calcination process.

### 2.2. Experimental procedure

At first, RH is analyzed and tested. Then RH is calcined to concentrate the silica content by two methods: i) pre-treatment of RH followed by calcination and ii) pre-treatment then leaching of RH with organic and inorganic acids followed by calcination. The detailed steps of experiments are: 1) Pretreatment step where RH is washed thoroughly several times with water to remove any adhering materials, soluble particles, dust and heavy impurities like sand, then it is dried in an air oven at 60 °C for 6 hours to remove part of the volatile components and moisture content. Then the dried RH is grinded, 2) Leaching step where the dried RH is refluxed with inorganic acids (HCl, H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub>) and organic acid (CH<sub>3</sub>COOH) at different concentrations (0.1 N, 1.0 N, 4.0 N) for 2 hours and stirred frequently. Followed by decantation and thoroughly washing with distilled water until the rinse became free from acid, then dried in an oven at 105 °C and 3) Calcination step where pretreated RH and leached RH are calcined in a programmable muffle furnace directly at different temperatures (450, 500, 550, 600, 650 and 700 °C) for different times (1, 2 and 3 hours), and the heating rate is maintained at 10 °C /min. Mass loss for RH is measured after washing, drying and calcination with or without leaching.

### 2.3. Adsorption experiments

To investigate the adsorption capacity for native RH, calcined RH (CRH) and finally for calcined leached RH (CLRH) a group of experiments were done at different pH's (3,7 and 11), 100 ml methylene blue as basic dye and 0.1 g adsorbent. The final dye solution will be analyzed using DR2000 spectrophotometer.

### 2.4. Characterization

Physical and chemical analysis of RH, CRH and CLRH were performed. Thermal gravimetric analysis (TGA) is used to evaluate the thermal stability of the RH and to determine the suitable range of calcination temperature of the RH. X-ray fluorescence (XRF) is used for chemical analysis of the RH, CRH and CLRH at different operating conditions. Amorphous and crystalline structure of produced silica are examined by X-ray diffraction (XRD) and electron scanning microscopy (SEM).

## 3 Results and Discussion

### 3.1. Physical and chemical properties of RH

The physical properties of the Egyptian RH are: odorless, length of 6 mm long and 2 mm thick, bulk density of 98 kg/m<sup>3</sup>, mineral hardness 5-6 Mohr's scale. RH chemical compositions were 36% cellulose, 24% hemi cellulose and 25% lignin and the rest was silica and metal oxides. These results are nearly the same for different regions [19,8,3].

#### 3.1.1. XRF of RH

Table 1 presents the XRF of the raw RH, where SiO<sub>2</sub> content is 18.09%, the summation of metal oxides (Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub>, MgO, CaO, Na<sub>2</sub>O, P<sub>2</sub>O<sub>5</sub>, MnO, CuO and other traces of metal oxides) are 1.04% and the loss of ignition (LOI) is 80.87%. The LOI presents the volatile organic and moisture content in RH.

TABLE 1. XRF results of raw RH.

Metal Oxides	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	TiO <sub>2</sub>	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>	Other traces of metal oxides	LOI
mass %	18.09	0.11	0.06	0.10	0.11	0.16	0.10	0.20	0.07	traces	80.87

### 3.1.2. TGA curve and heat flow of RH

Thermogravimetric analysis (TGA) is an analytical technique used to determine a material's thermal stability and its fraction of volatile components by monitoring the weight change that occurs as a sample is heated at a constant rate. The gas used was Nitrogen, it is inert gas, therefore, showing the effect of heat degradation including carbonization. TGA curve of RH is presented in Fig.1. It is characterized by three temperature zones: from 56 to 225 °C, from 225 to 450 °C and from 450 to 980 °C. The first zone presents the mass loss due to the evaporation of the moisture content and a part of organic compounds, the second zone presents the mass loss changes during heating resulting from vaporizing of the rest of organic matters and the third zone represents a constant mass of the formed amorphous and crystalline structures of silica in RHA. In a desired temperature range, if a species is thermally stable, there will be no mass change observed. The heat flow is nearly constant in the range of 400 -750 °C as noticeable in Fig.2. The TGA and heat flow curves are in good agreement with published data [7,8,17]. Based on the TGA and heat flow results, the calcination temperature of RH was investigated in the range of 450 - 700 °C to determine the suitable calcination temperature for achieving high % of amorphous silica content.

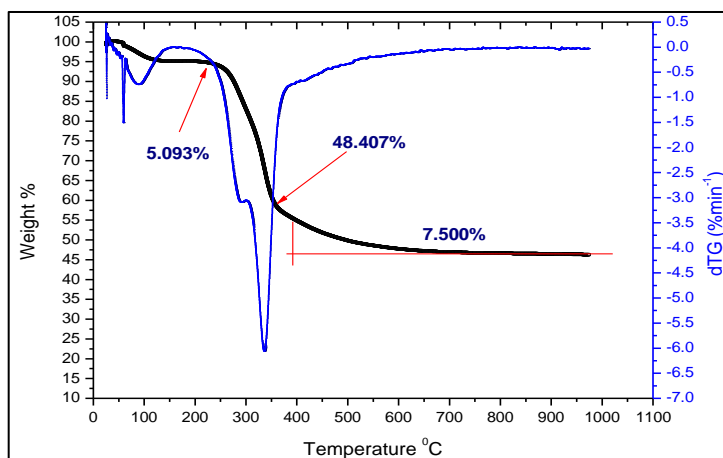


Fig.1: Thermal gravimetric analysis (TGA) for RH.

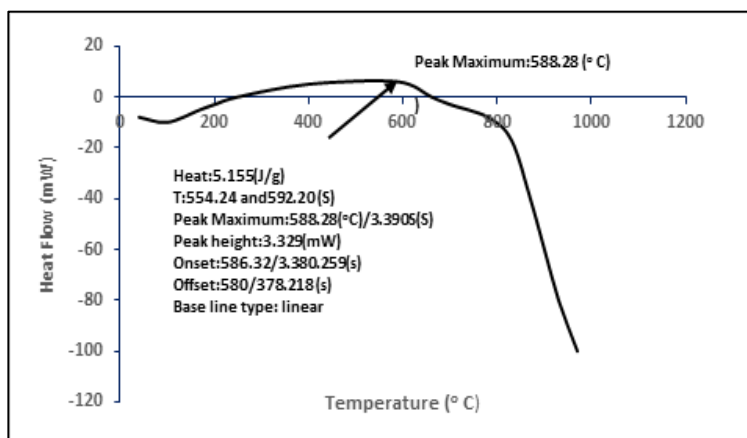


Fig.2: Heat flow for RH.

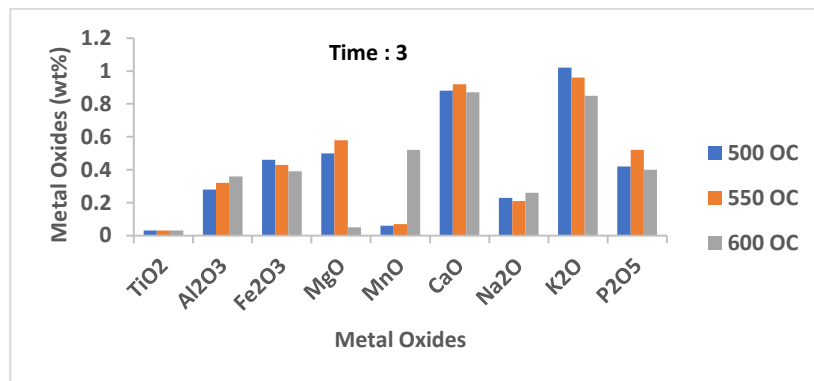
### 3.2. Calcination of RH

Table 2 presents the XRF results for all calcination runs for different temperatures (450 -700 °C) at different times (1, 2, 3 hours) for heating rate of 10 °C /min. It depicts that: i) the silica content percent in the RH was 18.09% and the amorphous silica content in CRH was in the range of 89- 92.3% for the different calcination temperature and time, ii) the highest silica content as amorphous structure was 92.3% in CRH at 500 °C calcination temperature for heating time 2 hours and at heating rate 10 °C /min., iii) metal oxides increases from 1% in RH to 4.45-5.87% in RHA based on the same conditions as a result of volume reduction, and iv) the loss of ignition (LOI) decreased from 80.87% in RH to 2.25 - 5.89% in CRH due to the evaporation of organic and moisture content during heating temperature (450 - 600 °C) for the different times. Fig.3. and Fig.4. present the metal oxides % in CRH at three different temperatures 500, 550, 600 °C for 3 and 2 hours calcination time respectively. It is noticeable that the high % of metals oxides are K<sub>2</sub>O followed by CaO. By raising temperature, K<sub>2</sub>O% decreases from 1.02 to 0.85% and CaO increased from 0.88 to 0.93% for 3 hours calcination time and K<sub>2</sub>O% decreases from 0.97 to 0.94% and CaO increased from 0.83 to 0.89% for 2 hours calcination time.

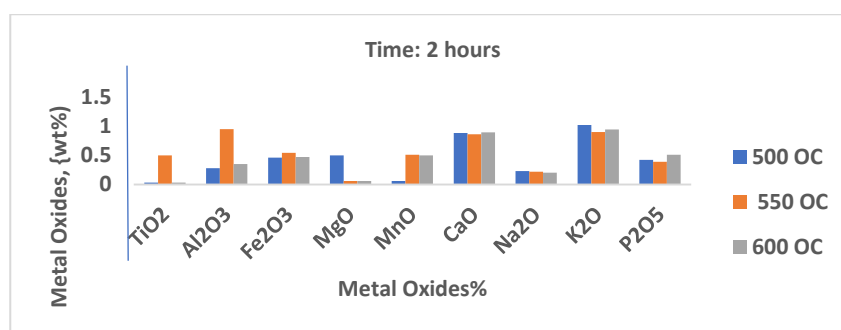
From Table 2 by comparing the silica content for CRH (89.27%) at 450 °C for 3 hours and the silica content (91.85%) at 600 °C at the same calcination time, we can conclude that increasing temperature lead to increasing silica content but raising the temperature higher than 550 °C will led to increase of the existing of crystalline structure of silica which is not appropriate as we need to prepare CRH of amorphous structure with high surface area to be used for adsorption applications.

**TABLE 2:** XRF of RH and RHA at different temperatures and times.

Metal Oxide	Raw RH	RH after Calcination											
		450 °C	500 °C			550 °C			600 °C			650 °C	700 °C
		3 hours	1 hours	2 hours	3 hours	1 hours	2 hours	3 hours	1 hours	2 hours	3 hours	3 hours	3 hours
wt%													
SiO <sub>2</sub>	18.09	89.27	89.66	92.30	91.83	90.90	89.85	92.29	89.40	92.01	91.89	93.42	94.32
TiO <sub>2</sub>	0.11	0.09	0.04	0.03	0.03	0.05	0.5	0.03	0.04	0.03	0.03	0.07	0.07
Al <sub>2</sub> O <sub>3</sub>	0.11	0.40	0.37	0.37	0.28	0.60	0.95	0.32	0.38	0.35	0.36	0.46	0.50
Fe <sub>2</sub> O <sub>3</sub>	0.06	0.53	0.47	0.41	0.46	0.52	0.54	0.43	0.48	0.47	0.39	0.62	0.07
MgO	0.10	0.55	0.46	0.47	0.50	0.07	0.06	0.58	0.06	0.06	0.05	0.61	0.59
MnO	0.01	0.06	0.07	0.06	0.06	0.45	0.51	0.07	0.48	0.50	0.52	0.06	0.07
CaO	0.16	0.88	0.82	0.83	0.88	0.93	0.86	0.92	0.86	0.89	0.87	1.03	0.99
Na <sub>2</sub> O	0.10	0.18	0.17	0.26	0.23	0.19	0.22	0.21	0.19	0.2	0.26	0.25	0.24
K <sub>2</sub> O	0.20	1.55	0.98	0.97	1.02	0.94	0.90	0.96	0.95	0.94	0.85	1.58	1.51
P <sub>2</sub> O <sub>5</sub>	0.07	0.54	0.51	0.41	0.42	0.61	0.39	0.52	0.52	0.51	0.40	0.53	0.61
Traces of NiO, CuO ZnO and traces of metal oxides	0.12	0.56	0.56	1.01	1.34	0.63	0.40	1.42	0.55	0.67	0.98	0.66	0.40
Summation of metal oxides	0.93	5.35	4.45	4.85	5.22	4.99	5.33	5.46	4.51	4.62	4.71	5.87	5.65
LOI	80.87	5.38	5.89	2.85	2.95	4.11	4.82	2.25	6.09	3.37	3.40	0.71	0.03



**Fig. 3:** Metal Oxides % in CRH for different temperatures at 3 hours Calcination time.



**Fig. 4:** Metal Oxides % of CRH for different temperatures at 2 hours Calcination time.

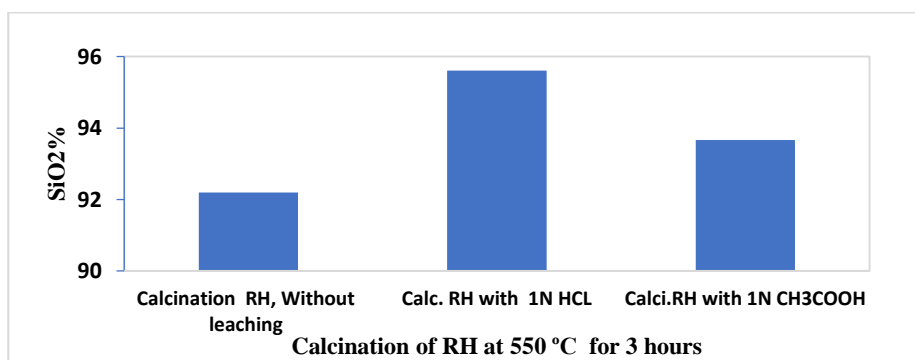
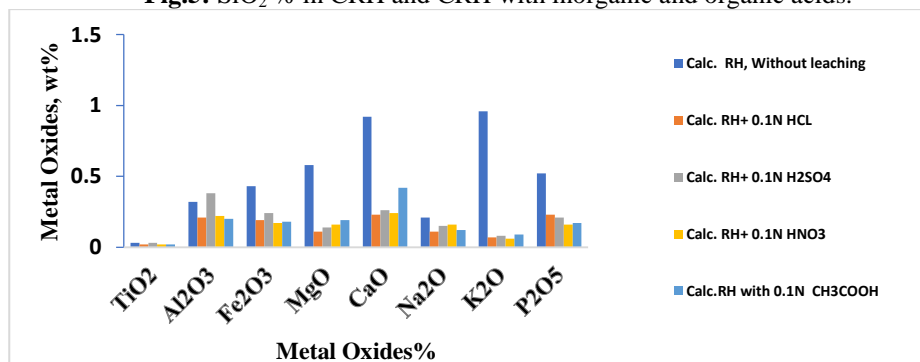
### 3.3. Calcination of leached RH (CLRH)

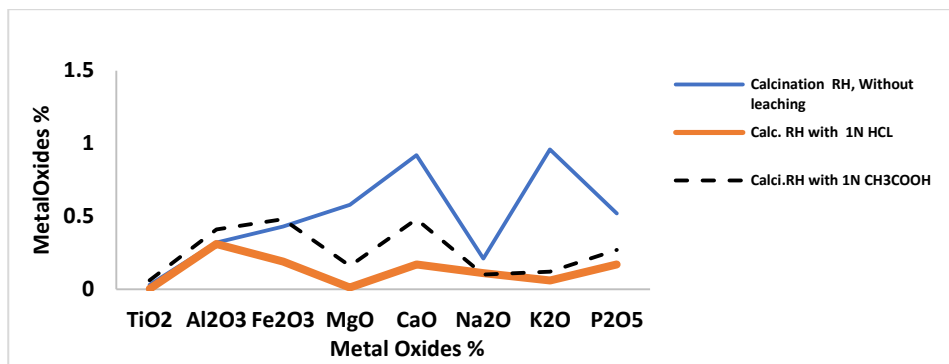
Table 3 depicts the XRF results of CLRH at 550 °C for 3 hours by using three inorganic acids (HCl, H<sub>2</sub>SO<sub>4</sub>, HNO<sub>3</sub>) and one organic acid (CH<sub>3</sub>COOH) at different concentrations. Also the table covered the comparison between the XRF of RH, CRH and CLRH under the same conditions. Calcination temperature and time for CLRH were chosen based on literature survey [17,10]. From Table 3, it is remarkable that the highest silica content achieved was 95.61%, by using 1.0 N HCl at 550 °C as leaching agent for calcination of LRH. The sample treated at 550 °C is characterized by white and grey color as it is mixture of silica and carbon and the silica at 600 °C is a mixture of amorphous silica and crystalline silica. In addition the silica content is raised from 18% in RH to 92.29% in case of CRH and to 95.61% for CLRH by using 1.0 N HCl. Also silica content was 95.61% for CLRH by using inorganic acid (1.0 N HCl) which is high as compared with the 93.66% silica content of the CLRH by using organic acid (1.0 N CH<sub>3</sub>COOH). A comparison between silica content % for CRH and CLRH is presented in Fig.5, which proved that the lowest silica content was 92.29% for CRH without leaching. In addition, the metal oxides percentage in CRH was high compared with RH and CLRH. Fig.6. presents a comparison between the metal oxides remaining in CRH and CLRH with inorganic and organic acid. Fig.7. compares the metal oxides% in RH, CRH and CLRH. It is notable that the metal oxides% is higher in RH without calcination than in CLRH using 1.0 N organic or 1.0 N inorganic acid. Metal Oxides in CRH was 5.55% which is decreased from 4.44 to 1.29 % by leaching with acids with different concentrations. CLRH, leached by inorganic acid (1.0 N HCl) proved 1.29% of metal oxides which is half value (2.48%) of CLRH which leached with organic acid (1.0 N CH<sub>3</sub>COOH).

It can be concluded that leaching process increases the silica content but the difference between the silica content of CRH (91.3%) at 500 °C, 2 hours and the leached one (95.61%) at 550 °C, 3 hours is only 3%, in addition using the un-leached RH is economic as leaching needs using of chemicals (HCl) and high energy as temperature and time of calcination are higher. So it is recommended to use CRH later on for extraction of silica in amorphous form to be used in adsorption applications.

**TABLE 3:**The XRF analyses of RH, calcined RH and calcined leached RH at 550 °C for 3 hours.

Metal Oxides %	Raw RH	Calcined RH Without leaching	Calcined Leached RH							
			0.1 N HCl	1.0 N HCl	4.0 N HCl	0.1 N H <sub>2</sub> SO <sub>4</sub>	0.1 N HNO <sub>3</sub>	0.1 N CH <sub>3</sub> COOH	1.0 N CH <sub>3</sub> COOH	4.0 N CH <sub>3</sub> COOH
			wt%							
SiO <sub>2</sub>	18.09	92.20	92.47	95.61	93.41	94.72	94.33	94.37	93.66	93.47
TiO <sub>2</sub>	0.11	0.03	0.02	0.003	0.02	0.03	0.02	0.02	0.06	0.02
Al <sub>2</sub> O <sub>3</sub>	0.11	0.32	0.21	0.31	1.38	0.38	0.22	0.20	0.41	0.63
Fe <sub>2</sub> O <sub>3</sub>	0.06	0.43	0.19	0.19	0.15	0.24	0.17	0.18	0.48	0.19
MgO	0.10	0.58	0.11	0.011	0.12	0.14	0.16	0.19	0.16	0.13
CaO	0.16	0.92	0.23	0.17	0.21	0.26	0.24	0.42	0.48	0.30
Na <sub>2</sub> O	0.10	0.21	0.11	0.11	0.11	0.15	0.16	0.12	0.10	0.15
K <sub>2</sub> O	0.20	0.96	0.07	0.06	0.8	0.08	0.06	0.09	0.12	0.06
P <sub>2</sub> O <sub>5</sub>	0.07	0.52	0.23	0.17	0.14	0.21	0.16	0.17	0.27	0.22
Traces of NiO, CuO ZnO and other metal oxides	0.018	0.124	0.025	0.025	0.042	0.035	0.029	0.039	0.057	0.063
Summation of metal oxides	1.04	5.55	4.43	1.29	2.47	2.00	1.47	1.75	2.48	1.99
LOI	80.87	2.25	3.10	3.10	4.12	3.28	4.21	3.88	3.86	4.54

**Fig.5:** SiO<sub>2</sub> % in CRH and CRH with inorganic and organic acids.**Fig.6:** Metal oxides in calcined RH without leaching and leaching with 0.1 N of different acids.



**Fig.7:** Metal Oxides comparison between CRH without leaching and CLRH using 1.0 N HCl at 550 °C, 3 hours.

Accordingly, an additional experiment (run #22) was operated at same conditions, but at 2 hours calcination time for the sake of comparison and cost wise point of view. The comparison results are illustrated in Table 4, revealing that CLRH of high silica content is achieved at 550 °C and 3hours.

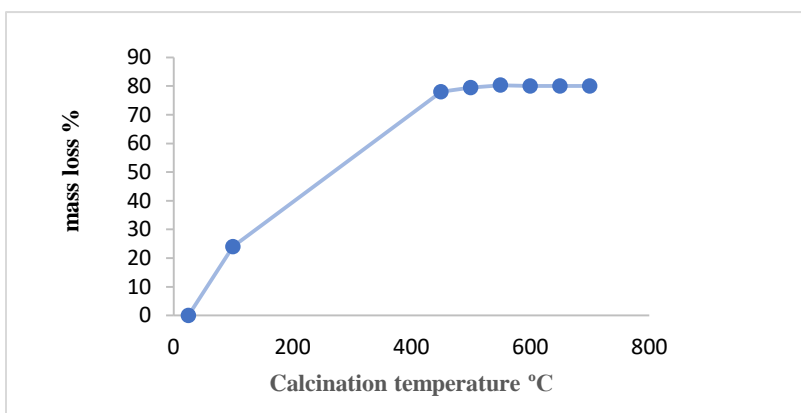
**Table 4:** XRF comparison of CLRH (by using 1.0 N HCl) for two different calcination times (3 & 2 hours) for 550 °C.

Metal Oxides %	Calcination of leached RH at 3 hours	Calcination of leached RH at 2 hours
SiO <sub>2</sub>	95.61	92.47
TiO <sub>2</sub>	0.003	0.28
Al <sub>2</sub> O <sub>3</sub>	0.31	0.2
Fe <sub>2</sub> O <sub>3</sub>	0.19	0.21
MgO	0.011	0.008
CaO	0.17	0.28
Na <sub>2</sub> O	0.11	0.11
K <sub>2</sub> O	0.06	0.051
P <sub>2</sub> O <sub>5</sub>	0.17	0.067
Traces of NiO, CuO ZnO and other metal oxides	0.025	0.174
Summation of metal oxides	1.29	1.206
LOI	3.10	6.15

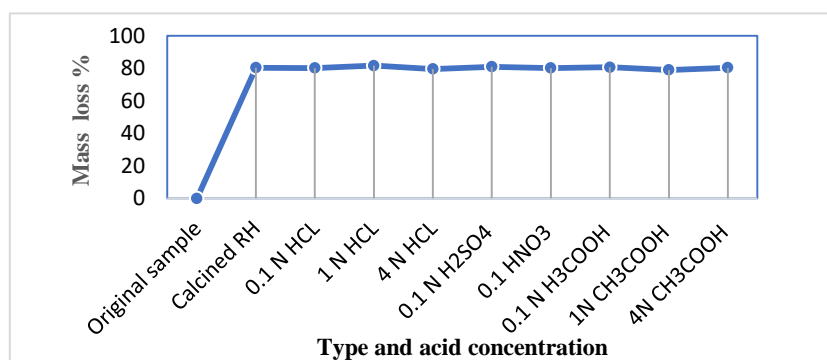
### 3.4 Effect of calcination temperature on mass loss of CRH and CLRH

From Fig.8., the mass loss of the CRH in the first hour of calcination was 24% then increased to 78% at 450 °C and nearly remains constant thereafter, for the higher different temperatures (450 -700 °C) and different times (1-3 hours). Fig.9. demonstrates the mass loss of LRH after calcination at 550 °C for 3 hours. The mass loss was in the range of 79-81% based on different acids concentrations. The results of both CRH and CLRH are in good agreement with the TGA Test.





**Fig.8:** Mass Loss of CRH after Calcination.



**Fig.9:** Mass loss of CRH and CLRH at 550 °C for 3 hours calcination.

### 3.5. Appearance of raw RH and RHA

Figs. 10a, 10b, 10c, 10d and 10e illustrate the appearance of RH, CRH and CLRH at different conditions, where Fig.10a. presents the Raw RH. It is yellow color of needles shape, Fig.10b. presents the raw grind RH. It is yellow powder, Fig.10c. presents the silica content produced by combustion of RH at 550 °C for 3 hours without air flow. It is produced in a powder form of grey color (a mixture of white and black color) as it contains unburned carbon resulted from the lack of air flow, Fig.10d. presents the production of a pure white silica in CRH as a result of complete combustion due to using of steady state air flow, where calcination was investigated at 500 °C for 2 hours. Fig.10e. presents also pure white color silica resulted from CLRH which leached by using 1N HCl at 550 °C for 3 hours in presence of steady state air flow to complete combustion of LRH.

### 3.6. X-Ray Diffraction Analysis (XRD)

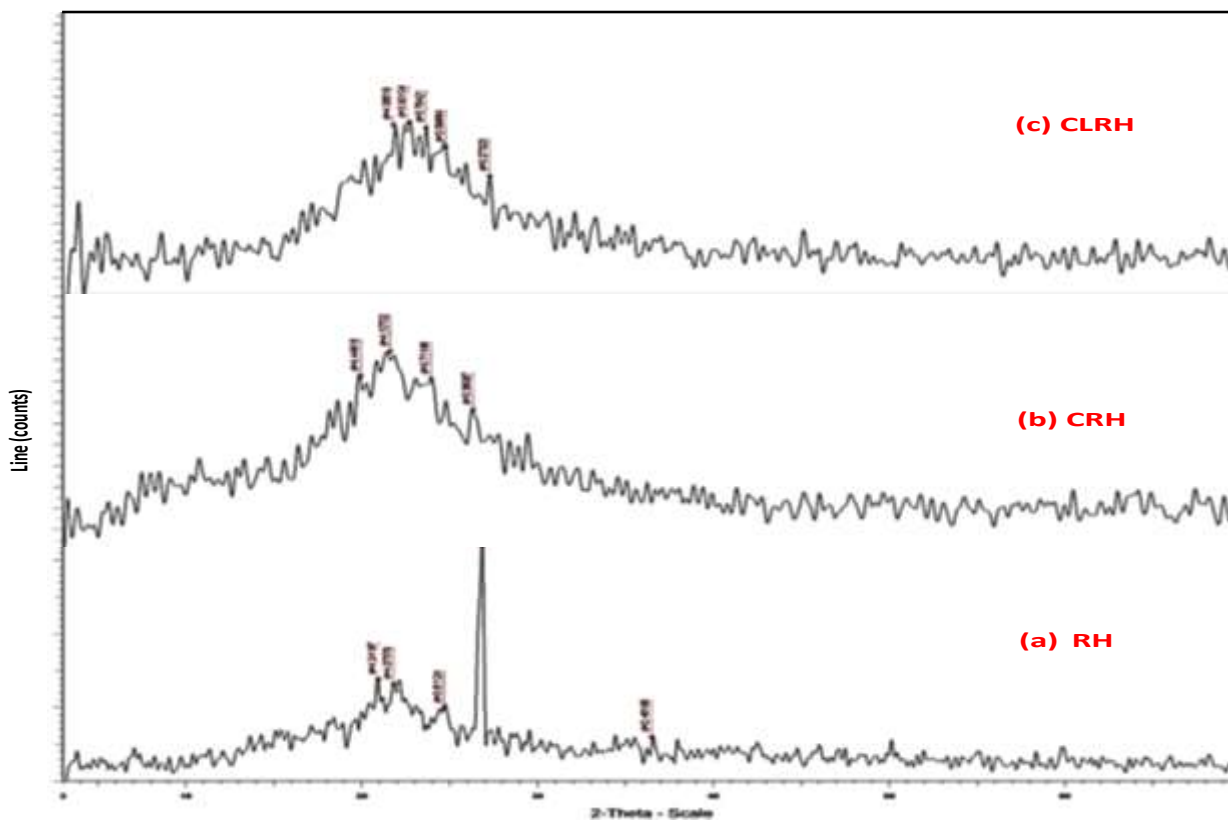
The XRD of grind RH, CRH and LCRH are presented in Figs. 11a, 11b, 11c, 11d, where Fig. 11a. presents grind RH. It is characterized by silica in two theta position ranging from 19 - 27 and from 24 - 45 (two large peaks). The major silica oxide percentage is existing in form of zeolite (84.7%) and the rest are silicon dioxide (15.3%). The small peaks indicate the impurities. Fig. 11b. presents the high silica content (92%) of CRH at 500 °C and for calcination time of 2 hours. Sharp peak is observed in the 2 theta position ranging from 9 - 11 and 22 - 27. It is mainly amorphous silica particles (85% Tridymite). These results in good agreement with Chandrasekhar et al.; 2003 [10]. Fig. 11c. presents the high silica content (95.6%) of CLRH at 550 °C, 3 hours calcination time. Hump peak is observed in the 2 theta position ranging from 10 - 12 and 19 - 30. It is mainly amorphous silica particles, these results in good agreement with Rosario et al.; 2012 [16]. Fig.11d. presents the CLRH at 700 °C for 3 hours. It is remarkable that there are many peaks which indicate a mixture of amorphous and crystalline structure. The crystalline phase is  $\alpha$ -quartz. Therefore the amorphous silica structure in CRH is existing in the range of 450 – 600



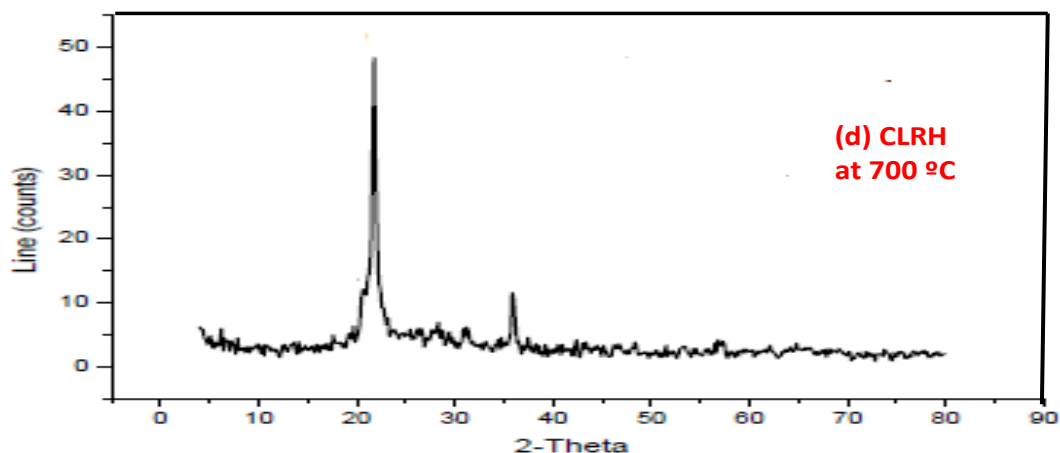
°C and a mixture of amorphous and crystalline structures are found above this range as shown in Fig.11d of CLRH which is matched with Ghosh et al.; 2013 [20] and Libreal et al.; 2019 [1].



**Figs.10.** (a-e) Appearance of RH, CRH and CLRH by different treatment schemes.

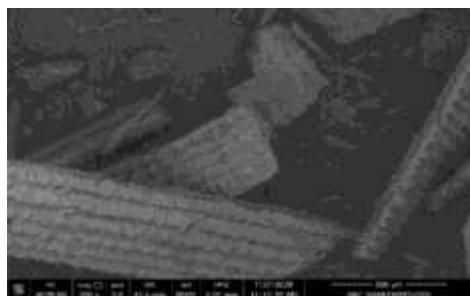


**Fig.11:** XRD by different treatment scheme (grind RH, CRH at 500 °C, 2 hours and CLRH at 550 °C for 3 hours).

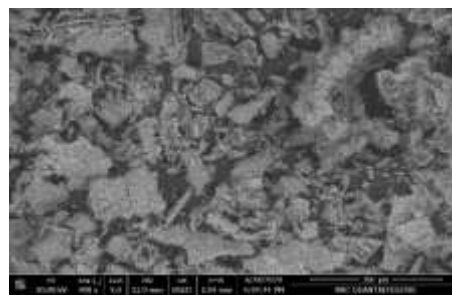


### Microscopic examination

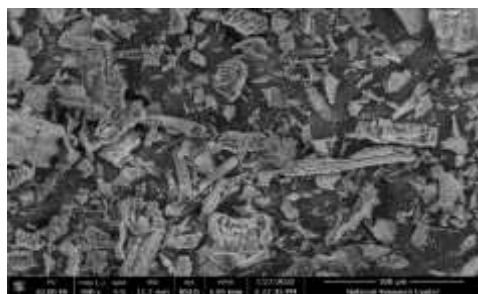
The surface morphology of raw RH and selective samples of CRH and CLRH was examined by using a scanning electron microscope (SEM). The morphological features of the samples are presented in Figs. 12(a-d). and characterized. In Fig. 12 a., RH has a globular structure in nature, of which its main components are in the lemma or palea form, tightly interlock with another. The corrugate structural outer epidermis is highly ridged, whereas the biomass is assembled around the stable Si-O. Many cavities having varying particle sizes were indicated distributing within the sample, evidenced of the interconnected porous network and large internal specific surface area. In Fig. 12 b., it is observed that, the structure of CRH is plan; the silica backbone is probably grown over the plan surface. In Figs. 12 (c & d), CLRH samples, it is observed various sizes and geometry, tubular-shaped porous aggregates and spherical and fibrous particles in the inner surface of the CLRH (for inorganic and organic leaching acids (HCl and acetic $\text{CH}_3\text{COOH}$ ) at 550 °C and for 3 hr. The spherical particles contain mostly silica and metal oxides. Also the particle became spongy and porous structure. These results are in good agreement to Farshid et.al.; 2015 [4] results.



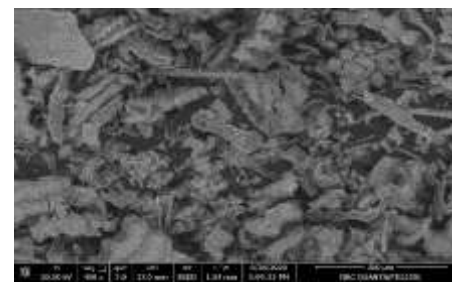
(a) Grind RH



(b) CRH, 500 °C, 2 hours



(c) CLRH by leaching with 1N HCL,  
550 °C, 3 hrs.



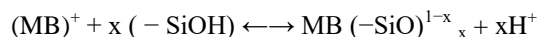
(d) CLRH by leaching with 0.1 N  
 $\text{CH}_3\text{COOH}$ , 550 °C, 3 hrs.

**Fig.12 (a-d).** Microscopic Examinations of RH, CRH and CLRH.

### 3.8. Adsorption Capacity

Table (5) represents the adsorption capacity (mg/g) of RH, CRH and CLRH for the removal of methylene blue. The highest capacity was found for CRH at pH 11.

Basic dyes upon dissolution release dye cations in the solution. Adsorption of these charged dye groups on to the adsorbent surface is primarily influenced by the surface charge on the adsorbent. The surface of rice husk ash is negatively charged due to the presence of –OH groups and hence has a high adsorption capacity for cationic (basic) dyes. The higher pH of the ash also supports this. It is primarily the hydroxyl groups that determine the chemistry (acid base character) and reactivity of these surfaces.



Even though the pH has impact on the adsorption capacity, its influence is not as critical as that of surface area and pore volume. For a good adsorbent of methylene blue, its pH should be  $\geq 7$  and surface area and pore volume should be large.

Table ( ): The adsorption capacity of RH, CRH and CLRH for the removal of methylene blue.

pH	Capacity, mg/g (RH)	Capacity, mg/g (CRH)	Capacity, mg/g (CLRH)
3	1.9	72.1	69
7	83.65	92.9	93.69
11	90.9	98.16	96.29

**Table (3):** BET of RH, CRH, CLRH.

	RH	CRH	CLRH
<b>Surface area</b>	of 36.4031 m <sup>2</sup> /g	155.944 m <sup>2</sup> /g	291.298 m <sup>2</sup> /g
<b>Pore volume</b>	ND	ND	3.6482e-001 cc/g
<b>Pore radius</b>	ND	ND	2.5048e+000 nm

ND; Not determine for problem in the BET instrument

## 4 Conclusion and recommendations

Preparation of RHA of high silica content, were investigated based on the calcinations of RH (CRH) and leached RH (CLRH). The RH is calcined to concentrate the silica content by two methods: 1) pre-treatment of RH followed by calcination and 2) pre-treatment then leaching of RH followed by calcination. Characterization of RH, CRH and CLRH was carried out by XRF and XRD. XRF Analyses of the Egyptian RH proved 18.09% SiO<sub>2</sub> content. Metal oxides in RH was nearly 1% which increases to 5.5% in case of CRH and decreases in the range of 1.47 - 4.43% for CLRH based on temperature and time of calcination. Amorphous silica content of 91.3% was obtained at 500 °C after 2 hours calcination time of RH, but CL RH proved higher silica content of 95.61% by using 1.0 N HCl as leaching agent at 550 °C for 3 hours. For both CRH and CLRH metal oxides were less than 0.1% for CRH and 0.025% for CLRH. Therefore it can be concluded that leaching process increases the silica content but the difference between the silica content of CRH (91.3%) at 500 °C, 2 hours and the leached one (95.61%) at 550 °C, 3 hours is only 3%, in addition using the un-leached RH is economic as leaching needs using of chemicals (HCl) and high energy as temperature and time of calcination are higher. So it is recommended to use CRH later on for extraction of amorphous silica to be used in adsorption applications

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