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Production of Silica from Rice husk

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Abstract: Rice husk (RH) is an agricultural waste, which is easily available in the market and Silica (SiO_2) is the most abundant material in the earth's crust. This paper addresses the production of silica from the rice husk. The Rice husk ash (RHA) is obtained by burning of rice husk in a muffle furnace at a temperature of 500, 600 and 700°C, which is bio-organic Silica (SiO_2) and has high pozzolanic activity. The temperature and the time duration for the combustion process are optimized. X-ray Fluorescence (XRF) analysis is carried out to determine the chemical composition of rice husk and that of the rice husk ash. The formed rice husk ash is treated with Sodium hydroxide (NaOH) and Hydrochloric acid (HCl) to produce silica. The obtained silica is used for the various applications like automotive industry, cosmetic industry, etc. The compound of silica like Sodium silicate (NaSiO_3) is used for water treatment, concrete treatment, cement production, etc. The brief explanation for the process is given below.

Keywords: Rice husk, Rice husk ash, Muffle furnace, Sodium hydroxide, Silica and X-ray Fluorescence.

Introduction:

Rice is the seed of the monocot plants *Oryza sativa* (Asian rice) or *Oryza glaberrima* (African rice). It is normally grown as an annual plant, although in tropical areas it can survive as a perennial and can produce aratoon crop for up to 30 years. Since a large portion of maize crops are grown for purposes other than human consumption, rice is the most important grain with regard to human nutrition and caloric intake, providing more than one fifth of the calories consumed worldwide by the human species. The rice plant can grow to 1–1.8 m (3.3–5.9 ft) tall, occasionally more depending on the variety and soil fertility. It has long, slender leaves 50–100 cm (20–39 in) long and 2–2.5 cm (0.79–0.98 in) broad. The small wind-pollinated flowers are produced in a branched arching to pendulous inflorescence 30–50 cm (12–20 in) long. The edible seed is a grain (caryopsis) 5–12 mm (0.20–0.47 in) long and 2–3 mm (0.079–0.12 in) thick. Rice is the staple food of over half the world's population. It is the predominant dietary energy source for 17 countries in Asia and the Pacific, 9 countries in North and South America and 8 countries in Africa. Rice provides 20% of the world's dietary energy supply, while wheat supplies 19% and maize 5%.¹⁻³

Rice Husk

Rice husks are the hard protecting covering of grains of rice. Rice hulls are the coating for the seeds, or grains, of the rice plant. To protect the seed during the growing season, the hull forms from hard materials, including opaline silica and lignin.

One practice, started in the seventeenth century, to separate the rice from hulls, it to put the whole rice into a pan and throw it into the air while the wind blows. The hulls are blown away while the rice fell back into the pan. This happens because the hull isn't nearly as dense as the rice. These steps are known as winnowing. Later pestles and a simple machine called a rice pounder were developed to remove hulls. In 1885 the modern rice hulling machine was invented in Brazil. During the milling processes, the hulls are removed from the raw grain to reveal whole brown rice, which may then sometimes be milled further to remove the bran layer, resulting in white rice (Table- 1)

Table 1: Composition of rice Husk

Composition	Percentage
Cellulose	31.12
Hemi Cellulose	22.48
Lignin	22.34
Mineral Ash	13.87
Water	7.86
Extractives	2.33

Stage 1: Rice Husk Ash Production

Production Methods

Burning In Electric Furnace

The rice husk is burned in an electric furnace at 900⁰C under atmospheric pressure for 6 hrs. It produces a black residue with high crystallinity. The residues black colour can be significantly reduced only if heat treated at temperatures above 1100⁰C for over 2 hrs. The properties of this type of residue are very similar with the residues produced in thermoelectric plants.

Acid Treatment of the Husk

Organic acids such as acetic or citric acid are used to remove alkaline impurities as the initial step. The next step is the calcination in which the treated acid reacts with oxygen to produce carbon-dioxide and water. The reaction between acetic acid and rice husk may results in the formation of esters which is water insoluble and less dense will float on the surface of the water in the autoclave or may be expelled from the autoclave together with water vapour.

Fluidized Bed Combustor

Rice husk has poor flow characteristics rendering it difficult to be handled and fed into the fluidised bed. It has low bulk density and interlocking nature. Feeding of such low density biomass materials into the fluidised bed combustor is difficult; so, various feeding methods are required which can be done either mechanically by a screw feeder or pneumatically by air.⁴⁻⁸

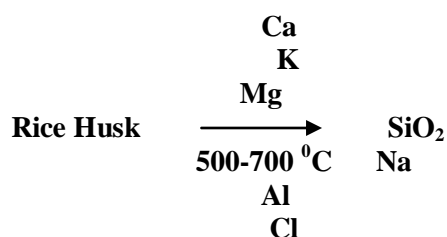
Procedure

Seive Analysis

For obtaining the rice husk of uniform size, we have done the sieve analysis by using mesh size of 1.4mm, 850 microns, 500 microns, 425 microns, 355 microns, 250 microns, 75 microns and pan respectively. Place a small quantity of rice husk in the first mesh. Then, do the sieve analysis and take the rice husk of mesh size 1.4 mm because it is in large quantity.

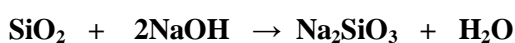
Stage 1: Preparation of Rice Husk Ash

5grams of rice husk is taken in the crucible. Place it in the muffle furnace at a set temperature of 700⁰C⁴. After reaching 700⁰C, place it for 1 hour inside the furnace itself. Then, switch off the furnace. And take the sample out by using tongs. Then, check the amount of ash retained by using weighing balance. Do this for 2 hours, 3 hours, 4 hours, 5 hours and 6 hours respectively. Then, the same procedure is repeated for 600⁰C and 500⁰C⁸. X-Ray fluorescence method is used to determine the silica composi



Stage 2: Preparation of Sodium Silicate from Rice Husk Ash

Take 2grams of NaOH pellets and put in a 50ml of water. Stir the solution till the pellets get dissolved in water. Take 1gram of ash from the above experiments in the stage 1. Put it in the NaOH solution and mix it thoroughly by using glass rod. Place it in the hot air oven at a temperature of 100°C for 1 hour. After 1 hour, take it from the hot air oven and take the obtained precipitate by using filter paper. Again, dry the residue at 60°C in the hot air oven for half an hour. The obtained product is sodium silicate.



Preparation of Silica from Sodium Silicate

Prepare 300 ml 1N HCl. HCl solution is added to sodium silicate. Keep it in an ice water bath for 1hour. Filter the solution. Residue is taken out and dried it in a hot air oven at about 60°C^{6,10}. The obtained product is called as silica which is in nano scale¹.



Results and Discussions

The production of silica has been progressed in two stages as said in the above procedure. Rice husk ash has been produced and the ash weight is found to lie around 17 grams. The temperature has been varied along with different time intervals to optimize the time and temperature required to produce rice husk ash. The below (Tables- 2-15) shows a comparison of weight percentages of the ash produced for different temperatures and time periods.

Table 2: Comparison of weight percentages of ash produced for different temperatures and time periods

Time (Hours)	500 ^o c (Temperature)	600 ^o c (Temperature)	700 ^o c (Temperature)
1	23.15	22.46	18.90
2	19.02	17.94	17.42
3	18.98	17.52	17.15
4	18.96	17.50	17.18
5	18.74	17.31	17.18
6	18.74	17.35	17.15

X-Ray Fluorescence Analysis

Rice Husk

Table 3: X-Ray Fluorescence analysis for Rice Husk

Compound No:	Compounds Present	Concentration Units (Cps)	% Of Compounds Present
1.	Na	62.812	0.68
2.	Mg	10.311	0.11
3.	Al	9.940	0.109
4.	Si	2969.177	32.60
5.	Cl	6075.056	66.70
6.	K	22.879	0.25
7.	Ca	-42.583	-

FOR 700°C**Table 4 : X-Ray Fluorescence analysis for Rice Husk ash for 3 hours at 700°C**

Compound No:	Compounds Present	Concentration Units (Cps)	% Of Compounds Present
1.	Na	22.816	0.199
2.	Mg	8.356	0.073
3.	Al	32.553	0.284
4.	Si	7185.981	62.82
5.	Cl	1608.976	14.066
6.	K	1677.282	14.663
7.	Ca	902.381	7.889

Table 5: X-Ray Fluorescence analysis for Rice Husk ash for 2 hours at 700°C

Compound No:	Compounds Present	Concentration Units (Cps)	% Of Compounds Present
1.	Na	44.066	0.33
2.	Mg	12.910	0.097
3.	Al	31.976	0.24
4.	Si	8161.131	61.53
5.	Cl	4920.219	37.096
6.	K	82.445	0.6215
7.	Ca	10.693	0.0806

Table 6: X-Ray Fluorescence analysis for Rice Husk ash for 4 hours at 700°C

Compound No:	Compounds Present	Concentration Units (Cps)	% Of Compounds Present
1.	Na	39.658	0.3024
2.	Mg	11.198	0.0854
3.	Al	29.626	0.22
4.	Si	7386.775	56.33
5.	Cl	5479.839	41.792
6.	K	130.803	0.997
7.	Ca	33.991	0.259

Table 7: X-Ray Fluorescence analysis for Rice Husk ash for 5 hours at 700°C

Compound No:	Compounds Present	Concentration Units (Cps)	% Of Compounds Present
1.	Na	34.950	0.3033
2.	Mg	10.086	0.0875
3.	Al	24.644	0.213
4.	Si	7002.873	60.78
5.	Cl	4381.544	38.03
6.	K	47.856	0.415
7.	Ca	18.981	0.164

FOR 600°C:**Table 8 : X-Ray Fluorescence analysis for Rice Husk ash for 3 hours at 600°C**

Compound No:	Compounds Present	Concentration Units (Cps)	% Of Compounds Present
1.	Na	23.944	0.1869
2.	Mg	8.843	0.06
3.	Al	35.606	0.27
4.	Si	8212.423	64.12
5.	Cl	1773.346	13.84
6.	K	1811.536	14.14
7.	Ca	941.938	7.35

Table 9: X-Ray Fluorescence analysis for Rice Husk ash for 4 hours at 600°C

Compound No:	Compounds Present	Concentration Units (Cps)	% Of Compounds Present
1.	Na	14.173	0.118
2.	Mg	7.944	0.06
3.	Al	35.816	0.298
4.	Si	7914.609	65.95
5.	Cl	1097.065	9.14
6.	K	1812.573	15.10
7.	Ca	1116.975	9.30

Table 10: X-Ray Fluorescence analysis for Rice Husk ash for 4.5 hours at 600°C

Compound No:	Compounds Present	Concentration Units (Cps)	% Of Compounds Present
1.	Na	15.921	0.137
2.	Mg	68.92	0.059
3.	Al	34.880	0.300
4.	Si	7802.924	67.22
5.	Cl	1010.824	8.70
6.	K	1739.700	14.987
7.	Ca	996.547	8.58

AT 500°C**Table 11: X-Ray Fluorescence analysis for Rice Husk ash for 5 hours at 500°C**

Compound No:	Compounds Present	Concentration Units (Cps)	% Of Compounds Present
1.	Na	21.405	0.170
2.	Mg	7.546	0.060
3.	Al	44.929	0.357
4.	Si	8333.721	66.29
5.	Cl	1694.598	13.480
6.	K	1585.131	12.609
7.	Ca	883.675	7.029

Table 12: X-Ray Fluorescence analysis for Rice Husk ash for 6 hours at 500°C

Compound No:	Compounds Present	Concentration Units (Cps)	% Of Compounds Present
1.	Na	35.520	0.253
2.	Mg	8.124	0.057
3.	Al	35.166	0.25
4.	Si	8139.479	58.07
5.	Cl	5549.598	39.59
6.	K	192.055	1.370
7.	Ca	35.520	0.253

Table 13: X-Ray Fluorescence analysis for Rice Husk ash for 7 hours at 500°C

Compound No:	Compounds Present	Concentration Units (Cps)	% Of Compounds Present
1.	Na	24.647	0.193
2.	Mg	9.929	0.077
3.	Al	39.451	0.309
4.	Si	8189.558	64.18
5.	Cl	1924.368	15.08
6.	K	1650.145	12.933
7.	Ca	920.929	7.21

Stage 2

Ash Treated With Sodium Hydroxide (NaOH)

Different normalities of sodium hydroxide are used for different time periods and the weight of sodium silicate has been found and tabulated below.

Table 13: Weight of sodium silicate produced for different concentrations and time period

Normality (N)	Time (Hour)	Weight Of Na_2SiO_3 (G)
1	1	0.66
1	2	0.35
2	1	0.42
2	2	0.34

Sodium Silicate Treated With Hydrochloric Acid

To the precipitated sodium silicate solution, 1N Hydrochloric acid has been added and the percentage of silica extracted is found to be 62.8 %

Table 14: Weight of silica produced for 1N Hcl

Normality (N)	Time (Hour)	% Of Silica
1	1	62.8

The below graph (Figure- 1-12) shows the optimized trend for the rice husk ash produced for different time intervals and different temperatures

Figure 1: Weight percentages of ash produced for different time periods

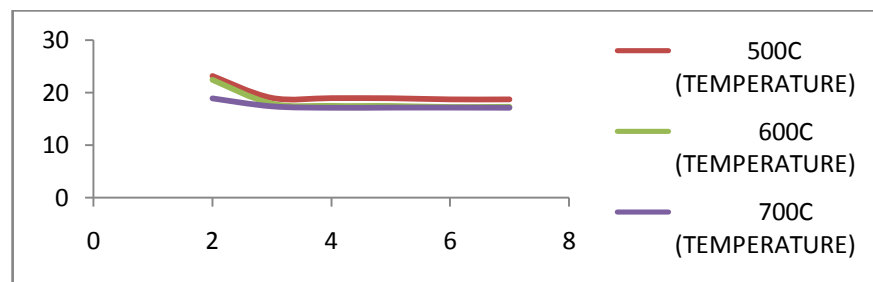


Figure 2 : X-Ray Fluorescence analysis for Rice Husk

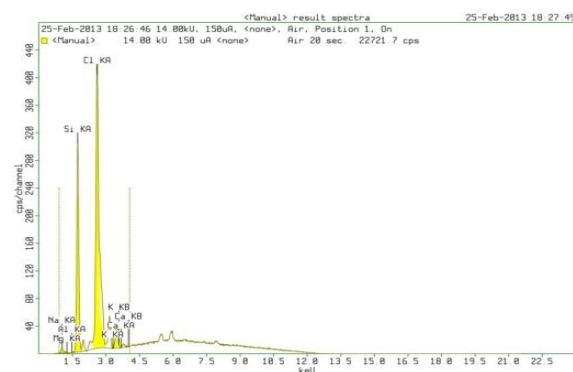


Figure 3 : X-Ray Fluorescence analysis for Rice Husk ash for 3 hours at 700°C

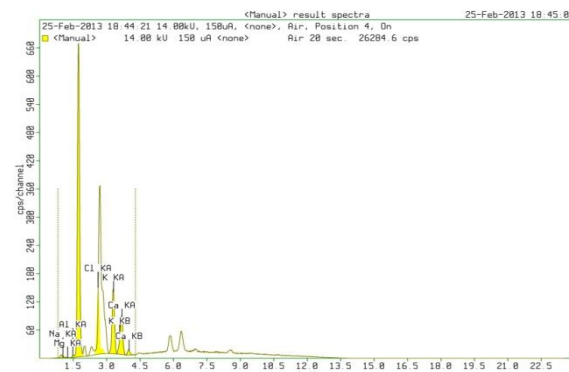


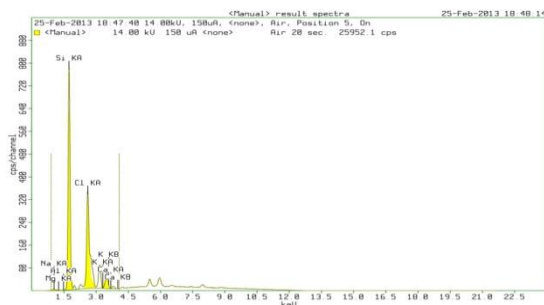
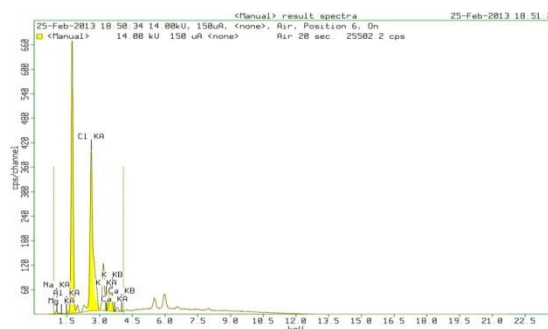
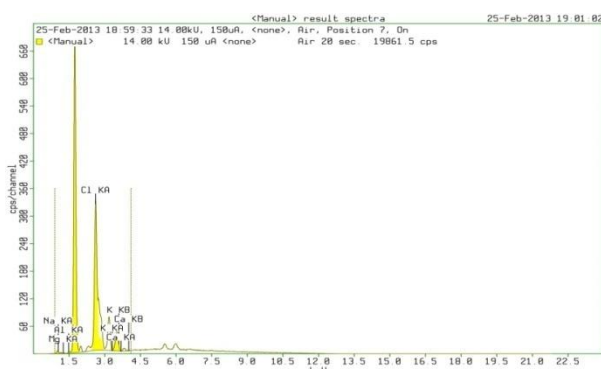
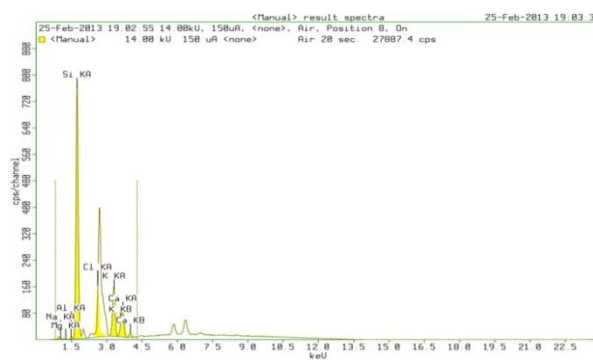
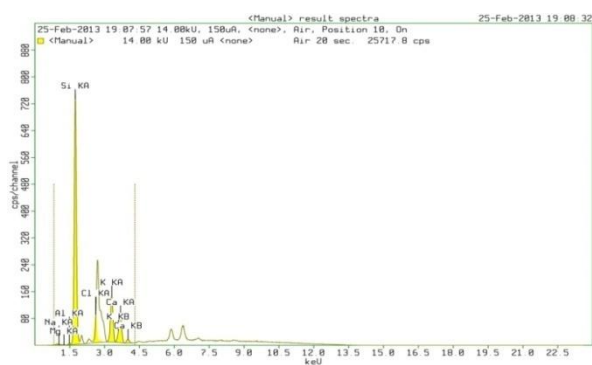
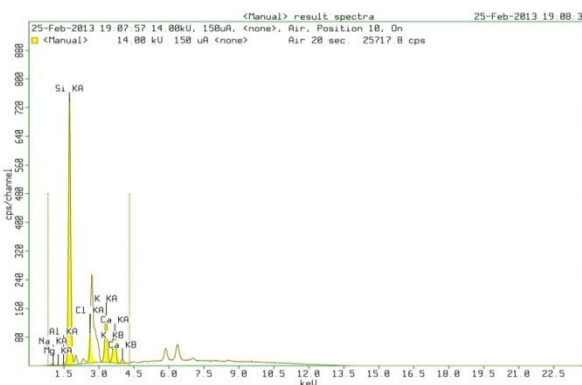
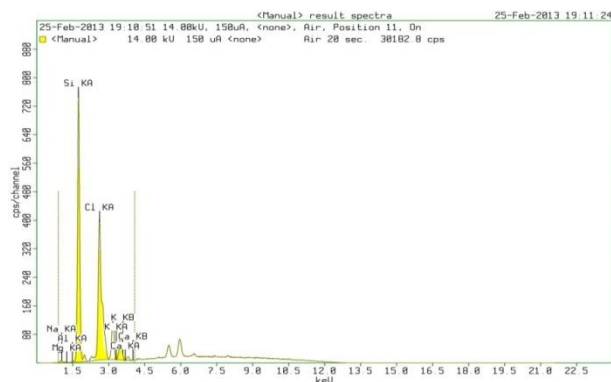
Figure 4 : X-Ray Fluorescence analysis for Rice Husk ash for 2 hours at 700⁰C**Figure 5 : X-Ray Fluorescence analysis for Rice Husk ash for 4 hours at 700⁰C****Figure 6 : X-Ray Fluorescence analysis for Rice Husk ash for 5 hours at 700⁰C****Figure 7 : X-Ray Fluorescence analysis for Rice Husk ash for 3 hours at 600⁰C****Figure 8 : X-Ray Fluorescence analysis for Rice for 4 hours at 600⁰C****Figure 9 : X-Ray Fluorescence analysis for Husk ash Rice Husk ash for 4.5 hours at 600⁰C**

Figure 10: X-Ray Fluorescence analysis for Rice Husk ash for 5 hours at 600⁰C



For Rice Husk Ash (6 Hours):

Figure 11 : X-Ray Fluorescence analysis for Husk ash for 6 hours at 500⁰C

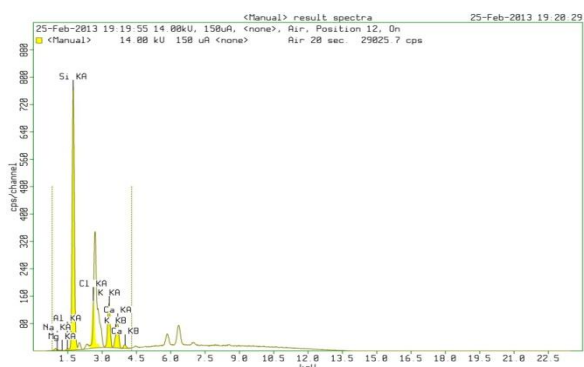
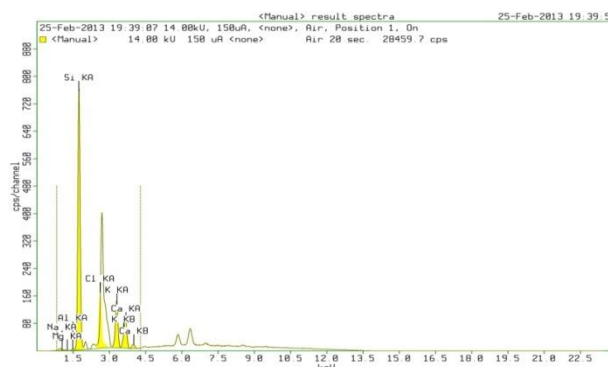


Figure 12 : X-Ray Fluorescence analysis for Rice Husk ash for 7 hours at 500⁰C



Conclusion

Rice husk ash has been produced from rice husk using a muffle furnace, optimizing the temperatures and time durations. Optimum time for producing rice husk ash at 700⁰C is 3 hours, 800⁰C is 4.5 hours, 900⁰C is 6 hours respectively. The Silica composition in the rice husk ash has been analysed using X-ray Fluorescence (XRF) analysis. Silica has been produced by a two step process and the percentage extraction lies around 62%.

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