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A Review Study on Precipitated Silica and Activated Carbon from Rice Husk

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Abstract

The present work entitled "Precipitated Silica and Activated Carbon from Rice Husk" was taken up to study the optimum operating conditions at laboratory scale. Precipitation of Silica was carried out using various parameters such as pre-treatment or acid washing, carbonization temperature of acid washed rice husk, activating agent such as sodium hydroxide, time of carbonization, time of heating with activating agent. The activated carbon was obtained from rice husk ash at various concentration of activating agent (Sodium Hydroxide) and different activation temperature. These studies indicate that nano-silica powder can be produced by optimizing the parameters used in the study. Without optimization, one would get different grades of silica with different yields. Similarly, different grades of activated carbon can be obtained by varying the parameters of activating agent, carbonizing temperature after activation and time of carbonization. The article presents a procedure to produce nano-silica powder from rice husk along with wastewater treatment grade of activated carbon.

Keywords: Activated carbon; Pretreatment; Optimum condition; Laboratory scale; Nano-silica powder; Carbonizing temperature

Introduction

Rice husk, an agro waste material, contains about 20% ash which can be retrieved as amorphous, chemically reactive silica. This silica finds wide applications as filler, catalyst support, adsorbent and a source for synthesizing high performance silicon and its compounds. Various metal ions and unburned carbon influence the purity and color of the ash. Controlled burning of the husk after removing these ions can produce white silica of high purity. India produces around 25 million tons of rice husks (widely available waste). 78% of weight as rice, broken rice and bran, rest 22% of weight of paddy as husk. 75% of organic volatile matter and 25% of weight is converted as Rice Husk Ash (RHA) during firing process. Husk contains 17%-20% silica in complex form and RHA contains 85%-95% amorphous silica. RHA is a great environment threat causing damage to the land and the surrounding area in which it is dumped.

The study focuses on samples from the state of Andhra Pradesh Rice Husk (APRH) in the central part of India and the other from Kerala (KRH) the southernmost part of the country. Leaching the husk with acetic and oxalic acids was attempted for the first time and the improvement in properties of the ash was studied. The husk samples were also treated with hydrochloric and nitric acids of different concentrations for comparison. The ashes produced by controlled burning of these samples before and after acid treatment, were characterized for the optical properties in addition to the chemical and physical nature. The APRH ash was found to be inferior to the KRH ash in all properties. Pretreatment of the husks with the organic acids improved the properties of ashes and the effect was comparable to that achieved by mineral acid leaching. Amorphous, reactive and high purity silica with high surface area and pore volume and good optical properties could be prepared from both the husks under specific conditions. Rice husk ash or silica ash, as it is commonly called, is classified as an industrial waste obtained after burning the rice husks. It has approximately 55-97% silica in partly crystalline and amorphous forms, depending upon the prior combustion conditions. Though there are limitations in its application as filler in thermoplastics [1,2], its usage in various industries varies depending upon its purity and particle characteristics [3,4]. Industrial by-products (e.g., saw dust, rice husks) and a recent entry in the form of silica ash – an industrial waste material – obtained by burning rice husks. Rice hulls possess an unusually high percentage of 'opaline silica'. Its annual worldwide output is more than 80 million tons, which corresponds to 3.2 million tons of silica. Rice husk on burning gives 14–20% ash which contains 80–95% silica in the crystalline form and minor amounts of metallic elements. By controlling the burning conditions like temperature and time, amorphous silica of ultrafine size and reactivity can be produced. Pretreatment of the husk with mineral acids followed by controlled ashing gives silica with high purity. Rice husk is a byproduct in rice mills and creates disposal and pollution problems. It is reported that about 30 million tons of rice husk per annum is produced in India. Therefore, an efficient utilization of RH is urgently needed. A number of papers have been published on various aspects of rice husk [5-7]. The major constituents of rice husk are cellulose, lignin and ash [7]. The chemical constituents is found, it varies from sample to sample which may be due to the different geographical conditions, type of paddy, climatic variation, soil chemistry and fertilizers used in the paddy growth (Table 1). The silicon atoms are concentrated in the protuberances and hairs

Constituents	weight
Silica (SiO_2)	94.50
Calcium oxide (CaO)	00.48
Manganese oxide (MnO)	01.09
Magnesium oxide (MgO)	00.23
Iron oxide(Fe_2O_3)	00.54
Aluminum oxide(Al_2O_3)	00.21
S,P_2O_5,K_2O,Na_2O	Traces

Table 1: A typical composition of rice husk

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on the outer and inner epidermis of the husk [8]. The rice husk ash is used as a good pozzolana in cement industry [9-11] and also as a support material for metal catalysts. Preparation of different value added products like SiC, porous carbon, zeolites, cordierite, etc from rice husk ash has been reported [12-19]. It has extensive uses as filler, additive, abrasive, oil adsorbent, sweeping component, suspension agent for porcelain enamels etc. It is also used for soil treatment, water purification, glazing pottery ware, in ceramics and refractory materials, making special quality bricks [20-24].

History of Contribution/Background

Silica is not a new commodity in the plastics market. Its usage as extenders and reinforcing fillers, as pozzolanic material and as glass-microspheres for specific engineering applications are well known in the market [25-28]. Because of its high silica and lignin content, rice husk is tough, woody and abrasive in nature with low nutritive properties and resistance to weathering [29]. With growing environmental concern, open burning has been outlawed in many major rice-producing countries (Figure 1). World rice production shown in percentage wise, are obtained from the U.S. department of agriculture and show that total production is around 80 million ton per annum. The U.S. department of agriculture shows that about 27 million metric ton of milled rice was globally traded for the year 2002. Based on 20% silica content of the rice hulls, it is estimated that effectively 1.2 MT (million ton) of silica ash can be produced from commercial rice. Burning rice hulls – as a preparative step for energy production – is a useful solution, but the desirable situation would be a better economic use of the resulting ash. Silica is the predominant component of the ash with trace amount of various elements such as potassium, sodium, magnesium and calcium (<0.5%). Rice productions around the world in 1999 [1.CHINA-40%, 2.INDIA-20%, 3.INDONESIA-10%, 4. OTHERS-20%].

Silica ash composites It is understood that characteristics such as a fine particle size (<50 micron) and high reactivity (due to its irregular shape, porosity and presence of surface hydroxyl groups) mar the performance of silica ash as a filler. Also, impurities reduce its efficiency as filler in composites. The impurity level of silica is significantly affected by the production mode. With open air burning, a variety of silica ash can be produced with acceptable level of carbon impurities [30]. Amorphous white (low-carbon) silica ash is obtained at temperatures above 500°C and depends on the time and the temperature of combustion [31]. It was postulated that at high temperature (800°C), the presence of potassium led to surface melting and carbon fixation in the lattice [32]. The effect of treating the husk with dilute HCl on the formation of black particles has been recently reported by him. Sidheswaran et al. [33] prepared pure white amorphous silica by pre-treating the husk with HNO_3 followed by ashing and measured the whiteness using a color scanner. Amorphous silica of high purity, small particle size and high surface area has tremendous potential as an adsorbent and catalyst/support in fine chemical synthesis. In order to prepare amorphous silica of high purity, treatment of the husk with chemicals before and after combustion was attempted by various researchers.

Figure 1: Major rice producing states in India.

Mineral acid leaching of rice husk is reported by Chakraverty et al. [31]. They have used acids like HCl, H_2SO_4, and HNO_3 of varying concentrations. They studied the effect of various acid treatments on the removal of metallic ingredients and reported that leaching with 1N HCl is highly effective. Yalcin et al. [34] have described pre and post treatments of husk using HCl, H_2SO_4 and NaOH. Russian inventor Raphael Osterjkio-discovered that adsorptive properties of char could be improved by treatment at higher temperatures in presence of air, steam or CO_2.

Lowitz in 1785 showed-activated carbons could be successfully used for decolorization (utilized in sugar industry). Present day carbons are produced from coal based materials like lignite, pine charcoal, soft-wood charcoal, teak waste, deoiled sandal-wood dust, coconut shells, cotton seeds hulls, and rice husk. Adsorptive properties of activated carbon can be explained as the formation of a complex network of very fine pores during its manufacture. The internal surface area of these pores contributes to a very high total internal surface area in the range of 1000 m^2/gm or more.

Materials & Methods

Analysis of rice husk ash

Precipitation of Silica – Chemical Reaction Involved

$SiO_2 + 2NaOH \rightarrow Na_2SiO_3 + H_2O$

$Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2 + Na_2SO_4 + H_2O$

General process for precipitation of silica and activated carbon from rice husk

A known weight of rice husk is taken and sieved through 20 mesh size. It is then washed, cleaned and heated/carbonized to around 600°C for different time intervals (figure 2). It is treated with an activating reagent such as sodium hydroxide, zinc chloride, phosphoric acid for about 1 hour at 60°C. The activated rice husk and the solution containing the activating agent are then filtered. The activated rice husk so obtained is heated at a temperature of 900°C to get activated carbon. The filtered

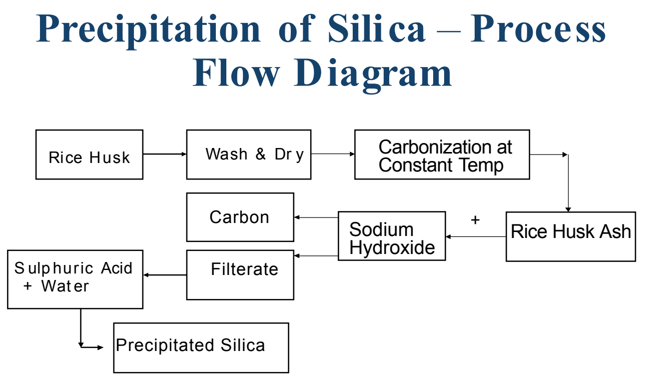

Figure 2: Precipitation of Silica – Process flow diagram.

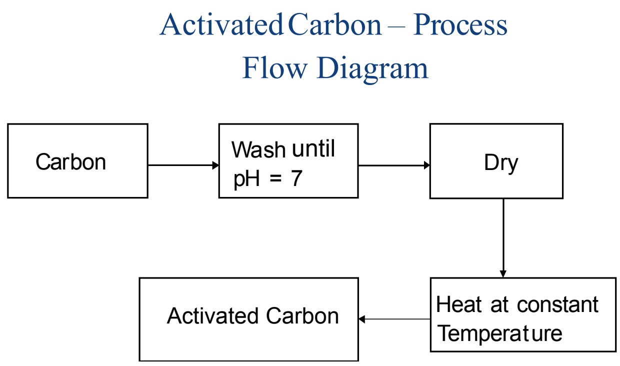

Figure 3: Activated carbon-Process flow diagram.

solution containing the activating agent is titrated with acid to precipitate silica powder.

The temperature at which rice husk is carbonized is very important since the surface area characteristic of ash depends on the temperature of formation of ash. The ash is obtained as follows rice husk is cleaned from dirt by sieving it with 20 mesh sieve. The rice husk ash so obtained produces a lot of smoke if kept directly in the furnace. In order to avoid this, the husk is first charred to black mass on a Bunsen burner. The mass is then heated & oxidized in an electric furnace at controlled temperature to obtain the ash. The ash obtained is finely ground & sieved through 150 mesh sieve. The fraction passing through this sieve is then used for experimental run. The charred husk fraction is further carbonized at 550°C, 600°C, 650°C, and 700°C for 3 hours.

The activation of the rice husk ash is done either by thermal or chemical activation. In thermal activation (eg: coal, lignite and coconut shell), raw material is carbonized to drive off the gases. A char of high carbon content is obtained. This char is treated with air at 450 to 500°C or with steam or carbon dioxide (CO_2) in the temperature range of 880 to 1100°C. In activation step the oxidizing gas reacts with carbon atoms in the interior of char to form gaseous reactant products and develop the channels and pores that contributes to high internal surface area.

In chemical activation, dehydrating agents such as zinc chloride is used to chemically decompose cellulose of the raw material (figure 3). The raw material is mixed with the chemical agent and soaked for known amount of time. It is then dried and carbonized in the absence of air. Obtained char is treated with alkali to form sodium silicate. Sodium silicate is treated with mineral acid and water to get precipitated silica. Filtrate is washed until pH is neutral.

Following are the sieving mesh sizes, composition of rice husk and the parameter employed in the procedure:

Rice Husk gradation (sieving)

Rice Husk is sieved using 20-200 mesh size sieves. The major fraction, which usually consists of 20 mesh size, is used for the experiment.

Composition of rice husk ash

SiO_2=80-90 %

Alumina=1-2.5%

Ferric oxide=0.5%

Trace elements=remaining

Carbonization of rice husk

The rice husk obtained above in step 1 is first carbonized for 2 hours at and above 700°C. If the temperature is below 500°C, the rice husk is not carbonized completely.

Effect of temperature

Below 500°C incomplete carbonization, at and above 700°C, nano-silica obtained.

Effect of time length

Minimum time duration is 2 hours

Treating with activating agent

The carbonized rice husk is then treated with activating agents such as listed below at 0.2 N for a minimum time of 2 hours at 60°C. This makes the silica soluble in the activating agent and subjects the rice husk to have more surface area during the carbonization step mentioned below.

Types of activating agents used

Sodium hydroxide, phosphoric acid and Zinc chloride

a) **Concentration:** Sodium Hydroxide - 0.2 N

b) **Length of time:** Minimum 2 hours

c) **Temperature:**

Rice Husk on being treated with activating agent - 60°C

Temperature of carbonization – 500°C, 600°C, 650°C, 700°C

Concentration of sodium hydroxide – 5%, 10%, 15%, 20% (w/v)

Temperature of activation – 800°C, 850°C, 900°C

Precipitated silica

The silica is precipitated by adding concentrated sulfuric acid to the filtered solution of Step 3 at room temperature. This yields very pure silica. The types of silica obtained depend on the carbonization temperature and the any pre-treatment before carbonization. The yield is between 95-98% of the amount of silica present in the initial rice husk.

A. Acid used: Sulfuric acid

B. Concentration used: Concentrated

C. Temperature: Room temperature

D. Types of silica obtained: Depends on carbonization temperature

E. Purity: Very pure silica can be obtained (Rice husk is burnt under controlled conditions gives amorphous silica of high purity)

F. Size: Nano-silica, powder, different grades of silica (small particle size)

G. Yield: 95-98 %

H. Surface area:

1. Nature: Amorphous powder
2. Appearance: White fluffy powder
3. Purity: > 98 %
4. Surface Area: 150 - 200 m2/gm
5. Bulk density: 120 - 200 g/liter
6. Loss on Ignition: 3.0 - 6.0 %
7. pH of 5 % slurry: 6.3 + 0.5
8. Heat loss: 4.0 - 7.0 % the properties like surface area, pH, Tap density can be tailor made for the requirement.

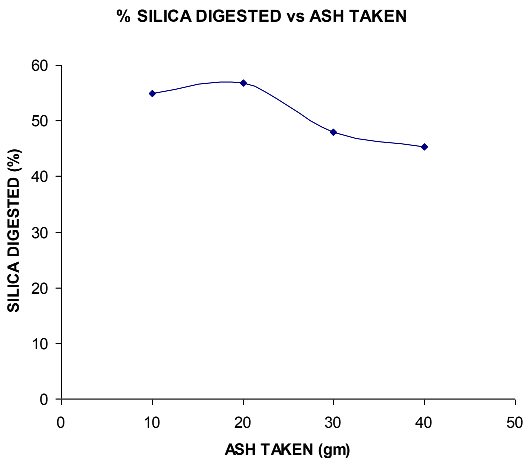

Figure 4: Digestion of rice husk ash for 1 hour in 0.5N NaOH solution.

Activated carbon

The activated rice husk which has been treated with sodium hydroxide in Step 2 and 3 is further carbonized at temperatures of 700 to 900 °C for 2 hours to obtain activated carbon of wastewater grade.

A. Temp effect: 700-900 °C

B. Duration/Time: Minimum 2 hours

C. Types/grades of carbon obtained: Mechanical grade and electronic grade

D. Surface area: High surface area

Application

Activated carbon obtained by the above procedure can be used in wastewater treatment as an adsorbent to remove organics. Elemental silica is used as a constituent of building material. In amorphous form it is used as desiccant, adsorbent, refining agent filter, catalyst compound, etc. In vitreous form, it is used in the manufacture of optical elements and glass wires. It is also used as a basic material for glass, ceramic and refractory industries. Other applications include in the manufacture of soluble silicate, silicon and its alloy, silicon carbide, silicon based chemicals and the silicones. It also finds uses in reinforcing filter, in natural and synthetic rubber and is used in adhesives to enhance bond strength. Other uses include control of porosity in battery separators, for controlling oil spillage, used in brake linings as anti-caking agent. It has also found use in cosmetics as an absorbent and thickening agent, in inks as thickening and matting agent, in tooth paste as an abrasive additive powder and in paper as filler for opacity and brightness, polishes as a mild abrasive. It can be used as a catalyst support for fine chemical synthesis.

Results and Discussion

Although there are different procedures to obtain silica from rice husk, the most important step is to first treat the rice husk with acid treatment to remove any impurities such as metals that are present in the rice husk. The acid treatment also gives a high surface area for the silica when it is precipitated. The next important aspect of producing nano-silica powder depends on the carbonization temperature and duration of carbonization. Different grades of silica with different yields can be obtained based on the carbonization temperature and duration of carbonization. The activating agent, temperature and time of heating the pre-acid washed rice husk, concentration of activating agent, all influence the yield, surface area of the precipitated nano-silica.

Sl no	Ash taken (gm)	NaOH taken (gm)	Vol of NaOH (ml)	Vol of silicate (ml)	Specific gravity	% silica in solution	Undigested ash (gm)	% silica digested
1	10	10	500	450	1.026	1.07	2.2	54.89
2	20	10	500	415	1.033	2.2	4.7	56.82
3	30	10	500	380	1.036	2.78	11.2	48
4	40	10	500	310	1.038	3.49	15.2	45.28

Table 2: Digestion of rice husk ash for 1 hour in 0.5n NaOH solution.

Sl no.	Ash taken (gm)	NaOH taken (gm)	Vol of NaOH (ml)	Vol of silicate (ml)	Specific gravity	% silica in solution	Undigested ash (gm)	% silica digested
1	10	20	500	490	1.04	1.23	1.7	63.96
2	20	20	500	450	1.052	2.33	3.6	61.28
3	30	20	500	375	1.06	3.58	6	63.24
4	40	20	500	340	1.07	5.11	8.5	68.35
5	50	20	500	310	1.077	5.66	12.6	60.96

Table 3: Digestion of rice husk ash for 1 hour in 1.0N NaOH solution.

The activated carbon grades obtained depends on the temperature and duration of carbonization after treatment with sodium hydroxide (Table 2, 3; Figures 4, 5). Also, the concentration of sodium hydroxide, time and temperature of heating produces different grades of activated carbon.

After carrying out a series of experiments and analysis it can be seen that the most optimized condition for the digestion of rice husk in sodium hydroxide solution are as follows:

- Concentration of NaOH solution: 1.5N (Tables 4, 5, Figure 6)
- Weight of ash: 120 gm/lit
- Temperature: 95 degree centigrade
- Time of digestion: 1 hour

To study the effect of temperature and time on the digestion of rice husk ash in sodium hydroxide solution (Table 6), further studies were carried out (Figure 7, 8).

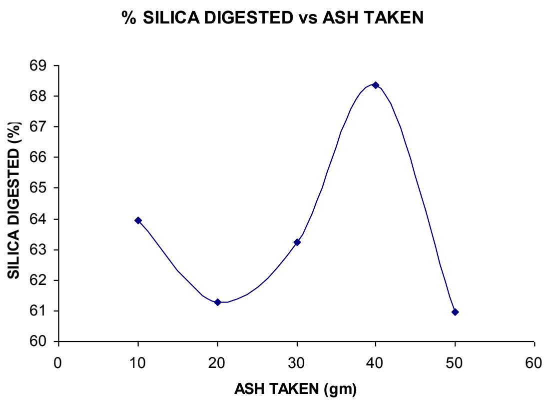

Figure 5: Digestion of rice husk ash for 1 hour in 1.0N NaOH solution.

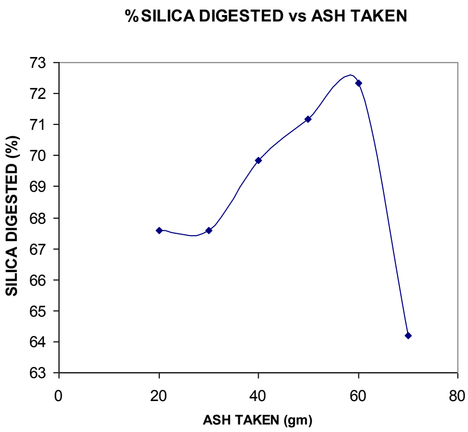

Figure 6: Digestion of rice husk ash for 1 hour in 1.5N NaOH solution.

Conclusion

- The procedure adopted in this project to prepare precipitated silica from rice husk ash was successful and practically very sound. This innovative idea can be used out in future in the precipitated silica industry. The manufacture of silica from rice husk ash works out to be very cheap and cost effective as the main raw material, the rice husk ash can be obtained at low cost. Activated carbon obtained as the byproduct has a good market value.
- A general procedure is outlined for silica precipitation and activated carbon from rice husk. The most important aspect is nano-silica powder is obtained (Table 7, Figure 9). The effect of different parameters like temperature of carbonization, temperature of mixture feed into the reactor, production of ash & conversion of silica has been reviewed. Precipitation of Silica carried out at 500°C, 600°C, 650°C, 700°C using 5%, 10%, 15% and 20% Sodium Hydroxide–Analysis awaiting
- Silica present in Rice Husk=30%
- Activated Carbon obtained at 850°C and 900°C–Analysis awaiting
- Precipitated silica and activated carbon was obtained from rice husk using the process outlined
- Silica in rice husk was 30%
- Analysis of yields of silica and activated carbon under progress

Sl.No.	Ash Taken (Gm)	NaOH Taken (Gm)	Vol of NaOH (Ml)	Vol of Silicate (Ml)	Specific Gravity	% Silica in Solution	Undigested Ash (Gm)	%Silica Silica Digested
1	20	30	500	425	1.077	2.44	3.8	67.58
2	30	30	500	400	1.08	3.76	5.3	67.59
3	40	30	500	355	1.09	4.97	9.4	69.84
4	50	30	500	340	1.101	6.152	9.5	71.17
5	60	30	500	295	1.105	7.64	14.1	72.32
6	70	30	500	270	1.113	7.52	16.7	64.19

Table 4: Digestion of rice husk ash for 1 hour in 1.5N NaOH solution.

Sl no	Ash taken (gm)	Naoh taken (gm)	Vol of naoh (ml)	Vol of silicate (ml)	Specific gravity	% silica in solution	Undigested ash (gm)	% silica digested
1	40	40	500	375	1.114	4.75	6.8	63.46
2	50	40	500	340	1,124	6.33	9.7	66.14
3	60	40	500	305	1.13	7.12	12.4	67.089
4	70	40	500	345	1.132	8.49	15.3	68.66
5	80	40	500	275	1.14	8.907	20.1	71.19
6	90	40	500	260	1.146	9.829	22.5	62.57

Table 5: Digestion of rice husk ash for 1 hour in 2N NaOH solution.

Scope for future work

Effect of three parameters (acid treatment, temperature and heating rate) on the formation of black particles in rice husk silica ash was studied. However, at low temperature (400°C) the oxidation of carbon in the acid treated rice husk is sluggish. Several efforts are being made to discover a suitable technology capable of producing large quantities of solar grade silicon at a cost less than $10/kg. An industrial by-product can be converted to a high value added product by using a simple inexpensive method. A powder consisted of 92% of silica was prepared after calcinations of RHA at 700°C for 5 hour. Silica from rice husk can be prepared under controlled temperature without any chemical treatment, the bulk of investigations indicate that producing silica ash composites directly or into related silica filler composites, suggest that improvement can be achieved in the composite performance by modifying the physical and chemical nature of the filler. The extent of improvement, however, is greatly influenced by the ability to control the many relevant characteristics of the filler. A systematic study from a thermodynamic point of view is expected to provide the user with a better understanding of the parameters that are significant & relevant. The husks on leaching with acetic and oxalic acids of different concentrations followed by thermal treatment resulted in products with improved properties like purity, reactivity, brightness, surface area and pore volume. The leaching with mineral acids like HCl and HNO_3 of different concentrations followed by thermal treatment and measurement of properties showed that the acid treatment is almost equally effective in improving the silica properties.

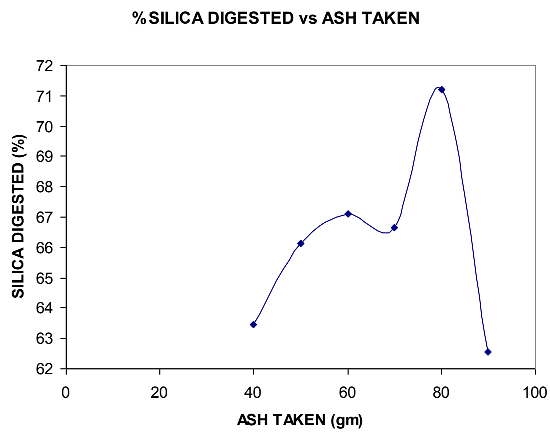

Figure 7: Digestion of rice husk ash for 1 hour in 2N NaOH solution.

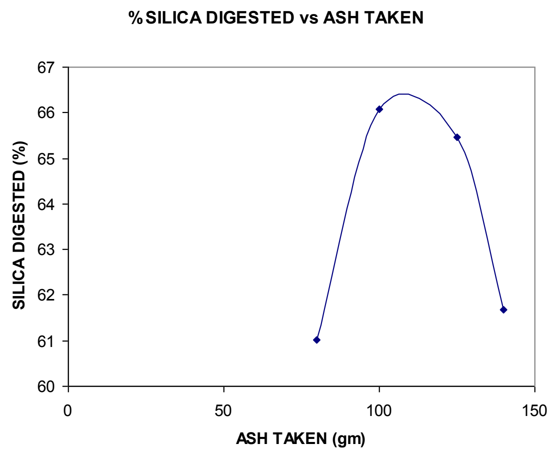

Figure 8: Digestion of rice husk ash for 1 hour in 2.5N NaOH solution.

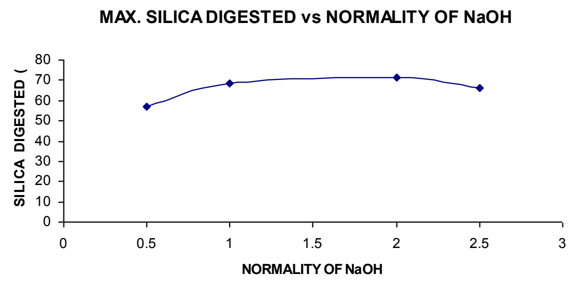

Figure 9: Comparisons of the peak values of %silica digested.

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Sl no	Ash taken (gm)	Naoh taken (gm)	Vol of naoh (ml)	Vol of silicate(ml)	Specific gravity	% silica in solution	Undigested ash (gm)	% silica digested
1	80	50	500	290	1.162	8.402	14.7	61.02
2	100	50	500	250	1.184	11.16	20	66.08
3	125	50	500	185	1.2	13.64	32.3	65.472
4	140	50	500	110	1.202	14.37	57.7	61.68

Table 6: Digestion of rice husk ash for 1 hour in 2.5N NaOH solution.

Sl No.	Normality of NaOH	Ash taken	Silica digested
1	0.5	20	56.82
2	1	40	68.35
3	1.5	60	70.35
4	2	80	71.19
5	2.5	100	66.08

Table 7: Comparisons of the peak values of %silica digested.

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