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Extraction of Silica from Rice Husk Ash

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Abstract—*Rice husk is a form of waste from the rice milling industries and is produced in abundance in and around the country. India alone produces 12 million tons of rice husks every year. Worldwide annual husk output is estimated is 80 million tons. Rice husk is considered as a form of waste from rice milling processes and are often left to rot in field or burnt in open. To some extent rice husk has been utilized as fuel for cooking and parboiling of paddy rice in some developing country.*

But rice husk contains silica in range of 80-90 % wt. The silica in rice husk exists in the hydrated amorphous form like silica gel. The amorphous nature of silica in rice husk makes it extractable at a lower temperature range. Energy is also produced in the process which could be recovered in the form of heat or electricity.

Silica, at present, is manufactured by sand fusion of high purity silica sand as the feedstock. This technique is highly energy intensive as it requires the reactants to be heated to temperature as high as 1400°C .In the conventional process, sodium silicate is obtained from silica sand. Sodium Sulphate is a waste that is generated and this liquid effluent requires elaborate treatment before disposal. Another preparation method is the sol- gel process but it involves high raw materials cost. All these processes involve some or the other disadvantage which increases the cost of production.

This paper aims at showing how production of silica can be made economical and since the process uses rice husk which is an agricultural waste, the process is also eco-friendly.

1. INTRODUCTION

Oxygen and silicon are the two most common elements in the earth's crust, together constituting an estimated 74.32 weight % and 83.77 atom % of crustal rocks. Thus it is not surprising that SiO_2 or silica is the most abundant oxide on the earth's surface. Silicon oxide is formed when silicon is exposed to oxygen (or air). A very shallow layer (approximately 1nm or 10 Å) of so-called native oxide is formed on the surface when silicon is exposed to air under ambient conditions. Higher temperatures and alternative environments are used to grow well controlled layers of silicon dioxide on silicon, for example at temperatures between 600^{o}C and 1200^{o}C, using so-called dry or wet oxidation with O_2 and H_2O, respectively. The depth of the layer of silicon replaced by the dioxide is 44% of the depth of the silicon dioxide layer produced.

It has been found that quartz alone comprises 12% of the crust by volume, ranking behind the mineral groups tthat include fledspar (59.5%) and amphibole/pyroxene (16.8%). Consequently, research into the silica system is motivated foremost by the prevalence of silica in man's immediate surroundings. The ubiquity of silica in ingenious, metamorphic, and sedimentary rocks has led earth scientists to seek its uses as an indicator of large scale geological processes, ranging from mountain-building to meteorite impacts. In industry, quartz has long played a role as an inexpensive and relatively inert constituent of concrete aggregates, and modern electronics technology still relies on quartz oscillators. Silica phases also have played a prominent role in our understanding of the solid state.

Silica is a common additive in food production, it is a primary component of diatomaceous earth, and it is also a primary element of rice husk ash. The drawbacks of the common commercial methods to produce silica have led to the development of this process of Silica production from rice husk ash.

2. CONVENTIONAL PROCESS OF MANUFACTURE OF SILICA

Silica is manufactured by sand fusion of high purity silica sand as the feedstock. This technique is highly energy intensive as it requires the reactants to be heated to temperature as high as 1400°C.

In the conventional process, sodium silicate is obtained from silica sand. The State-of-Art Technology is used to manufacture Sodium silicate by fusing pure silica sand with Soda ash in Rotary furnace at 1300°C, various SiO2/ Na2O ratios are produced. When the mould cools down a clear glass of sodium silicate is obtained. This Sodium silicate undergoes acid precipitation to produce precipitated silica. Sodium Sulphate is a waste that is generated and this liquid effluent requires elaborate treatment to meet emission Standards. The process requires effluent treatment for sodium sulphate. This call for additional financial implications and any carelessness in treating the effluent would damage the environment.

Another preparation method is the sol- gel process but it involves high raw materials cost. Such preparation methods results in extremely high production cost, which is subsequently reflected in its high market price.

3. RICE HUSK AS AN ALTERNATIVE

Rice husk is a form of waste from the rice milling industries and is produced in abundance in around the country. All rice-producing countries have abundant quantity of rice husk. India alone produces 12 million tons of rice husks every year. Thus worldwide annual husk output is estimated is 80 million tons. So it is an eco-friendly process as it minimizes the paddy husk ash waste.

Rice husk is considered as a form of waste form rice milling processes and are often left to rot in field or burnt in open. To some extent ,rice husk has been utilized as fuel for cooking and parboiling of paddy rice in some developing country, but neither fully or nor efficiently utilized as cheap and abundant source of silica. Rice husk contains silica in range of 20-25wt%. The silica in rice husk exists in the hydrated amorphous form like silica gel. The amorphous nature of silica in rice husk makes it extractable at a lower temperature range. Thermal degradation and pyrolysis of rice husk followed by the combustion of the char results in a highly porous and amorphous silica with a varying percentage of unburnt carbon. Combusted at moderate temperature, the white ash obtained from rice husk contains approximately 92-97% amorphous silica. And hence thermal treatment of rice husk to produce amorphous silica is viewed as a more economical process having the potential to replace the conventional high temperature processes. This is because thermal treatment of rice husk actually produces energy instead of consuming energy. The energy produced could be recovered in the form of heat or electricity.

Table 1: Composition of Rice Husk Ash

Element	Mass fraction %
Silica (SiO2)	80-90
Alumina	1-2.5
Ferric Oxide	0.5
Titanium Oxide	Nil
Calcium Oxide	1-2
Magnesium Oxide	0.5-2
Sodium Oxide	0.2-0.5
Potash	0.2
Loss on ignition	10-20

Following are the advantages of the silica obtained from rice husk ash precipitation process.

1. Eco-friendly process as it minimizes the paddy husk ash waste. The current uses of rice husk in boilers for heat and in combustion process for generation of electricity. Both uses will be left with 20%of the quantity used in the form of ash. As such this ash is a waste and disposal is a challenge. This ash is raw material for producing silica using this process.
2. The process suggests closed loop operation hence no unwanted or hazardous chemical is derived as a bye product unlike the conventional processes.
3. Source of silica is replenish-able. All rice producing countries have abundant quantity of rice husk. India alone produces about 12 million tons of rice husk every year.

4. PRECIPITATED SILICA USING RICE HUSK ASH

Rice Husk ash has a silica content of around 80-90% most of which is in amorphous nature, depending on the temperature of combustion. This silica can be extracted economically by the proposed process, which meets the requirements of the various industries. This novel process consists of four processes namely;

Digestion
Precipitation
Regeneration
Calcination
In the process the chemicals used are recycled thus eliminating expensive effluent treatment plant and also cutting down the plant operation cost.

4.1 Digestion

$$2NaOH + ash \rightarrow Na_2SiO_3 + H_2O$$

4.2 Precipitation

$Na_2SiO_3 + 2CO_2 \rightarrow 2Na_2CO_3 + 2SiO_2$

4.3 Regeneration

$$Ca(OH)_3 + Na_2CO_3 \rightarrow 2NaOH + CaCO_3$$

4.4 Calcination and Slaking

$CaCO_3 \rightarrow CaO + CO_2$

$Ca(OH)_3 \rightarrow CaO + H_2O$

$$CaO + H_2O \rightarrow Ca(OH)_3$$

4.1 Digestion

Digestion refers to extraction of the insoluble silica present in the ash to soluble salt in the form of sodium silicate. The required quantity of sodium hydroxide (ratio 1:1 with respect to dry ash) is dissolved in water and the ash is added after the temperature of the caustic reaches 95°C. Digestion temperature of around 95°C gives optimum yield.

The digestion is carried out at this temperature for a period of 1 hour, which is the optimum time established by the experimental results as decrease in time results in decrease in the silica extracted and further increase in time of digestion hardly increases the yield obtained. Hence the optimum time for extraction is around 1 hour at around 95°C. Lower temperature extractions yield lower silica and thus increasing the cost of extraction in the form of heat supplied.

4.2 Precipitation

Precipitation is the process in which the soluble sodium silicate reacts with carbon dioxide to form silicon dioxide. This is the crucial step to obtain the precipitated silica of required specifications, by varying the parameters. It was observed that the silica produced during the process created a blockage of sparger holes when the carbon dioxide pressure was less than 1.75 kg/cm^2(gauge). During this step the various parameters like temperature, flow of carbon dioxide and silica concentration in the sodium silicate solution play a vital role in obtaining silica of different types. Precipitation was carried out at various temperatures and it was observed that lower temperatures produces gel like silica with high density. It was also observed that the rate of carbonation affected the density of the silica produced, the density increasing with increase in flow rate.

The two main requirements or controlling factors for the silica produced are:

1. Surface Area

Surface area depends upon the quantity of mixing that takes place inside the precipitator which varies with the agitator use. The surface area is low (<80 m^2/g) when the agitator is paddle type due to insufficient mixing. The surface area increases (>150m^2/g) when propeller type agitator is used which creates a more vigorous mixing compared to paddle type.

2. Tap Density

Tap density of the material is the density of the powder when a fixed quantity of the silica powder is tapped in a standard closed measuring cylinder till the volume of the powder remains constant and does not decrease with further tapping. The tap density of the material is dependent on the ratio of initial caustic to carbon dioxide flow rate. It is possible to control the tap density in the two ranges mentioned below, by controlling the carbonation rate.

i. Around 100 – 150 g/lit. (Required by most applications)
ii. Around 200 – 250 g/lit. (Required by tire industries)

4.3 Regeneration

Regeneration of the solution is the conversion of sodium carbonate to sodium hydroxide by the use of calcium hydroxide.

$$Ca(OH)_3 + Na_2CO_3 \rightarrow 2NaOH + CaCO_3$$

The regenerated sodium hydroxide is used for the digestion of fresh ash. Calcium hydroxide can be either purchased from the market and the resulting calcium carbonate can be sold at the market rate or the calcium carbonate can be heated at around 850°C for converting it to calcium oxide which when comes in contact with water becomes calcium hydroxide. Resulting carbon dioxide can be used for the precipitation step.

4.4 Calcination and Slaking

$$CaCO_3 \rightarrow CaO + CO_2$$

Calcination is a thermal treatment process in presence of air in which decomposition of calcium carbonate (lime) and carbon dioxide. The reaction takes place at around 850°C in a reactor or a furnace sometimes also referred to as kilns or calciners. Calcium oxide is made to react with water in a slaker to give calcium hydroxide which is sent to the regenerator so the sodium hydroxide and calcium carbonate is produced when sodium carbonate reacts with it. The carbon dioxide gas produced is sent to the precipitation step.

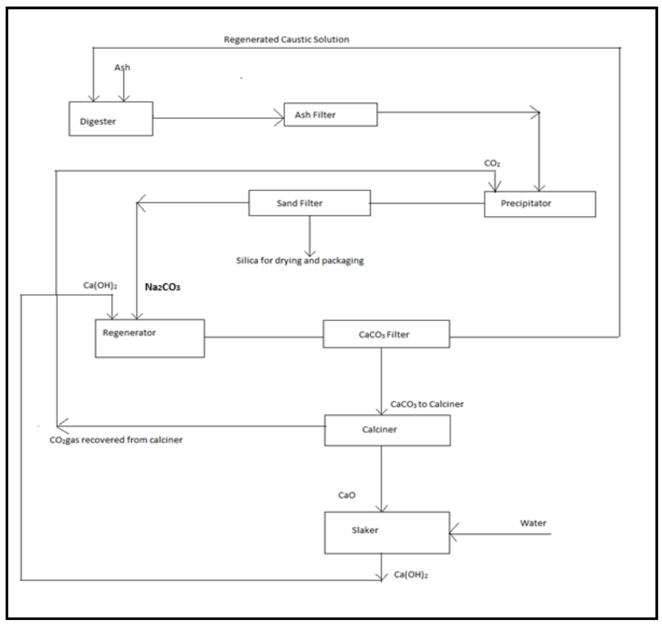

Fig. 1: The Silica Precipitation Process

Table 2: Typical Properties of Silica Precipitated

Nature	Amorphous powder
Appearance	White fluffy Powder
Purity	> 98%
Surface area	50 – 300 m2/g
Bulk Density	120 – 400 g/lit.
Loss on Ignition	3.0 – 6.0 %
pH of 5% slurry	6.3 + 0.5
Heat loss	4.0 – 7.0 %

5. CONCLUSION

Hence, Silica Precipitation from rice husk is not only an alternative process to the conventional energy intensive processes for silica production but it can also help produce electricity from the energy generated in the process. This electricity can be used to run the process or can be sent to the grid. Also, the process reduces significantly the volume of waste ash generated from rice husk and adds value to the waste.

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