

PRECIPITATED SILICA FROM RICE HUSK ASH BY IPSIT PROCESS

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Abstract:

Rice husk is an agricultural residue abundantly available in rice producing countries like India, China, Brazil, and South East Asia. Rice husk is mainly used as a fuel in industries in boilers for process energy requirements and for power generation. Rice husk is a fuel having high ash content, varying from 20 – 25 % of rice husk, the major constituent (>90 of ash) being silica. This ash has some limited use as a heat insulator in steel rolling mills, in brick making and in cement manufacturing, but largely remains unused. Due to the low bulk density of the ash (around 200 kg/m³) its disposal in large volumes creates environmental problems.

The patented Indian Institute of Science Precipitated Silica Technology (IPSIT) developed at CGPL, IISc, Bangalore is a novel method of extracting precipitated silica in commercially viable way from rice husk ash. Precipitated silica finds application in many of the industries like from tyre industry, food industry, paint industry, cosmetics etc and is used as reinforcing agent in rubber, thickening agent in paints, anti caking agent in food industry. These applications require silica of different grades which are characterized by surface area, tapped density, oil adsorption capacity, silica content etc. The IPSIT process is able to meet the different industry demands with respect to the quality of the silica.

The investment cost for 5 TPD precipitated silica production by this process is around 1.6 million USD and the production cost is roughly 0.6 US. The selling cost is around 1 USD.

Key words: rice husk ash, precipitated silica

1. Introduction

Rice husk is an agricultural residue abundantly available in rice producing countries. The annual rice husk production in India amounts is generally approximately 12 million tons. Rice husk is generally not recommended as cattle feed since its cellulose and other sugar contents are low. Furfural and rice bran oil are extracted from rice husk. Industries use rice husk as fuel in boilers and for power generation. Among the different types of biomass used for power generation, rice husk has a high ash content varying from 18 – 20 %, silica is the major constituent of rice husk ash. With such large silica content in the ash it becomes economical to extract silica from the ash, which has wide market and also takes care of ash disposal. Silica is used in different industries and products like rubber industry as a reinforcing agent, in tooth pastes as a cleansing agent, as an anti-caking agent in salts, in cosmetics etc.

Table I gives typical composition of rice husk ash.

Table I: Typical composition of Rice husk ash on dry basis.

Element	Mass Fraction, %
Silica (SiO ₂)	80 – 90
Alumina	1 – 2.5
Ferric oxide	0.5
Titanium dioxide	Nil
Calcium oxide	1 – 2
Magnesium oxide	0.5 – 2.0
Sodium oxide	0.2 – 0.5
Potash	0.2
Loss on Ignition	10 – 20

From the table it is clear that silica is the major constituent of the rice husk ash. Experiments have been carried out successfully under lab scale and pilot scale to extract the silica from the rice husk ash. This not only provides value addition but also solves the problem of large amount of ash disposal.

2 Process

The silica precipitation technology developed at CGPL, Indian Institute of Science, Bangalore is a novel method for silica precipitation where the chemicals used is regenerated making it possible to recycle the chemicals. Successful studies for extraction of silica on laboratory scale and pilot scale which meet the industrial requirements have been carried out. Also studies are carried out for suitable application of the undigested ash obtained after extraction, in water treatment plants with or without further improving the activated carbon content of the ash. The yield of silica in the process is about 70 %, which amounts to conversion of around 80 – 90 % on silica in the ash.

Figure 1 shows the flow chart of the process. The major steps involved are digestion, ash filtration, precipitation, filtration of silica, and regeneration of the spent solution

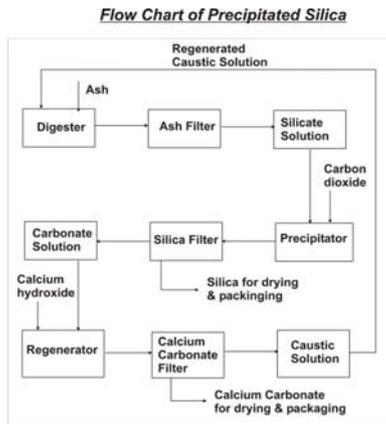


Figure 1 Flow chart showing the process of precipitated silica from rice husk ash

Brief descriptions of the various steps involved in the processing of rice husk ash to precipitated silica are given in the following sections.

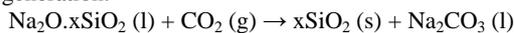
2.1 Digestion

This involves the digestion of the rice husk ash with caustic at specific conditions. In this process the silica in the ash is extracted with caustic soda to form soluble sodium silicate solution. After the completion of the digestion the solution is filtered for the residual undigested ash present in the solution. The clear filtrate is taken for precipitation.



2.2 Precipitation

This step involves precipitation of silica from the sodium silicate solution. Sodium silicate solution from the digestion step is taken in the precipitator. Carbon dioxide at a specific flow rate is passed through the silicate solution at design conditions. Continuous stirring is employed during the operation. Silica is precipitated during the reaction. The precipitated silica is filtered, washed with water to remove the soluble salts and dried. The filtrate containing sodium carbonate is taken for regeneration.



2.3 Regeneration

Regeneration is the step where calcium compound reacts with the sodium carbonate to form calcium carbonate and sodium hydroxide. The filtrate from the precipitation stage is taken to a regenerator where the sodium carbonate present in the solution is converted to sodium hydroxide using calcium hydroxide. The resulting solution is filtered to remove the solid calcium carbonate and the aqueous sodium hydroxide is used for digestion again. The calcium carbonate is washed with water and dried. The dried calcium carbonate can be either calcined to get calcium oxide, which is reused, for regeneration or the calcium carbonate is sold and fresh calcium hydroxide is used for regeneration which gives an option of one more value addition.

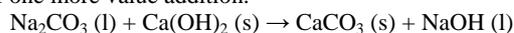


Figure 2 shows a photograph of the pilot plant set up in the laboratory for this process.



Figure 2: The photograph of the pilot plant for precipitated silica.

3 Typical Properties of precipitated silica obtained by IPSIT process

1. Nature: Amorphous powder
2. Appearance: White fluffy powder
3. Purity: > 98 %
4. Surface Area: 50 - 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 %

A scanning electron microscope picture of the silica is shown in Fig. 3. The amorphous nature of silica can be seen from the photograph. It can also be seen that the primary particle size is in the range of nanometers. The properties like surface area, pH, tap density can be tailor made for the specific industrial requirement.

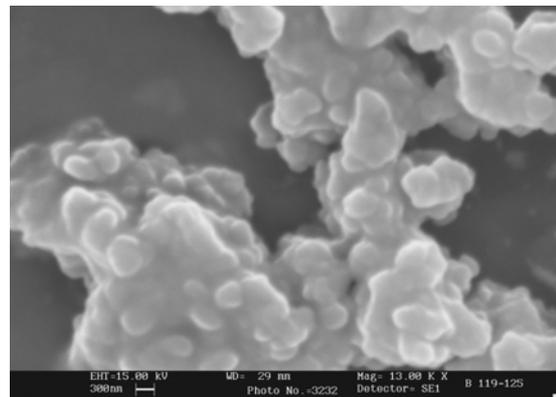


Figure 3 The SEM picture of the silica obtained with the process

4 Experimental Work & Results

The experimental work involved studying different parameters that affect the quantity and quality of silica produced. The quality of silica produced mainly depends on the precipitation conditions.

4.1 Digestion

Studies were carried out by varying the process parameters such as temperature, concentration of NaOH solution used for digestion.

- Quantity of silica extracted from ash increases with temperature

- Silica yield increased marginally with increase in concentration of caustic (at same caustic to ash ratio).
- It was observed that as the Caustic to Ash ratio increases the yield increases upto a ratio of 1:3 after which the yield decreases. The optimum digestion of around 60 – 70 % yield can be achieved by having a caustic to ash ratio varying from 1:1 to 1:3. Decrease in the ratio results in higher alkalinity of the silicate solution and consequently higher consumption of CO₂ for precipitation.
 - A environmentally benign process
 - Lower cost of production
- Process temperatures around 100 °C compared to conventional process where the temperatures can go upto 1500 °C.
- Quality of silica obtained similar to the available silica in the market.

The economics of silica precipitation using 1:4 ratio of Caustic to Ash have to be looked into as the yield at this ratio is lower compared to other ratios where as the requirement of the carbon dioxide for precipitation decreases due to the decrease in the alkalinity for the same quantity of silica extracted when compared to other ratios.

4.2 Precipitation

The quality of silica produced is determined in the precipitation stage. The surface area, the bulk density, etc are dependent on several process parameters such as concentration of the silicate solution taken for precipitation, temperature at which the precipitation is carried out, carbon dioxide flow rate and the duration of precipitation. Studies were conducted with lab scale plant to understand the effects of different parameters mentioned above on the quality of silica.

- The surface area silica is affected by the precipitation conditions like temperature, agitation etc with different initial concentrations of silica. These parameters also affect the bulk density of silica produced. Therefore, for obtaining a given set of these properties the conditions of precipitation should be carefully set.
- The bulk density of silica produced is a function of ratio of initial silica to CO₂ flow rate.
- CO₂ consumption decreases with increase in the speed of agitation.
- The number of silanol groups is also a parameter for bonding with rubber when used as filler in tires. This parameter can be tuned by the temperature of drying.
- Required grade of silica could be obtained by prior knowledge of effect of the individual parameters on the grade of silica (i.e. Surface area, bulk density etc).

5 Salient Features of the technology

- Silica of industrial requirement or grade can be manufactured from rice husk ash using IPSIT process.
- Rice husk ash which is a environmental burden is used as a resource.
- The chemicals used are regenerated making it

6 Estimated Costing for Silica by IPSIT Process for Indian conditions

The cost of production includes the consumables, energy (both thermal and electrical), and other operational costs, in addition to the financial costs such as depreciation and interest. Table II gives an estimate of these costs and the possible net profit from this plant.

Table II The estimate cost of production and possible revenue from the plant

Plant Capacity & Investment cost		24 MT/day & 5.9 million USD	4.8 MT/day & 1.6 million USD
Sr. No	Particulars	Cost, USD/kg of Silica	Cost, USD/kg of Silica
1	Raw material	0.22	0.22
2	Cost of manpower	0.05	0.06
3	Maintenance cost	0.01	0.01
4	Power cost @ USD 0.1/kWh	0.1	0.11
5	Packing & handling cost	0.05	0.05
6	Thermal energy cost	0.14	0.14
7	Total production cost	0.57	0.59
8	Revenue generated		
	Cost of silica/kg	0.95	0.95
	Cost of CaCO ₃ /kg	0.20	0.20
	Total	1.15	1.15
9	Depreciation (10 years)	0.07	0.09
10	Interest @ 10 %	0.07	0.09
11	Net Profit	0.44	0.38

7 Concluding Remarks

A process for generation of precipitated silica has been developed from rice husk ash, which can be used for commercial exploitation. This not only brings additional income for the power plant operators, but also solves the disposal problem of the ash. The properties required for the different applications of silica can be obtained by tuning the operating parameters of the process.