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Extraction of Silica from Rice Husk

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ABSTRACT:- Rice is one of the major crops grown through the world. Once the paddy is separated from the rice grain, the kernel (hull) is removed from rest of the grain. This constitutes about, one third of the total mass of grains, commonly termed as 'Rice Husk' or 'Rice Hull'. Rice husk is an agricultural residue abundantly available in rice producing countries. The annual rice husk produced in India amounts generally approximately 12 million tones. 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 gasification, rice husk has a high ash varying from 18-20%. Silica is the major constituent of rice husk ash varying from 85-95%. With such a large ash content silica content in the rice husk it becomes economical to extract silica from the ash, which has wide market and also takes care of the ash disposal.

In this project an attempt is made to introduce a simple process to manufacture precipitated silica from this waste rice husk. It solves the disposal problem of husk as well as valuable product is manufactured from it. Experimental data shows better performance as well as easy industrial implementation of the process.

Keywords:- Extraction, Silica, Concentrated H_2S0_4, Rice husk, Filter press.

I. INTRODUCTION

Silica (SiO_2) is one of the valuable inorganic multipurpose chemical compounds. It can exist in gel, crystalline and amorphous forms. It is the most abandon material on the earth's crust. However, manufacture of pure silica is energy intensive. A variety of industrial process, involving conventional raw materials require high furnace temperatures (more than 700 degree C). In our Project, a simple chemical process is described which uses a non-conventional raw material rice husk ash for extraction of silica. Rice Husk ash is one of the most silica rich raw materials containing about 90-98% silica (after complete combustion) among the family of other agro-wastes. Rice husk is a popular boiler fuel and the ash generated usually creates disposal problems. The chemical process discussed not only provides a solution for waste disposal but also recovers a valuable silica product, together with certain useful associate recoveries. The other associate recovery is sodium sulphate. Effluent wash water obtained after washing precipitated silica (wet impure silica) contains sodium sulphate. By evaporation of water in multiple effect evaporators, followed by crystallization, filtration and drying, crystals of sodium sulphate are obtained. The residue ash in sodium silica production can be utilized for making good quality bricks. Retained sodium silicate in residue ash acts as a binder and with incorporation of suitable ingredients high quality bricks can be manufactured.From rough estimation the production of paddy in the country about twenty five million tons of rice husk is obtained from rice mills. This is usually burnt resulting in mounts of ash in and around the mills, causing major health problem and pollution. Using rice husk as the raw material two million tons of pure high grade silica can be produced to meet the high demand of various industries that are using it. The main states with large number of rice mills are Andhra Pradesh, Tamil Nadu, Karnataka, and Kerala etc.

The conventional process of manufacturing silica is the reaction of sand with soda ash at about 1500º C. In our project, a new method of manufacturing silica by reacting rice husk ash with caustic soda is developed.

II. MAIN ADVANTAGE

In Advantage of using rice husk as raw material for precipitated silica are superior and cost effective compared to present technology of producing Silica from quartz. Starts from a raw material of little or no cost and value, which otherwise would cause environmental pollution. Process is energy efficient and also consumes much lower energy compared to an alternative process involving fusion of selected quality of sand.

III. USES

The precipitated silica market can be broadly classified as rubber grade and non- rubber grade. The total size of the silica market in India is about 16000 tones. (5) Out of this rubber grade silica market is 11000 tones and non-rubber grade market is 5000 tones. Tires, rice roller mills, adhesive, toothpastes, free-flowing salt and certain food products are major users of silica in the country. The market is currently growing at the rate of 8% per annum. Some uses are as listed below

- Reinforcement of elastomeric products like shoe soles.
- Reinforcement of silicone rubber.
- Reinforcing material in tires.
- In sheathing compounds for cables.
- Constituent of adhesives in for bonding of unvulcanized rubber to textiles or steel tire cord.
- In thermoplastics used to act as anti-blocking agents and to prevent plate out effects in films and film production.
- To improve mechanical properties of PVC flooring.
- As carrier silica for materials and as free flow agents for powder formulation, particularly of hygroscopic and adhesive substances.
- As adsorbent.
- In toothpaste to control rheological properties and as a cleansing agent.
- Hydrophobic precipitated silica is used in mineral oil and silicone oil antifoaming effect.
- Purification and stabilization of beer.
- Analysis of blood.
- Cosmetics.
- Food industry as an anti-caking agent.
- Specially prepared silica gels from silica are used for making thermal insulation material.
- As a dehumidifying agent for air and other gases.
- As a filtering agent to clarify of juices.

IV. SILICA IN RICE HUSK&OVERVIEW

The plants belonging to graminae family, namely, rice, wheat, barley, oats, maize are the main sources of biogenic the roots as dissolved monosilicic acid, $Si(OH)_4$& precipitated within around cells as hydrated deposits.(4,6) The monosilicic and moves by evaporation of polymerized to force a cellulose silica membrane. It has been formal that silica is highly concentrated in the inner and outermost surfaces of the epidermal twice. As a result, rice husks are relatively friable, brittle and abrasive. The term white ash refers to residue obtained from complete combustion of rice husk. It if chiefly composed of silica (86-97%). This silica has following structure, in which the atom is bonded to four surrounding O atoms by Si-O-Si bonds. Above fig. shows that each Si atom is surrounded by four O atoms and O atom serves as a neighbor to two Si atoms there by leading to a condensation product with a formula SiO_2.Silica occurs in nature mainly in three crystalline fours namely quartz (hexagonal), cristobolite (white) and tridymite (hexagonal) and also in amorphous form like opal and silica in rice husk being of bioquenic origin is essentially of amorphous nature. But it can be connected into any of above three crystalline forms by heat treatment at different temperature.

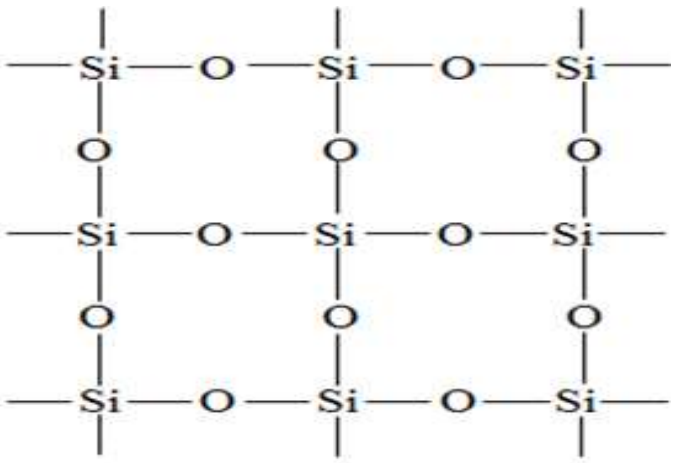

Fig. 1: Structure of silica

A. Properties of Raw Material

Molecular Weight	**60.06**
Nature	Amorphous powder
Appearance	White fluffy powder
Odor	Odorless
Melting Point	1740ºC
Boiling Point	2230ºC
Specific gravity	2.650 at 20ºC
Particle size	1-10 µm

B. Composition of Rice Husk

Element	Percentage
Carbon	37.5
Hydrogen	5.4
Oxygen	36.6
Nitrogen	0.5
Ash	20.0

C. Composition of Rice Husk Ash

Content	Percentage	Adjusted Percentage
Si_2O	90.50	90.00
CaO	1.48	1.307
MgO	1.23	1.005
MnO	1.094	2.356
Fe_2O_3	1.54	1.160
Al_2O_3	1.21	1.452
Others	2.946	3.000

V. ALTERNATIVE PROCESS

A. Conventional

The conventional method of manufacturing silica from quartz or sand in a furnace resembling that used for the manufacturing a glass. The reaction takes place at 1400ºC. The reaction is as follows.(1,3,9)

$$Na_2CO_3 + n\,Si \longrightarrow Na_2O.nSiO_2 + H_2O$$

This process is energy intensive and gives low purity silica

B. Rice Husk, Superheated Steam

Rice husk is subjected to pyrolysis removing volatiles such as hydrogen, carbon dioxide and monoxide. The carbon is simultaneously reacted with superheated steam and converted to gases below 700ºC.

High energy for pyrolysis would be required, even though some of it may be obtained from the gases coming out. The downstream processing of gases would be expensive.

C. Rice Husk, HF, NH_3

Treating rice husk with hydrofluoric acid to give H2SiF6 to this ammonia is added to give a complex (NH_4) 2SiF6 and decomposes into NH4F and SiO2. The filtrate is reacted with strong acid to give HF which is recycled.

High purity silica is obtained (99.9%) 1:10 ratio of Rice husk: HF is high

HF is highly corrosive the organic constituent is not utilized.

D. Proposed Process

This is new process, silica is manufactured by using rice husk ash as a source of silicate or silica and this ash is then reacted with sodium hydroxide which yields the sodium silicate, which on further reacting with sulphuric acid gives silica along with by-product sodium sulphate. The basis of the advantages for this proposed process is first the rice husk ash with higher silica content. The process to extract all the silicate contents from the rice husk is just by burning the rice husk under controlled conditions of air and temp by this condition we can get the clear white ash in the furnace. The basic steps in the production of precipitated silica are

- Obtaining silica from rice husk
- Dissolution of silica in alkali
- Preparation of silica from silicate solution

$$SiO_2 + 2NaOH \xrightarrow{80\text{-}90^oC} Na_2SiO_3 + H_2O$$
(Ash) (Caustic Soda) (Sodium silicate)

$$Na_2SiO_3 + H_2SO_4 \xrightarrow{85^oC} SiO_2 + Na_2SO_4 + H_2O$$
(Sodium Silicate) (Sulphuric acid) (Silica) (Sod. Sulphate)

VI. PROCESS SELECTION

From the above comparison we see that all operations are carried out at temperature (1,7,8) Less than 100ºC. Treating Rice Husk Ash with acid is a exothermic reaction. The energy from this exothermic reaction and combustion of rice husk will also be useful for steam generation which can be used in the process and the excess can be supplied to the nearby plants and generate some additional revenue. So this is the process we select for obtaining silica from rice husk.

A. Process Flow Sheet

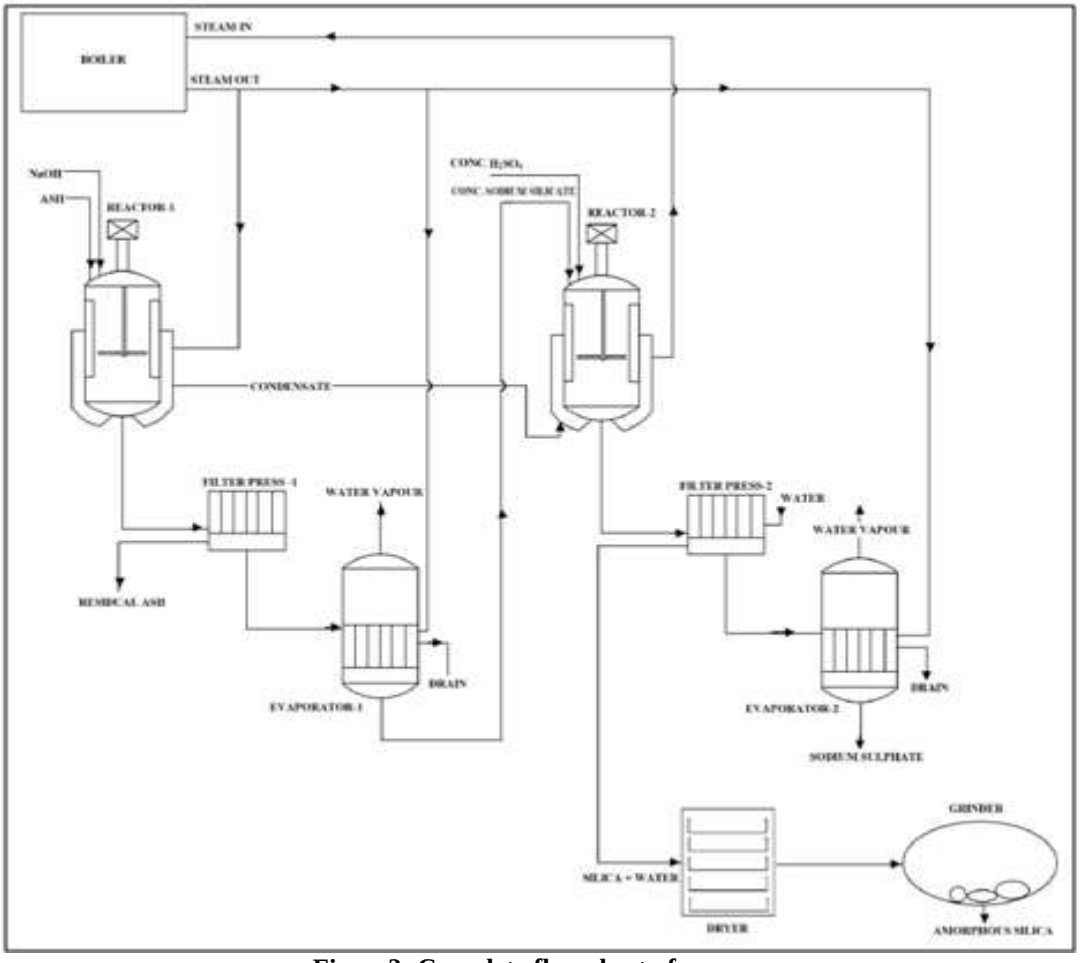

Figure2: Complete flow sheet of process

B. Process Description

The initial step is extraction of silica from ash as sodium silicate using caustic soda. This reaction is carried out at a temperature in the range 180ºC-200ºC and pressure ranging from 6-8 atmospheres. But high reaction temperature and pressure can be avoided if ash obtained by burning rice husk at 650ºC is used. This ash is mostly amorphous silica which is reactive around 100ºC with NaOH solution at atmospheric pressure to yield sodium silicate.

$$\underset{\text{(Ash)}}{SiO_2} + 2NaOH \xrightarrow{80\text{-}90^oC} \underset{\text{(Sodium silicate)}}{Na_2SiO_3} + H_2O$$
(Caustic Soda)

A viscous, transparent, colorless sodium silicate solution (~15%w/w) is obtained after filtration of the reacted slurry (consisting of residue digested ash, sodium silicate, water and free sodium hydroxide). In the second step of the process, silica is precipitated from sodium silicate using sulphuric acid. This step requires controlled conditions of addition rate of sulphuric acid and temperature of reacting mass in a neutralizer. The temperature is in the range of 80-90ºC and pressure is the normal atmospheric pressure.
The reaction is as follows:

$$Na_2SiO_3 + H_2SO_4 \xrightarrow{85^oC} SiO_2 + Na_2SO_4 + H_2O$$

The addition of sulphuric acid is done very slowly (otherwise the chemistry of such mass may change along with physical properties) until acidic conditions are reached. The acidic conditions indicate approximately complete precipitation of silica from sodium silicate. A white precipitate of silica in solution of sodium sulphate is obtained. The silica (wet impure silica) obtained above is filtered. Purification of this silica for removal of sulphate impurities constitutes the third step of process. For this successive demineralized water washings are given in the filter process itself. The conductivity of the effluent follows a decreasing trend owing to removal of sodium sulphate. Thus, conductivity can be used as the criteria to decide the number of washings for obtaining silica of desired purity. Silica after removal of sulphates (wet silica) is generally spray dried to obtained the amorphous powder form in the final step of the process.The purification and drying produce silica in while amorphous powder form. Sodium sulphate from the effluent water and good quality bricks from ash residue are other recoveries.

C. Experimental Procedure

- 100 mg of rice husk is taken as a feed to a muffle furnace.
- It is burnt off, at a temperature range between 800-850ºC, with the help of digital temp probe.
- Heating is continued, till white ash is formed.
- Total time required for burning the husk is about 1.5 to 2 hrs.
- Once, the white ash is formed, it is weighed, which is found to be ¼ of the mass of rice husk i.e. 25 mg.
- Meanwhile, 12% caustic soda (NaOH) solution is prepared for this; 12mg of NaOH is dissolved in 100 ml of distilled water.
- For our convenience and according to the requirements, we have dissolve 24 gm of NaOH in 200 ml of distilled water.
- In a round bottom flask, we are adding ash and caustic soda in 1:7 ratio i.e. 25 gm of ash an175ml of NaOH (to make wet all ash by caustic soda).

This involves the digestion of the rice husk ash with caustic at specific conditions. In this process the silica in the ash gets extracted with caustic to form 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.
So for the proper digestion of RHA in caustic we take 12 gm of NaOH in 100 ml of water i.e. it become 3N solution of caustic.

- The flask is then kept for heating on a heating mantle provide with a temperature probe. Both the reaction time and the temperature for heating mantle provided with a temperature probe.
- Then sample is filtered out from any unburned, carbon impurities by using filter press.
- The residue cake of filter is then weighed and noted down. Clear filtrate obtained is aqueous sodium silicate solution.
- This solution is further concentrated in the oven by keeping temperature
- Range between 100 to 150ºC for 60-90min.

- The final concentrated solution is the gelatinous sodium silicate and its quantity is measured by using electronic weight balance.
- For analysis of sodium silicate, we have carried out some preliminary test such as specific gravity, pH in our laboratory.
- The concentrated solution is the gelatinous sodium silicate taken in 200ml beaker and concentrated sulphuric acid added until solution become acidic. Silica is precipitated from the solution, and sodium sulphate as a by-product remains at bottom. Little quantity of water is added to reduce high exothermic temperature Solution is kept as it is for half an hour to become cool.

Solution is filtered by using filter press by giving water wash. Cake formed is silica and filtered is sodium sulphate, both containing water, place it in different round bottom flask for evaporation of water.
After half an hour take it out and weighed.

VII. CONCLUSION

Rice husk is not just a waste material but more than that thus instead of just disposing it off one can add value to it. By effectively utilizing silica content present in it and to produce precipitated silica from it.From the experiment, it has been observed that the approximately more than 90% of SiO_2 is recovered. An accurate cost estimation of this process is not possible, although rough estimation gives same cost estimation as that of traditional process, the main advantage of this proposed process is that waste disposal problem of rice husk is eliminated and also valuable product is obtained. The amorphous silica obtained using this method have many application, e.g. as filter in rubber products and paper, anti-sticking agents. It is an important catalyst in chemical industry and also serves as the raw material for the production of silicon. Also by-product obtained has industrial importance to produce different derivatives.

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