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- (71) Applicant (for all designated States except US): **THE REGISTRAR, INDIAN INSTITUTE OF SCIENCE** [INRN]; Bangalore 560 012, Karnataka (IN).

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(54) Title: A NOVEL PROCESS AND APPRATUS FOR THE MANUFACTURE OF PRECIPITATED SILICA FROM RICE HUSK ASH

(57) Abstract: A novel process for the manufacture of precipitated silica from rice husk ash, having a surface area ranging from 50-400m<sup>2</sup>/g and tap density of 80-600kg/m<sup>3</sup> having multiple applications in the field of rubbers and plastics, paints, toothpastes, catalyst, carrier agent, insulation, stabilizing and desiccants. The process method for silica precipitation is novel, wherein the chemicals used are regenerated making it a closed loop operation. The extraction process through digestion, precipitation and regeneration are done based on the application specifies, so as to get the required particle size and densities.

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**“A novel process and apparatus for the manufacture of Precipitated Silica from Rice Husk Ash”**

5 This invention relates to a novel process and apparatus for the manufacture of Precipitated Silica from Rice Husk Ash.

10 Process for the production of precipitated silica  $\text{SiO}_2$  from rice husk ash having multiple applications in the field of rubbers and plastics, paints, toothpaste, catalysts, carrier agent, insulation, stabilizing and desiccants. The process for silica precipitation is novel, wherein the chemicals used are regenerated making it a closed loop operation. The extraction process through digestion, precipitation and regeneration are done based on the application specifics, so as to get the required particle size and densities.

**Field of Invention:**

15 The present invention related generally to silica and more precisely to a novel process of obtaining silica from rice husk ash by precipitation using carbon dioxide for the process of precipitation. The process has three stages i.e. digestion of rice husk, precipitation of silica from soluble silicate and regeneration of caustic solution and the final left over is used as activated carbon without involving any further processing.

20

**Introduction:**

25 Rice husk or paddy husk - an agricultural residue is available abundantly in rice producing countries. India alone produces approximately 12 million tons of rice husk annually. Rice husk is generally not advocated as cattle feed because of low cellulose and other sugar contents in it. Furfural and rice bran oil are extracted from rice husk. Rice husk is used by industries as fuel in boilers and for power generation. Rice husk has a high ash content varying from 18-20%. Silica is the major constituent of rice husk ash. High silica ( $\text{SiO}_2$ ) content in rice husk ash is economically feasible to extract silica, which has wide market. The process also addresses the issue of appropriate disposal of  
30 rice husk ash. The strength of this novel process of extracting silica through

precipitation is that with minor changes in the chemical composition the end product silica could be used for all the following application.

**Application of silica:**

- 5       • Reinforcement of rubbers and plastics
- Thickening and thixotropy of coatings and paints, printing inks, plastic and cosmetics
- Matting of lacquers, coating, paints and plastics
- Anti-blocking of plastic foils
- 10       • Free running and free flow of sticky solid or liquid substances
- Carrier for pesticides, catalysts
- High temperature insulation
- Stabilizing (eg of beer, silicon rubbers)
- Desiccants
- 15       • Non eutrophic water softening (eg “builder” materials in washing).

The global scenario on production and consumption pattern of precipitated silica is around 6,75,000 M.T/annum according to 1994 reports. The global market and demand for speciality silicas, which includes Precipitated silica, silica gel, fumed silica and colloidal silica, reached and estimated value of \$1.7 billion. Through the year 2002, the global market for speciality silicas is expected to grow at a rate of roughly 4.0 % a year (in real terms, excluding significant inflation) exceeding \$2 billion in that year.

**Prior Art:**

25 Synthetic amorphous silica, for the industrial applications are produced in four different forms.

**Fumed Silica (Aerosil, pyrogenic) :**

They are produced by vapour phase processes like hydrolysis of silicon tetrachloride or high temperature oxidation and hydrolysis of silicon compounds such as silicate esters.

**Colloidal Silica:**

It is a stable dispersion of amorphous silica particles in water (3-10 microns particle size) having specific surface of 50-270 m<sup>2</sup>/gm) with silica content in the range of 15-50 percent by weight.

5

**Silica Gel:**

It contains three-dimensional network or aggregated silica particles of colloidal dimension. The pores are filled with water. It is generally prepared in acidic conditions.

10 **Precipitated silica:**

They are prepared by destabilization of sodium silicate molecules under conditions that avoid the formation of continuous gel structure. Precipitated silica is powder obtained by coagulation of particles from aqueous solution under the influence of appropriate electrolyte concentration.

15

**Problems with the Prior Art:**

The present patent is specific to the category Conventionally Precipitated Silica is manufactured by of fusion silica sand and sodium carbonate at high temperatures of around 1400 °C and the sodium silicate obtained is precipitated using acid mainly, sulphuric acid.

20

**Following are the limitations of these routes:**

1. The Conventional process of manufacturing silica by sand fusion is highly energy intensive as the process requires the reactants to be heated to high temperatures of around 1400°C.
2. The conventional process and other process using sodium silicate obtained from rice husk ash involves acid (i.e., sulphuric acid) precipitation of the sodium silicate to produce Precipitated Silica. Sodium sulphate is a waste that is generated and this liquid effluent requires elaborate treatment to meet emission standards.

25

3. The process requires effluent treatment plant for treating sodium sulphate. This calls for additional financial implications and any carelessness in treating the effluent would damage the environment.

**5 Brief Description of the Tables:**

**Table 1** – Optimal temperature for Digestion

**Table 2** – Optimal time for digestion

**Table 3** - Product Characteristics and Description

**10 In the accompanying Drawings:**

**Figure 1** is the block diagram illustrating the flow chart of the Digestion step of the present invention;

**Figure 2** is the block diagram illustrating the flow chart of the Precipitation step of the present invention;

- 15 **Figure 3** is the block diagram illustrating the flow chart of the Regeneration step of the present invention

**Figure 4** illustrates a typical Silica Digester used in the process of the present invention;

**Figure 5** illustrates a typical Silica Precipitator used in the process of the present invention; and

- 20 **Figure 6** illustrates a typical Regenerator used in the process of the present invention;

**Description of the Invention:**

The silica precipitation technology developed by us is a novel process for silica precipitation where the chemicals used are regenerated making it a closed loop operation.

- 25 Successful studies for extraction of silica (Chemical and Physical properties of the precipitated silica is given in Table 3) on laboratory scale carried out also meet the industrial requirements. Further studies carried out for suitable application of the undigested ash obtained after extraction, finds application in water treatment plants with or without any further processing as activated carbon.

30

**Production of precipitated silica from 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  
5 industries. This novel process consists of three steps namely;

1. Digestion
2. Precipitation
3. Regeneration

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

**A. Digestion:**

Digestion refers to extraction of the insoluble silica present in the ash to soluble salt in  
15 the form of sodium silicate. The required quantity of sodium hydroxide (ratio varying 'from 1:1 to 1:4 with respect to dry ash) is dissolved in water and the ash is added after the temperature of the caustic reaches greater than 95°C. Digestion studies were carried out at various temperatures in order to optimize the same. Digestion temperature of around  
20 95°C was decided as the optimum because of the higher silica recovery as can be seen in Table 1. The digestion is carried out at this temperature for a period of 1hr, which is the optimum time established by the experimental results as decrease in the time decreases the silica extracted and further increase in time of digestion hardly increases the yield obtained, which is evident from the experimental results given in Table 2.

25 Hence the optimum time for extraction is around 1 hr at around 95°C. Lower temperature extractions yield lower silica and thus increasing the cost of extraction in the form of heat supplied.

**B. Precipitation.**

30 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. Sparging of required carbon dioxide was done with and without pressure and it was observed that the silica produced during the process created blockade of sparger holes when the carbon dioxide pressure was less than of  $1.75 \text{ kg/cm}^2$  (gauge), which posed problems during the final stages in the form of uncontrollable flow rate of carbon dioxide producing silica of different properties each time. During this step the various parameters like temperature, flow of carbon dioxide and silica concentration in the sodium silicate solution play vital role in obtaining silica of different types. Various silica concentrations in the sodium silicate solution ranging from 2.5 % to 10.0 % were tried for precipitation, The variation of parameters results in silica of different quality. It was observed that silica concentration in the range of 4.5 % to 6.0% were suitable for producing the silica required by most of the industries. Higher and lower silica concentrations than the above range produced silica of higher density, in addition to the difficulties in handling the slurry during precipitation at higher concentrations. Precipitation was carried out at various temperatures and it was observed that lower temperatures produced 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 increasing flow rate. The variation in the surface area can be controlled by the amount of carbonation, which is indicated by the determination of carbonate and bicarbonate at the end of precipitation.

20

Precipitation was also carried out with a mixture of air and carbon dioxide at different individual percentages. Similar properties of precipitated silica produced with pure carbon dioxide were obtained. These experiments were carried out to use the industrial flue gases, which contain carbon dioxide in the range of 10– 15 %. Precipitation was also carried out at various  $\text{Na}_2\text{O} : \text{SiO}_2$  ratios.

30

## CHEMICAL SCHEMATIC REPRESENTATION OF PRECIPITATED SILICA USING RICE HUSK ASH:.

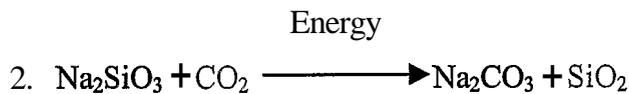
Digestion:

5



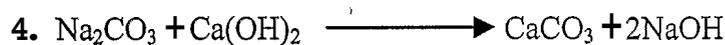
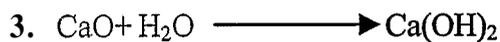
Precipitation:

10



Regeneration:

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The two main requirements for the silica produced are;

1. Surface area.
2. Tap density.

20

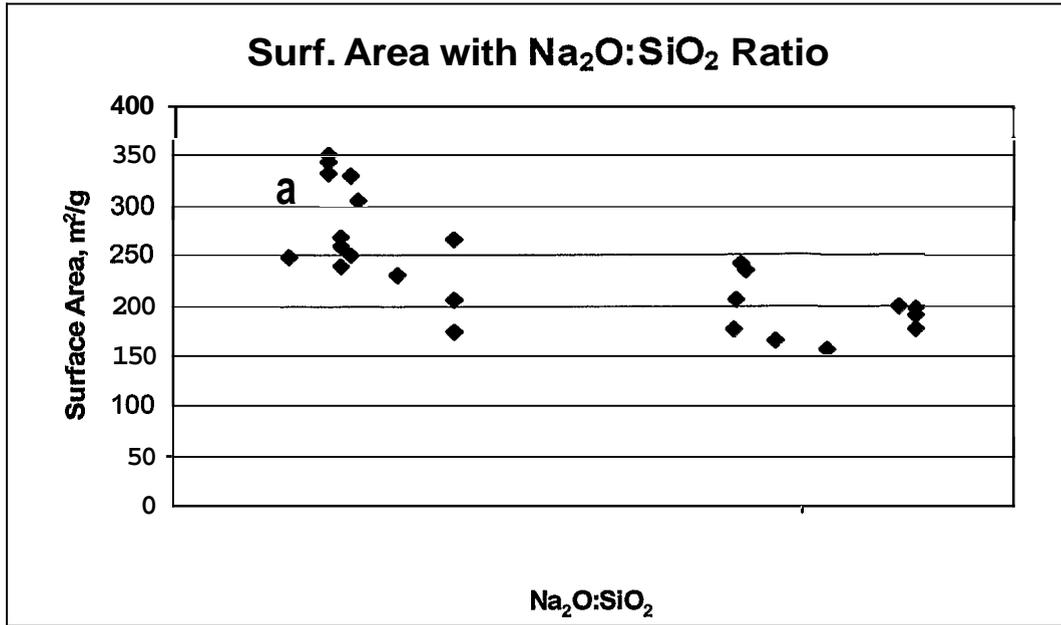
**Controlling factors:**

**a. Surface Area:**

- i. Surface area also depends upon the quality of mixing that takes place inside the precipitator, which varies with the agitator used. The surface area is low (< 80) when the agitator is paddle type (which is basically a horizontal disc with small vertical plate welded at 90 degrees) due to the inefficient mixing. The surface area increases with (>150 when other parameters are controlled) propeller type agitator is used which creates a more vigorous mixing compared to paddle type.

30

Increasing the ratio of Na<sub>2</sub>O: SiO<sub>2</sub> shows a decreasing trend in the surface area as described in the experiments and shown in figure below:

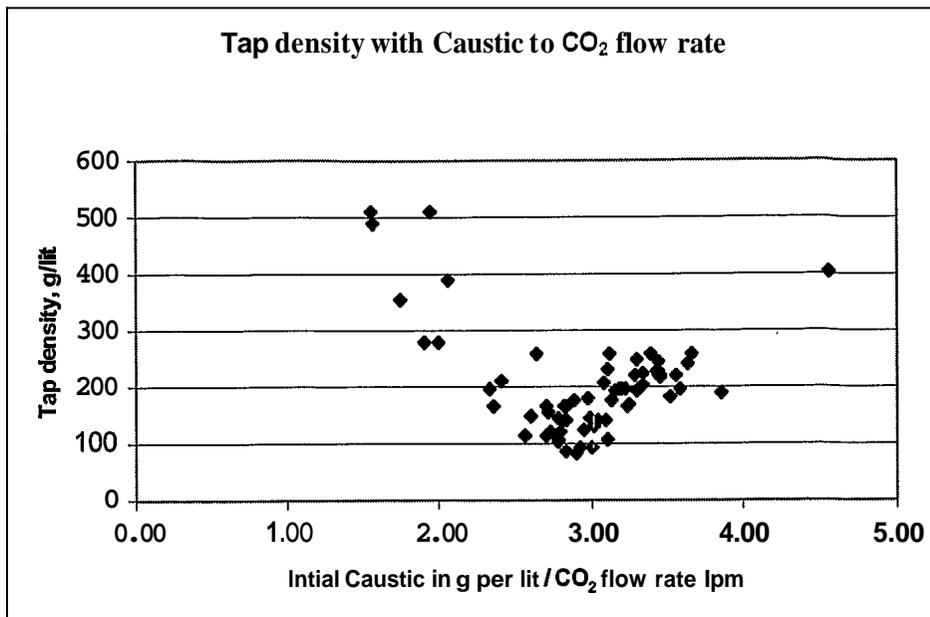


**b. Tap Density:**

- 5 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 tapings.

The tap density of the material is dependent on the ration of initial caustic to carbon dioxide flow rate as indicated in the figure below.

10



With the above-mentioned things in the background, it is possible to control the tap density in the two ranges mentioned below by controlling the carbonation rate;

- 5            a. Around 100 - 150 grams/liter (required by most of the applications).
- b. More than 200 - 250 grams/liter (required mainly by tyre industry).

With the various studies carried out, it is established this process can produce silica of the grades mentioned below by varying the parameters.

10

**C. Regeneration:**

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



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

hydroxide. This depends on the market value of each product and the costs involved for either processing or purchasing.

All the studies carried out for regeneration have yielded around  $\geq 95$  % conversion from sodium carbonate to sodium hydroxide. The regeneration was carried out at different temperatures above  $70^{\circ}\text{C}$  and it was found that at temperatures lower than  $90^{\circ}\text{C}$  the time for regeneration was more and around  $90^{\circ}\text{C}$  it is around **30** minutes for almost complete conversion. The calcium hydroxide used contained around 20 – **30%** of calcium carbonate and hence during the studies corresponding excess of calcium hydroxide was used. The calcium carbonate produced in this process has a purity of around 98.5 % and can be sold in the market.

Alternatively the calcium carbonate obtained can be heated to  $850^{\circ}\text{C}$  upon which it breaks up into calcium oxide and carbon dioxide. The carbon dioxide obtained is used for precipitation of silica from sodium silicate. The calcium oxide is then used for regeneration.



#### **Description of the Apparatus Used for Pilot Scale Studies:**

The Apparatus consists of three parts i.e a digester, a precipitator and a regenerator for carrying out the three main steps of the present invention. Each one is provided with a main reaction vessel 1, with a lid 2 and a flange 3 through which an agitator 4 is placed inside the reaction vessel 1. The flange 3 has an outside diameter of 600mm and an inner diameter of 400mm and is made of stainless steel. The flange 3 is provided with necessary holes 5 for placing bolts and nuts. The agitator 4 is provided with two propellers 6. On lid 2, a condenser 7, a temperature measurement slot 8 and feed inlets 9 & 10 are provided. The feed inlets are used to send the necessary reactants to the reaction vessel. A ball valve 11 is provided for product evacuation and drain valve 12 is provided for sampling. The reaction vessel and the lid are fully insulated with high quality ceramic wool insulation 13 with 0.5mm stainless sheet cladding. The height of the liquid from the bottom of the vessel will be up to level marked 14.

The precipitator is additionally equipped with a sparger 15 through the inlet 10, for carbon dioxide. Through the inlet 16 of the sparger 15, a working pressure of  $> 1.75\text{kg/cm}^2$  is applied to prevent blockade of sparger holes 17, by precipitated silica. In most of the experiments the hole dia was adjusted to let in the carbon dioxide or mixture  
5 of carbon dioxide & air at a velocity of  $> 400$  m/s. The agitator used in the precipitator has a paddle 18.

The length/Diameter of all the vessels is in the ratio of 2: 1.

10 **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 by 80%. 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.  
15 **As such this ash is a waste and disposal poses a challenge. This ash is raw material for producing silica using the process claimed in this patent.**
2. The process suggests closed loop operation hence no unwanted or hazardous chemical is derived **as** a bye-product unlike the conventional processes.  
20
3. Source of Silica is replenishable. It has been mentioned earlier in this document that all rice-producing countries have abundant quantity of rice husk. India alone produces about 12 million tons of rice husk every year.
- 25 4. Commercial Viability is achievable (cost of production can be reduced by 40 %).

**Example 1:**

70 liters of sodium silicate solution containing 5.0 % of silica was taken and heated to  $95^\circ\text{C}$  in a SS vessel with an L/D ratio of **2**. The carbon dioxide flow rate was adjusted in  
30 such a way that the required amount of carbon dioxide was supplied in **83** minutes and after this the carbonation was continued at the same flow rate for another 7 minutes. The

temperature was maintained at 94 – 95°C during precipitation. After the precipitation was completed the silica slurry was kept continuously stirred at the same temperature for another 1.5 hrs. At the end of the experiment the slurry was filtered and the precipitated silica obtained was washed with water, later acidified with small quantity of acid to  
5 adjust the pH of the product (5% slurry) obtained b/n 5.5 – 6.5 and washed again with water to remove minor quantity of salts formed during neutralization. The resultant material was dried in a hot air forced draft tray drier at 110°C. Dried silica was ground using a high-speed grinder. Propeller type agitators (2 Nos) at bottom and middle portion of the vessel and a Paddle type agitator was used in the top to ensure good mixing. The  
10 surface area of silica produced with such was in the range of 150 – 200 m<sup>2</sup>/gm and the tap density of the material was in the range of 160 -220 kg/m<sup>3</sup>. The ratio of Na<sub>2</sub>O:SiO<sub>2</sub> was around 1:1.8.

**Example 2:**

15 The conditions remained identical to example 1 except for usage of only Paddle type agitator (2 Nos) at the bottom and the middle portion of the vessel. Usage of this reduced the precipitation time by around 15 %. The surface area of silica produced with such a procedure was in the range of 50 – 90 m<sup>2</sup>/gm and the tap density of the material was in the range of 160 -220 kg/m<sup>3</sup>.

20

**Example 3:**

70 liters of sodium silicate solution containing around 5.0 % of silica was taken and heated to 95°C in a SS vessel with an L/D ratio of 2. The carbon dioxide flow rate was adjusted in such a way that the required amount of carbon dioxide was supplied in around  
25 140 minutes. The sparger used had 33 % reduced hole diameter compared to other experiments described. At the end of the experiment the slurry was filtered and the precipitated silica obtained was washed with water, later acidified with small quantity of acid to adjust the pH of the product(5 % slurry) obtained b/n 5.5 – 6.5 and washed again with water to remove minor quantity of salts formed during neutralization. The resultant  
30 material was dried in a hot air forced draft tray drier at 110°C. Dried silica was ground using a high speed grinder. Propeller type agitators (2 Nos) at bottom and middle portion

of the vessel and a Paddle type agitator was used in the top to ensure good mixing. The surface area of silica produce with such a procedure was in the range of 150 – 200 m<sup>2</sup>/gm and the tap density of the material was in the range of 80 – 120 kg/m<sup>3</sup>. The ratio of Na<sub>2</sub>O:SiO<sub>2</sub> was 1:3. The results of the experiments are given in Table 4.

5

**Example 4:**

70 liters of sodium silicate solution containing in the range of 5.1 – 5.3 % of silica was taken and heated to 95 °C in a SS vessel with an L/D ratio of 2. The carbon dioxide flow rate was adjusted in such a way that the required amount of carbon dioxide was supplied around 105 minutes. The temperature was maintained at 94 – 95°C during precipitation. The slurry was not agitated after the carbonation and it was immediately and the precipitated silica obtained was washed with water, later acidified with small quantity of acid to adjust the pH of the product (5% slurry) obtained b/n 5.5 – 6.5 and washed again with water to remove minor quantity of salts formed during neutralization. The resultant material was dried in a hot air forced draft tray drier at 110°C. Dried silica was ground using a high speed grinder. Propeller type agitators (2 Nos) at bottom and middle portion of the vessel and a Paddle type agitator was used in the top to ensure good mixing. The surface area of silica produce with such procedure was in the range of 300 – 350 m<sup>2</sup>/gm and the tap density of the material was in the range of 220 - 260 kg/m<sup>3</sup>. The ratio of Na<sub>2</sub>O:SiO<sub>2</sub> was around 1:1.8. The results of five experiments carried out with the above conditions are given Table 5.

**Example 5:**

70 liters of sodium silicate solution containing 4.2 - 4.3 % of silica was taken and heated to 95°C in a SS vessel with an L/D ratio of 2. The carbon dioxide flow rate was adjusted in such a way that the required amount of carbon dioxide was supplied around 80 minutes. The temperature was maintained at 94 – 95°C during precipitation. The slurry was not agitated after the carbonation and it was immediately and the precipitated silica obtained was washed with water, later acidified with small quantity of acid to adjust the pH of the product (5% slurry) obtained b/n 5.5 – 6.5 and washed again with water to remove minor quantity of salts formed during neutralization. The resultant material was

dried in a hot air forced ~~draft~~ tray drier at 110°C. Dried silica was ground using a high speed grinder. Propeller type agitators (2 Nos) at bottom and middle portion of the vessel and a Paddle type agitator was used in the top to ensure good mixing. The surface area of silica produce with such a procedure was in the range of 200 - 260 m<sup>2</sup>/gm and the tap  
 5 density of the material was in the range of 100 - 140 kg/m<sup>3</sup>. The ratio of Na<sub>2</sub>O:SiO<sub>2</sub> was around 1:1.75. The results of the experiments are given Table 6.

#### Example 6:

75 liters of sodium silicate solution containing 2.45 % of silica was taken and heated to  
 10 95°C in the SS vessel earlier used. The carbon dioxide flow rate was adjusted in such a way that the required amount of carbon dioxide was supplied around 40 minutes. The temperature was maintained at 94 - 95°C during precipitation. The slurry was not agitated after the carbonation and it was immediately and the precipitated silica obtained was washed with water, later acidified with small quantity of acid to adjust the pH of the  
 15 product (5% slurry) obtained b/n 5.5 - 6.5 and washed again with water to remove minor quantity of salts formed during neutralization. The resultant material was dried in a hot air forced ~~draft~~ tray drier at 110 °C. Dried silica was ground using a high speed grinder. Propeller type agitators (2 Nos) at bottom and middle portion of the vessel and a Paddle type agitator was used in the top to ensure good mixing. The surface area of silica  
 20 produced was 220 m<sup>2</sup>/gm and the tap density of the material was 510 kg/m<sup>3</sup>. The ratio of Na<sub>2</sub>O:SiO<sub>2</sub> was around 1:1.9.

**Table 1 - Optimal temperature for Digestion**

Experiment Number	Digestion Temp. °C	% Silica in Solution	%Silica digested w.r.t ash taken
1	70 - 72	5.25	48.5
2	80 - 82	6.71	62.08
3	90 - 92	7.13	66.08
4	95 - 97	7.40	68.80

Experiment Number	Digestion Time, hrs	% Silica in Solution	% Silica digested w.r.t ash taken
1	1	7.40	68.80
2	2	7.60	70.09
3	3	7.63	70.70

**Table 3**

**5 Product Characteristics and Description of the precipitated silica produced by the present invention.**

**General details**

Appearance	:	Precipitated Silica is a white free flowing powder. It is a very finely divided silica, in which the degree of polymerization is limited by the preparation technique
Chemical Formula	:	SiO <sub>2</sub>
Molecular weight	:	60.00
Specific gravity	:	2.1 to 2.3
Particle size distribution of the Product after pulverizing	:	1 – 30 microns
PH	:	6 to 8.0
Density bulk kg/m <sup>3</sup>	:	80-500
Solubility	:	Insoluble in water or acids except hydrofluoric acid Soluble in alkali
Surface area	:	50 –400 m <sup>2</sup> /g

Experiment Number	Initial Silica Concentration %	Carbonation Time, min	Tap Density, kg/m <sup>3</sup>	Surface Area, m <sup>2</sup> /g
1.	4.92	135	90	197
2	4.30	145	110	176
3	4.3 1	80	140	222

**Table 5:**

Experiment Number	Initial Silica Concentration %	Carbonation Time, min	Tap Density, kg/m <sup>3</sup>	Surface Area, m <sup>2</sup> /g
1.	5.3	105	260	334
2	5.3	103	221	344
3	5.3	107	260	351
4	5.1	108	260	301
5	5.1	109	260	317

5 **Table 6:**

Experiment Number	Initial Silica Concentration %	Carbonation Time, min	Tap Density, kg/m <sup>3</sup>	Surface Area, m <sup>2</sup> /g
1.	4.22	77	120	211
2	4.30	82	110	260
3	4.3 1	80	140	222

## CLAIMS:

1. A novel process for the manufacture of precipitated silica fi-om rice husk ash, having a surface area ranging from 50-400m<sup>2</sup>/g and tap density of 80-600kg/m<sup>3</sup> which comprises the steps of:
- 5
- a. Digesting insoluble silica (SiO<sub>2</sub>) present in rice husk ash by the addition of Sodium hydroxide (NaOH) to the said rice husk ash in the ratio varying fi-om 1:1 to 1:4 at a temperature of 70-95°C and for a period of 1 to 3 hours to manufacture sodium silicate;
- 10
- b. Precipitating silica fi-om sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>) obtained in step (a) by reaction with pure carbon dioxide CO<sub>2</sub> or with a mixture of carbon dioxide and other inert gas (e.g. air) or with flue gas containing carbon dioxide at a gauge pressure ranging fi-om atmospheric to 3.5 kg/cm<sup>2</sup>, followed by filtration of Precipitated silica, drying and pulverization by known methods; and
- 15
- c. Regenerating the Sodium hydroxide by reacting the sodium carbonate obtained in step (b) by reaction with either calcium oxide or calcium hydroxide at a temperature of 80-95°C, for use in the digestion step once again for a period of in the range of 30 – 90 minutes.
- 20
2. A novel process as claimed in claim 1, wherein digestion is carried out at a temperature of 90-95°C and for a period of one hour.
- 25
3. A novel process as claimed in claim 2, wherein the precipitation and regeneration are carried out for a period of 1 to 4 hours and 0.5 to 1.5 hour respectively.
- 30
4. A novel process as claimed in claim 1, wherein the concentration of sodium silicate in the solution is in the range of 2.5 to 10% by weight.

5. A novel process as claimed in claim 1, wherein the optimal gauge pressure is in the range of 1.75 – 3.5 kg/cm<sup>2</sup>.
- 5 6. An apparatus for carrying out the process claimed in claim 1, comprising a digester, a precipitator and a regenerator, each having a reaction vessel having a lid, an agitator for mixing the reactants, a flange to hold the said agitator in place, said flange having necessary holes for placing the required bolts and nuts, a condenser tube, a temperature measurement slot and two feed inlets for the reactants, a ball valve for product evacuation, a drain valve for sampling, the said  
10 reaction vessel and lid are being fully insulated and the precipitator being additionally provided with a sparger for applying working pressure during reaction.
7. An apparatus as claimed in claim 7, wherein the insulation is high quality ceramic  
15 wool insulation with stainless steel sheet cladding.
8. An apparatus as claimed in claim 7, wherein the reaction vessel, the lid, flange, agitator and the condenser tube are made of stainless steel.
- 20 9. An apparatus as claimed in claim 7, wherein the agitator in the precipitator is provided with a paddle.
10. A novel process for the manufacture of precipitated silica from rice husk ash, having a surface area ranging from 50-400m<sup>2</sup>/g and tap density of 80-600kg/m<sup>3</sup>  
25 substantially as herein before described and illustrated in the examples and figures 1 to 3.
11. An apparatus for carrying out a novel process for the manufacture of precipitated silica from rice husk ash, having a surface area ranging from 50-400m<sup>2</sup>/g and tap  
30 density of 80-600kg/m<sup>3</sup> substantially as herein before described and illustrated in figures 4 to 6.

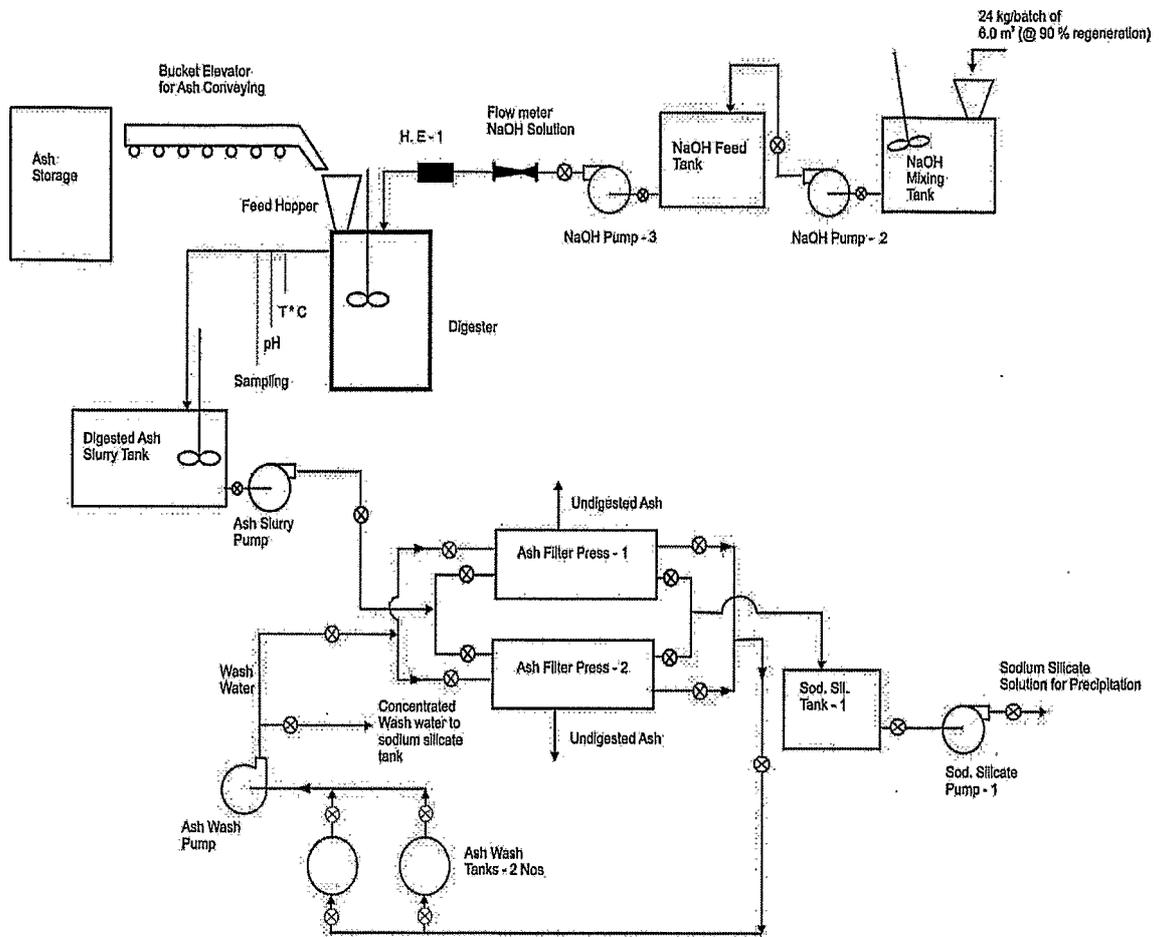


FIGURE - 1

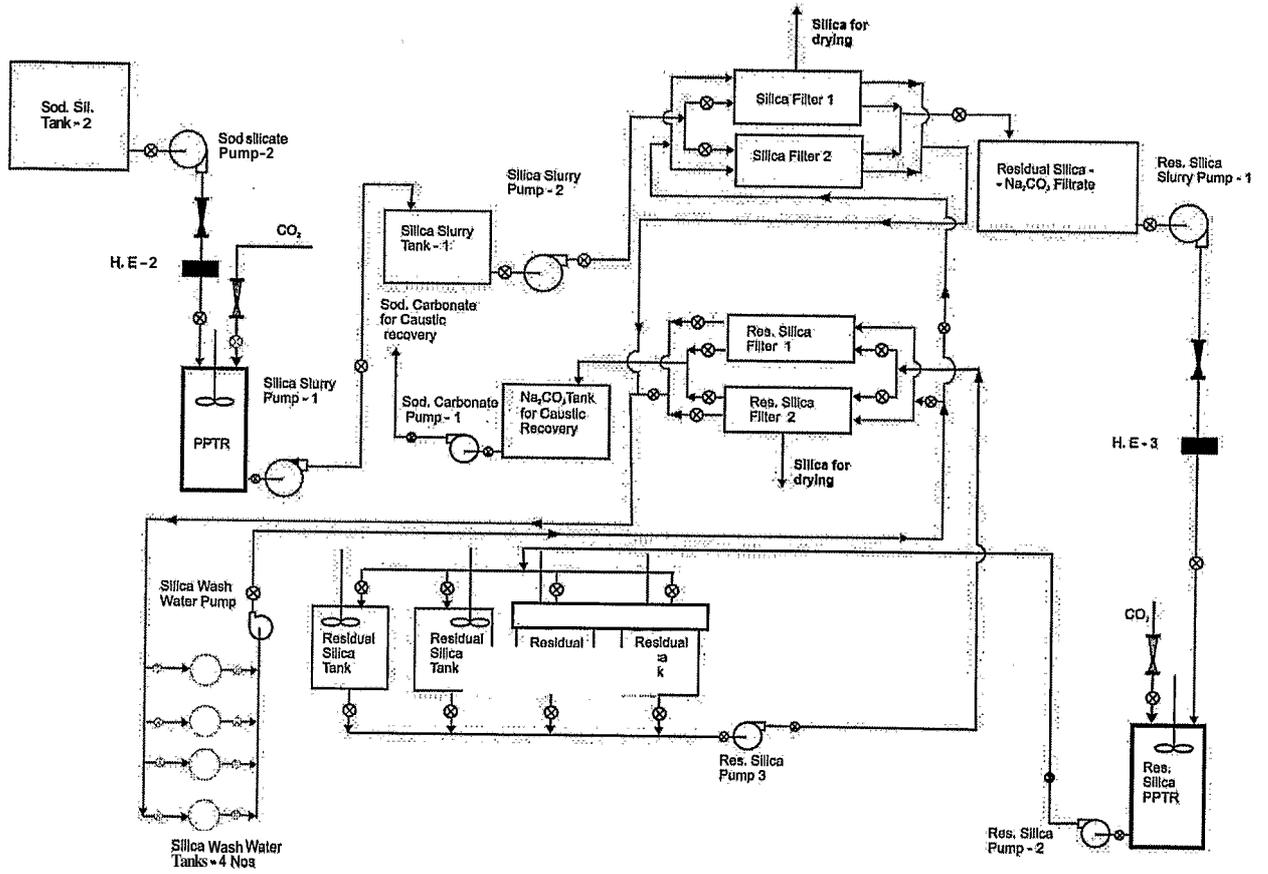


FIGURE-2

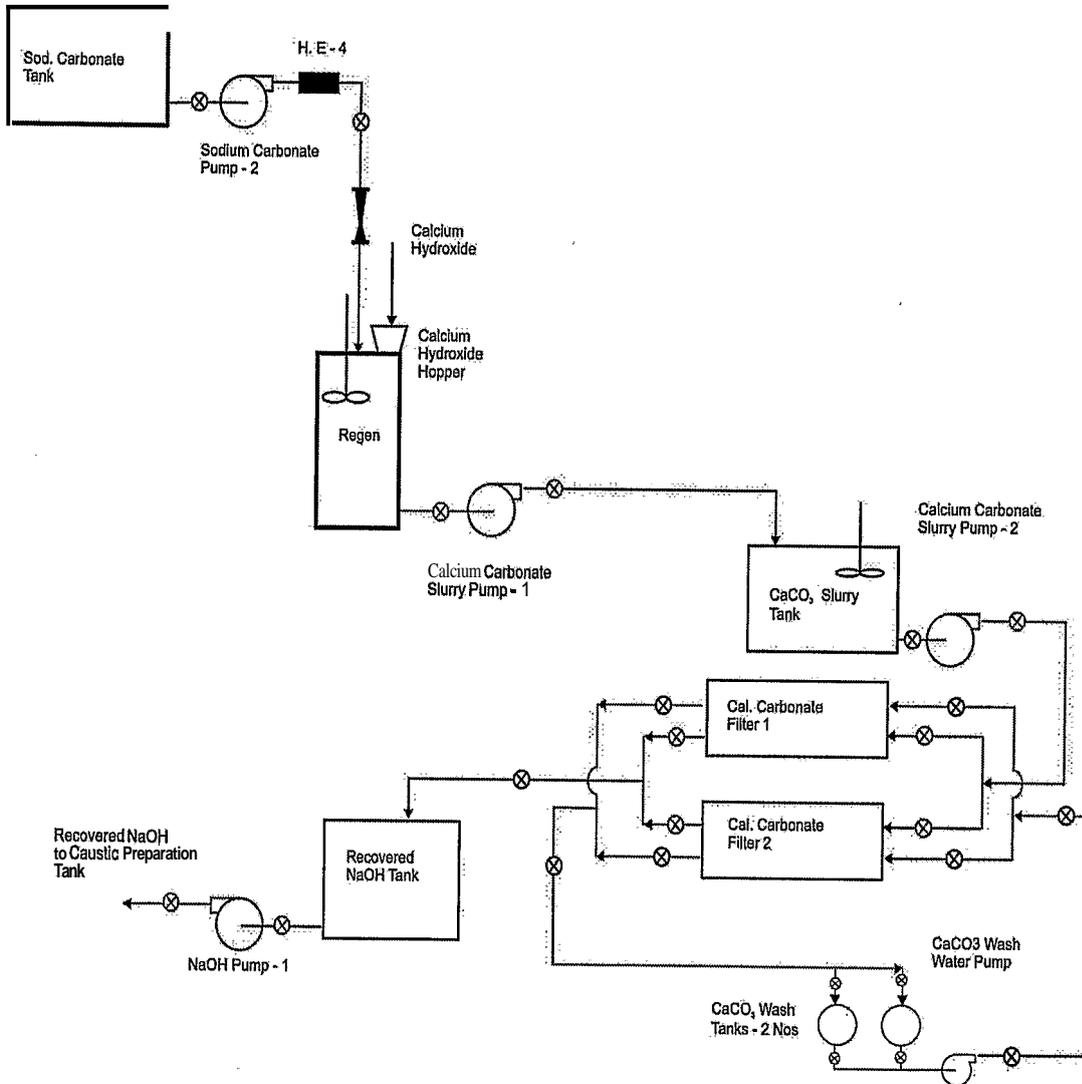
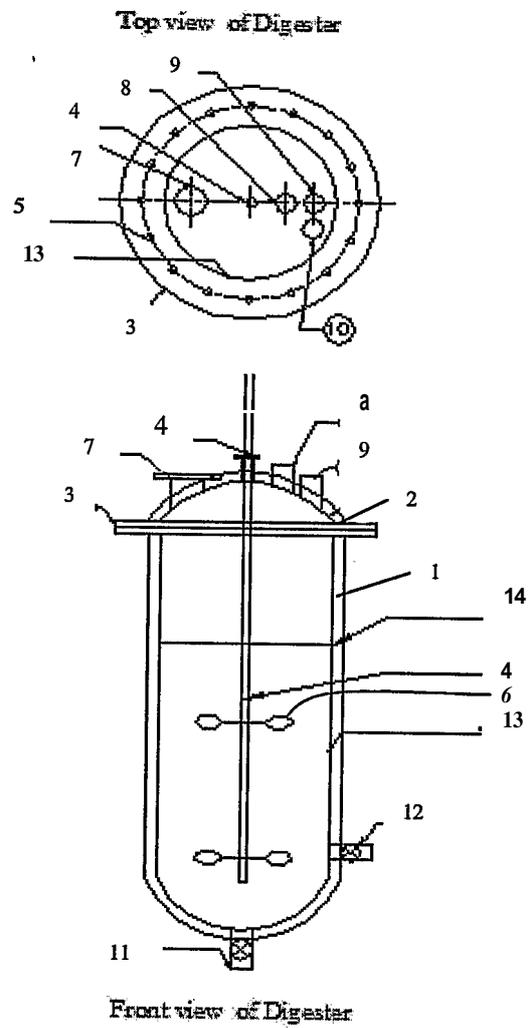


FIGURE-3



**FIGURE-4**

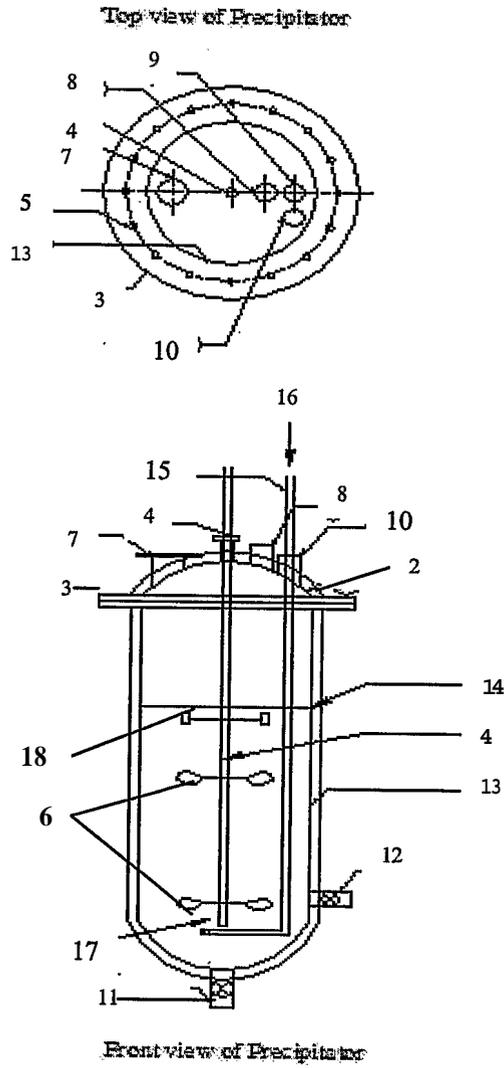
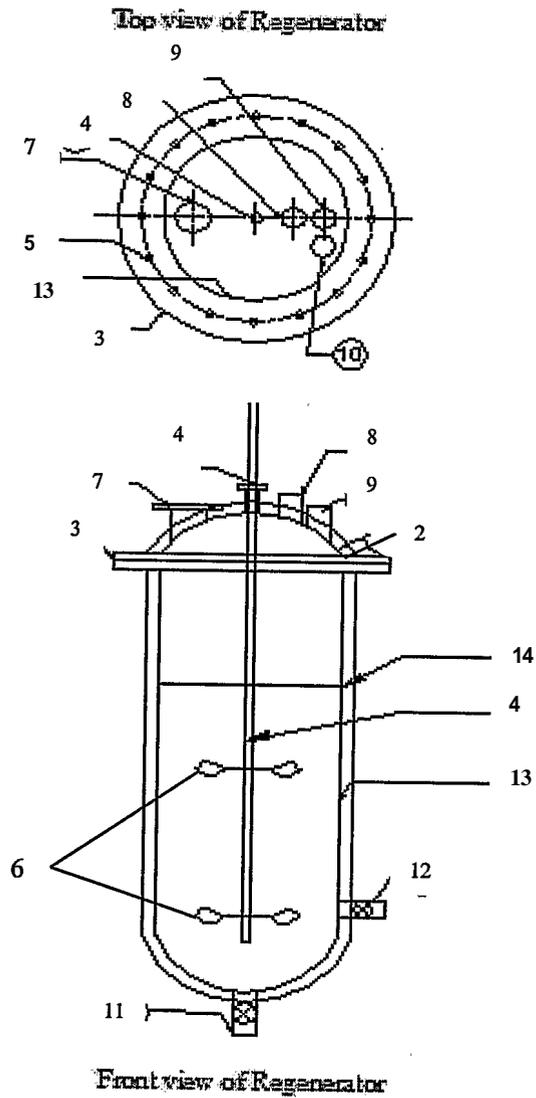


FIGURE-5



**FIGURE-6**