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Formulation of Ecofriendly Detergent Powder Using Paddy Husk Ash

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ABSTRACT

The project mainly aims in making a detergent powder from paddy husk ash. Mesoporous silica is used as a raw material in several areas: as a component of detergents and soaps as a refractory component. Sodium silicate is produced by reacting rice husk ash (RHA) with aqueous NaOH and silica is precipitated from the sodium silicate by acidification. In these conversion of about 90% of silica contained in RHA into sodium silicate was achieved in an open system at temperatures of about 100°C. The results showed that silica obtained from RHA is mesoporous, has a large surface area and small particle size. Rice Husk is usually mixed with coal and this mixture is used for firing boilers. The RHA therefore, usually contains carbon particles. Activated carbon embedded on silica has been prepared using the carbon already present in RHA. This carbon shows good adsorption capacity. The filtrate consists of sodium phosphate.

Keywords: Sodium silicate, Rice husk ash, Activated Carbon.

1. INTRODUCTION

Globally, approximately 600 million tons of rice paddy is produced each year. On an average 20% of the rice paddy is husk, giving an annual total production of 120 million tons. The husk contains about 75% organic volatile matter and the balance 25% of the weight of this husk is converted into ash during the firing process. The ash is known as rice husk ash (RHA). Silica in amorphous form is obtained from RHA produced when Rice Husk is burnt in controlled temperatures below 700 degree Celsius. RHA, a byproduct of the rice industry contain 60-90% of silica and are unique with in nature. The annual worldwide output of rice husk derived silica is more than 3.2 million tons. We have treated RHA with aqueous NaOH for about 1 hour and at low temperatures to convert the silica to sodium silicate. We have used o-phosphoric acid for neutralization. The precipitated silica is filtered off. The silica produced is mesoporous with a large pore surface area. The filtrate consists of sodium phosphate. Silica also has been used as a major precursor for a variety of inorganic and organometallic materials which have applications in synthetic chemistry as catalysts, and in thin films or coatings for electronic and optical materials.

2. RICE HUSK ASH

India has a major agribusiness sector which has achieved remarkable successes over the last three and a half decades. Agricultural waste or residue is made up of organic compounds from organic sources such as rice straw, oil palm empty fruit bunch, sugar cane, coconut shell, and others. Rice husk from paddy (Oryzasativa) is one example of alternative material that has a great potential. Rice husk a major by-product of the rice milling industry, is one of the most commonly available lignocellulosic materials that can be converted to different types of fuels and chemical feed stocks through a variety of thermo chemical conversion processes. Rice husk is an agricultural residue abundantly available in rice producing countries. The husk surrounds the paddy grain. During milling of paddy about 78 % of weight is received as rice, broken rice and bran. Rest 22 % of the weight of paddy is received as husk. This husk is used as fuel in the rice mills to generate steam for the parboiling process. This husk contains about 75% organic volatile matter and the balance 25% of the weight of this husk is converted into ash during the firing process, is known as rice husk ash. This RHA in turn contains around 85% to 90% amorphous silica. The moisture content ranged from 8·68 to 10·44%, and the bulk density ranged from 86 to 114 kg/m^3. Rice husk is unusually high in ash, which is 92 to 95% silica, highly porous and lightweight, with a very high external surface area. Its absorbent and insulating properties are useful to many industrial applications, such as acting as a strengthening agent in building materials. Rice husks are processed into rectangular shaped particle boards. Construction industry is one of the fastest growing sectors in India.

Rapid construction activity and growing demand of houses has lead to the short fall of traditional building materials. Bricks, Cement, sand, and wood are now becoming scares materials. Demand of good quality of building materials to replace the traditional materials and the need for cost effective and durable materials for the low cost housing has necessitated the researchers to develop variety of new and innovative building materials. Construction materials of special requirements for the houses in different geographical region to overcome the risk of natural hazard and for protection from sever climatic conditions has also emphazised the need for development of light weight, insulating, cost effective, durable and environment friendly building materials. Rice hulls can be put to use as building material, fertilizer, insulation material or fuel. Rice hulls uses include aggregates and fillers for concrete and board production, economical substitute for microsilica, absorbents for oils and chemicals, soil ameliorants, as a source of silicon, as insulation powder in steel mills so as to name a few. India is a major rice producing country, and the husk generated during milling is mostly used as a fuel in the boilers for processing paddy, producing energy through direct combustion and / or by gasification. About 20 million tones of RHA are produced annually. This RHA is a great environment threat causing damage to the land and the surrounding area in which it is dumped. Lots of ways are being thought of for disposing them by making commercial use of this RHA. The annual rice husk produce in India amounts is generally approximately 120 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 gasification, rice husk has a high ash content varying from 18 to 20 %. Silica is the major constituent of rice husk ash and the following tables gives typical composition of rice husk and rice husk ash. With such a large ash content and 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. A number of rice-producing countries are currently conducting research on industrial uses of rice hulls.

2.1 HOUSEHOLD DETERGENTS POWDER

The detergent market is a highly competitive on where several brands via with each other to get the customers attention. Each brand claims to clean whiter and better, boasting of power pearls and of ability of fight granules, and so on. In all this, what consumers may end up overlooking are the chemical composition and quality of the detergent powder they use, though their implications for personal health and the environment are critical enough to

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merit closer attention and action. The detergent market in India consist of two major categories: hand-wash and the machine-wash categories. Powder detergents and bar detergents form a major portion of the hand-wash segment. In the machine-wash segment, powder detergents and liquid detergents are the main type. The laundry soap that had been traditionally used for washing of cloths/fabrics has limitations in terms of performing in highly alkaline or acidic water. In alkaline water, part of the soap is consumed to first soften the water and in the process its cleaning property gets reduced. In acidic water the soap gets split into fatty acids and caustic solution, and this retards its cleaning property. These limitations of soaps have led to the development of synthetic detergents that are superior in performance.

2.2 DETERGENCY

Detergency is the ability to clean or remove soil, generally associated with the action of a cleaning agent such as soap, detergent, or alkaline salt. As per for detergent powders is: 65% minimum for Grade 1, 55% for Grade 2, and 45% for Grade 3.None of the brands met the specified requirements for Grade 1.The detergency percentage ranged from 57.68% to a low of 39.15%.

2.3 ACTIVE INGREDIENTS

Detergents have certain ingredients (Known as active ingredients) that are responsible for ensuring their cleaning performance. The national standards have specified different minimum active ingredient levels for the three grades of detergents: 19 percent for Grade 1, 16 percent for Grade 2, and 10 percent for Grade 3.

2.4 ASH BUILT-UP

This test determines the built-up of ash on a fabric. The Indian Standard has set the requirement as: 1 percent maximum for Grade 1, 5 percent for Grade 2, and 10 percent for Grade 3.

2.5 ACTIVE ALKALINITY

Alkalinity is a measure of the ability of a solution to neutralize acids to the equivalence point of carbonate or bicarbonate. As per the Indian Standard, the active alkalinity of detergent powder of Grade 1, 2 and 3 should be 15 percent, 20 percent and 30 percent, respectively. Most of the brands met the requirements for Grade 1 and the rest made it to Grade 2.

2.6 TOTAL PHOSPHATES

The Indian Standard specifies the minimum quantity of phosphates as an ingredient in detergents 11 percent for Grade 1 & 7 percent for Grade 2. No requirement has been specified for Grade 3. As per the ecomark criteria for detergent powders in the Indian Standard, any substitute used for phosphate shall be environment-friendly but should be of sufficient quantity to ensure similar performance of the product as compared to a detergent with phosphates.

2.7 STPP

Sodium TriPolyPhosphate (STPP) is mostly used as a phosphate ingredients in many detergents. It softens the water and prevents dirt particles from adhering to the garment. However, the use of STPP is also associated with environmental hazards. The Indian Standard specifies the minimum quantity of STPP in detergent powder at 9.5% and 6%by mass for Grade 1 and Grade 2, respectively. No requirement has been prescribed for Grade 3.

2.8 MOISTURE

The national standards have not prescribed a maximum limit for moisture content in detergents, but it is known that the presence of high moisture leads to the detergent powder turning lumpry.

2.9 LATHER

There is on specified requirements for lather in the national standards. In any case, foam generation should be high and at a faster rate.

3. MOTIVATION

Usually fragrance are added to many cleaners, our motivation for this project is to avoid the fragrance. The most notably laundry detergents may cause effects such as respiratory, headache, sneezing and watery eyes in sensitive individuals or allergy and asthma sufferers.

4. MATERIALS AND METHODS

The rice husk is mixed with coal (10% coal, 90% rice husk). The initial step is extraction of silica from ash as sodium silicate using aqueous sodium hydroxide. RHA contains mostly amorphous silica which reacts at around 90-100^{0}C with NaOH solution to yield sodium silicate. A viscous, transparent, colourless sodium silicate solution is obtained after filtration of the reacted slurry. The residue (on the filer medium) consists of the unburnt carbon and coal ash. In the second step of the process, silica was precipitated from sodium silicate by acidification using orthophosphoric acid. The addition of the acid was done very slowly till a pH of 6.5 was reached. A precipitate of white silica was obtained. The silica in aqueous solution of sodium phosphate obtained above was filtered. As per the norms of Indian standard for Grade 1 product the 0.01 % of phosphates used. To make more environmental free along with the 0.01% of phosphate we are adding sodium bicarbonate. The chemical equations involved in the process of synthesizing silica powder are:

Distilled water is applied for all synthesis and treatment processes.

$$SiO_2 + 2NaOH \rightarrow Na_2SiO_3 + H_2O \rightarrow \quad \textbf{(1)}$$

$$Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2 + Na_2SO_4 + H_2O \rightarrow \quad \textbf{(2)}$$

4.1 ACID PRE-TREATMENT OF MATERIAL

The aim of acid pre-treatment is to improve the purity of silica product. It proves to be an effective way in substantially removing most of the metallic impurities and producing ash silica completely white in colour. It was conducted in the following manner. Ten grams of RHA samples were dispersed in 60 ml of distilled water, and the pH was adjusted. These dispersions were stirred for 2 hours, filtered through ashless filter paper and then the RHA residues were washed with 100ml of water. The residues were used for silica extraction. The filtrate and washings at each pH were collected and dried in an evaporating dish.

4.2 SILICA EXTRACTION

60ml portions of 1N NaOH were added to the washed and unwashed RHA samples separately and boiled in a covered 250ml Erlenmeyer flasks for 1 hour with constant stirring. This step is to dissolve the silica and produce a sodium silicate solution. The solutions were filtered through ash less filter paper, and the carbon residues were washed with 100 ml of boiling water. The filtrates and washings were allowed to cool to room temperature and were titrated against 1N HCl with constant stirring to pH 7. Silica gels started to precipitate when the pH decreased to <10. The silica gels formed were aged for 18 hours. Deionised water (100ml) was added to gels and then the gels were broken to make slurry. Slurries were then centrifuged for 15 min at 2500rpm, the clear supernatants were discarded and the washing step was repeated. The gels were transferred into a beaker and dried at 80^oC for 12 hours to produce Xerogels.

4.3 MOISTURE CONTENT OF SILICA GELS

Moisture content of the silica gels was determined using an air oven method. About 1g of each sample was heated for 1 hour in an aluminium moisture pans at 130^oC. The samples were cooled in desiccators and weighed. The weight loss (%) was recorded as the moisture content of samples. Chemical Analysis of Silica Powders the silica samples obtained using different acids and degree of agglomeration of silica particles were analysed using Scanning Electron Microscopy (SEM). Use either SI (MKS) or CGS as primary units. (SI units are strongly encouraged.) English units may be used as secondary units (in parentheses).For example, write "15 Gb/cm^2 (100 Gb/in^2). An exception is when English units are used as identifiers in trade, such as "3½ in disk drive." Avoid combining SI and CGS units, such as current in amperes and magnetic field in oersteds. This often leads to confusion because equations do not balance dimensionally.

5. PROCEDURES

A) AMORPHOUS SILICA

The initial step is extraction of silica from ash as sodium silicate using aqueous sodium hydroxide. This reaction was carried out in an open stainless steel reactor for about 60 minutes at a temperature of 99^oC and at atmospheric pressure. RHA contains mostly amorphous silica which reacts at around 90 to 100^oC with NaOH solution to yield sodium silicate. The RHA also contains some unburnt carbon from the coal as also some ash from the burnt coal. A viscous, transparent, colourless sodium silicate solution is obtained after filtration of the reacted slurry. The residue (on the filer medium) consists of the unburnt carbon and coal ash. In the second step of the process, silica was precipitated from sodium silicate by acidification using orthophosphoric acid. The addition of the acid was done very slowly till a pH of 6.5 was reached. A precipitate of white silica was obtained. The silica in aqueous solution of sodium phosphate obtained above was filtered. Purification of this silica for removal of sodium phosphate trapped in the silica mass constituted the third step of the process. For this silica was subjected to successive washings with demineralized water. The washings were added to the bulk of sodium phosphate solution. The wet precipitate was dried in an oven for 24 hours at 110^0C in the final step of the process. Temperature of digestion was kept constant at 99^oC.

1) NAOH CONCENTRATION

12g of NaOH was dissolved in 30ml, 40ml and 50ml of water keeping all other parameters constant. Experiments were carried out to study the effect of NaOH concentration on yield of silica.

2) REACTION TIME

The digestion of RHA with aqueous sodium hydroxide was carried out for 40, 60, 90 and 120 minutes to study the effect of digestion time on yield of silica.

3) RHA CONCENTRATION

Different weights (10g, 11g, 12g, 13g, 14g) of RHA were dissolved in a fixed quantity of aqueous NaOH solution to study the effect of RHA concentration on silica yield Optimum quantity of silica is obtained with 10g RHA, 12g NaOH, 30ml water and a digestion time of 60 minutes.

B) ACTIVATED CARBON

RHA was digested with NaOH solution. The carbon was not separated as in precipitation of amorphous silica. The whole mass (sodium silicate solution and carbon) was acidified with sulphuric acid while under intense agitation. The black precipitate was separated by filtration, washed with hot water several times and dried.

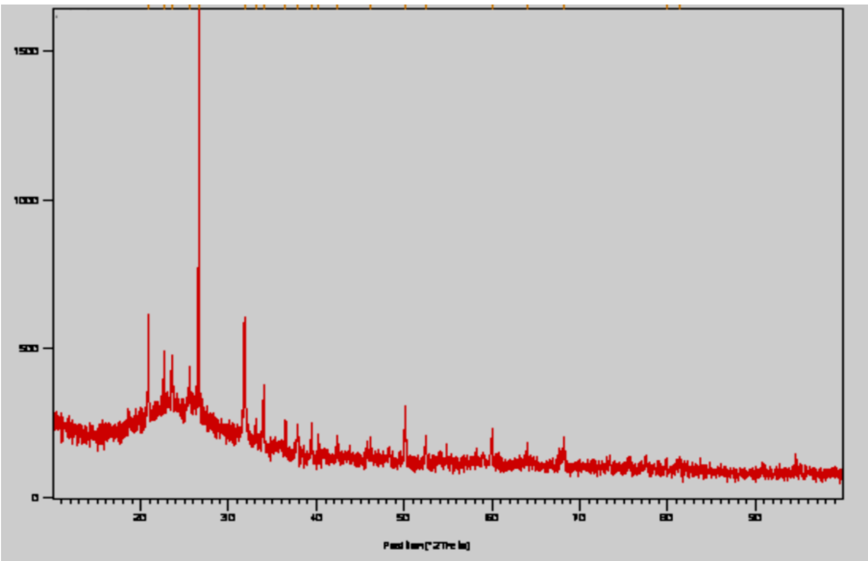

FIG 1: XRD OF A TYPICAL SILICA SAMPLE F1

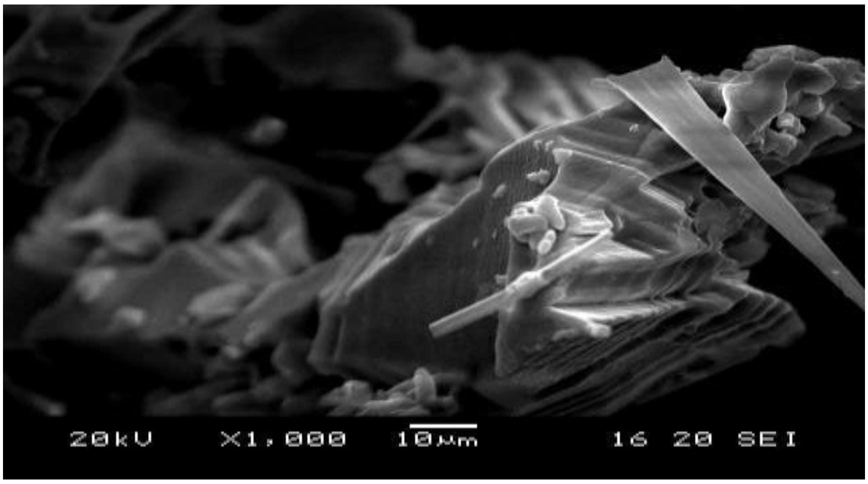

FIG 2: SEM IMAGE SHOWS SILICA PARTICLES OBTAINED BY USING HCL IN ACID WASHING

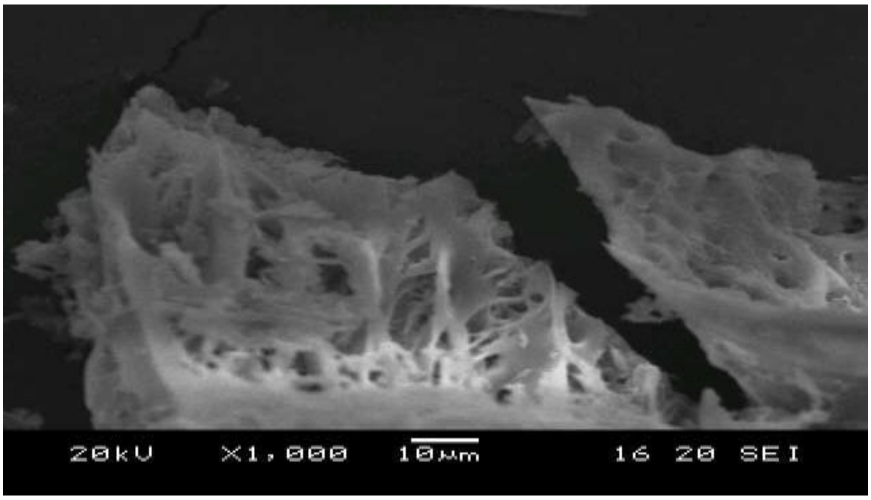

FIG 2.1: SEM IMAGE SHOWS SILICA PARTICLES OBTAINED BY USING HNO3 IN ACID WASHING

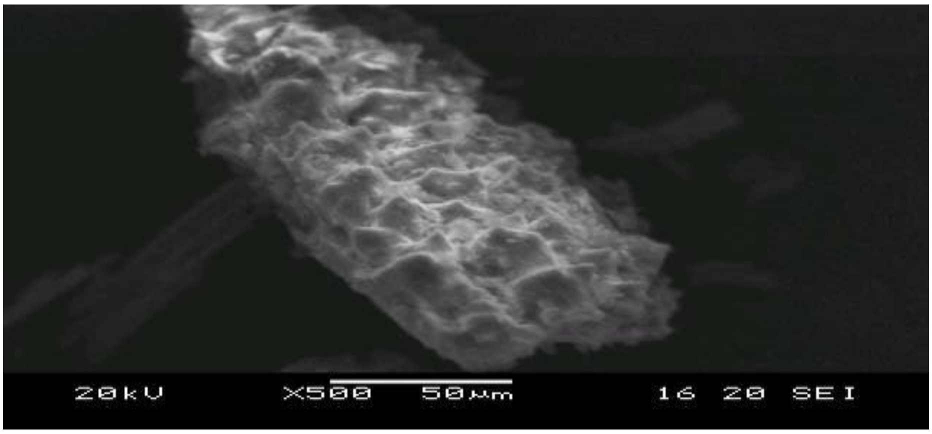

FIG 3 SEM IMAGE SHOWS SILICA PARTICLES OBTAINED BY USING H2SO4 IN ACID WASHING

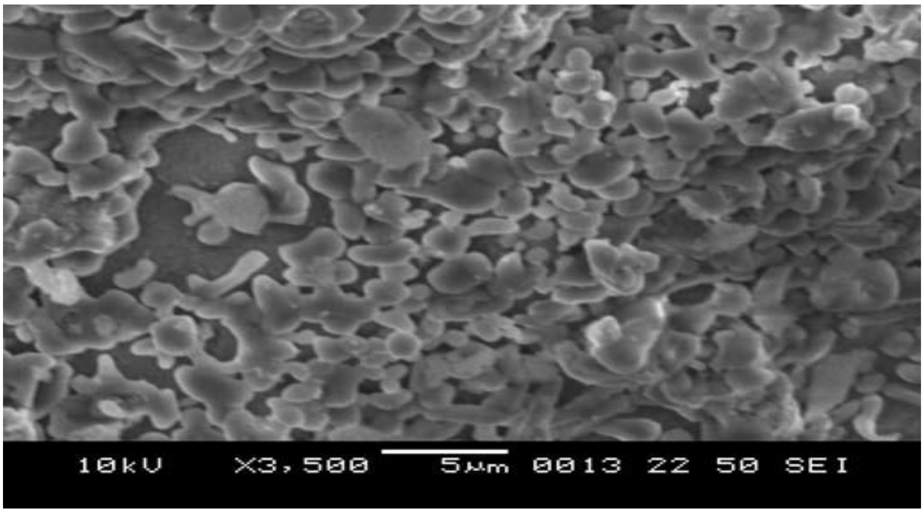

FIG 2.2: SEM MICROGRAPH OF A TYPICAL SILICA EXAMPLE

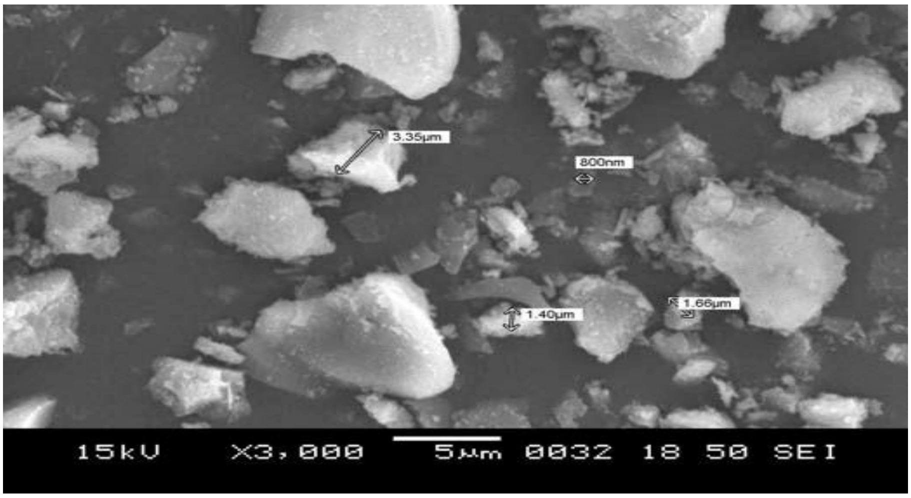

FIG 2.3: SEM MICROGRAPH OF CARBON ON SILICA

6. CHARACTERIZATION

The instruments used for analysis were for SEM, XRD, UV visible Spectro photometer, FTIR, RSM.

7. WORKING

G Salt is added into the bucket stir it for a while, then add a sodium carbonate. Keep stirring it for another minute, then add a sodium bicarbonate. Stir it for another couple of minutes, then add an extracted sodium phosphate from RHA stir it for another couple of minutes. Then add an acid slurry stir it for another minute or two, then let the stuff sit over night to cool.

8. RESULTS AND DISCUSSION

The physical properties of the recovered silica are:

- Nature: White amorphous powder
- Purity : 98 %

- Bulk density: 1.25 g/ml

A) MESOPOROUS SILICA

The silica samples obtained were subjected to XRD and SEM analyses. It is seen that the silica obtained is largely amorphous with a particle size of about 5 micrometers. BET pore surface area was measured and was found to be 120 m2/g. The average pore diameter was found to be 19.9317 namometers.

B) ACTIVATED CARBON

Fine particles of silica with a carbon background. The average silica particle size is less than 5 micrometers. The total BET surface area is found to be 100 m2/g. The average pore diameter was found to be 18.347 namometers.

9. ADVANTAGES

Eco-friendly, Contain no optical brighteners, No dyes, or No artificial fragrances.

10. CONCLUSION

It is found that it is possible to recover over 90% of the silica contained in RHA by simple digestion with aqueous sodium hydroxide and precipitation of silica by acidification of the sodium silicate solution so obtained. The silica obtained is very largely amorphous. From the results of our project detergents powder are eco friendly but still have some small harm to the environment.

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