International Research Journal of Engineering and Technology (IRJET) e-ISSN: 2395-0056
Volume: 10 Issue: 09 | Sep 2023 www.irjet.net p-ISSN: 2395-0072

Synthesis of Nano-Silica Material from Agricultural Wastes

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Abstract – *One of the major causes of pollution in India is the burning of agricultural waste. In this study, we are preparing nano-silica material from rice waste which is rice husk. Rice husk is an abundantly found agricultural waste and not only this it is also associated with disposal problems, therefore, but the best way of dealing with this problem also is to make the best out of this waste and therefore we have taken rice husk as a raw material which will ultimately help in boosting the Indian economy. Rice husk can be efficiently transformed into a valuable product like nano-silica which is highly pure and has a high surface area. In India, rice husk is generally used for landfilling but it is not environment friendly. So, we are using this waste rice husk to extract nano-silica powder. The rice husk contains many types of impurities such as metal impurities and unburned carbon which gives an adverse effect on the color and purity of silica. We have performed the alkali extraction method with acid preparation to get pure nano silica from rice husk. The use of RH and RHA in an efficient manner is likely to transform this agricultural byproduct or waste into a valuable product that might help in boosting the farm economy & rural development. As India is the second-largest rice-producing country in the world, a systematic approach to nano-silica material can give birth to a new industrial sector of rice husk ash in India.*

***Key Words*: Agriculture waste, Environment, Rice husk ash, Silica**

1. INTRODUCTION

In this chapter, rice husk ash (RHA) has been used for the synthesis or extraction of nano-silica material. Before proceeding to our objective, let us start with the basic introduction of the terms which are going to be used further in this chapter.

1.1. Nano-Silica:

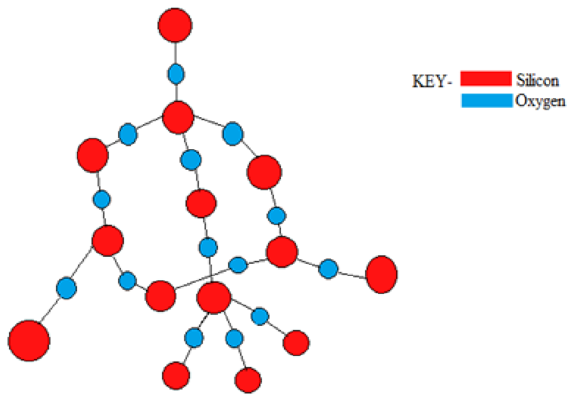

Fig -1: Structure of nano-silica

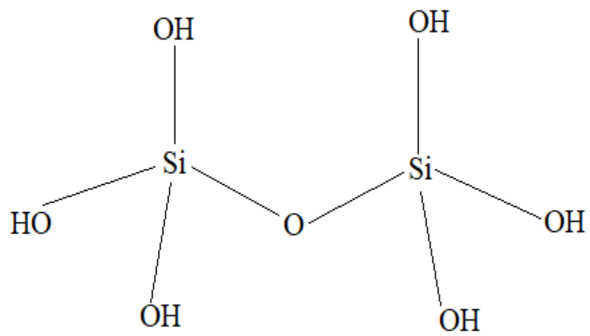

Fig -2: Structure of nano-silica

Nano-silica is the term used for Silicon Dioxide (SiO_2) nanoparticles with silicon (46.8%) and oxygen (53.3%). Silica nanoparticles are also known as silica dust or quartz dust. It is an acidic oxide as its surface contains –an OH bond. It can be identified as a white-colored amorphous silica powder of high purity (99.9%). Some properties of nano-silica are classified below;

1.1.1. Physical Properties:

It is a white-colored, amorphous and crystalline, and fluffy textured silica powder that has a density of 2.4 g/cm^3 and 59.96 g/mol of molar mass. Its particle size is small (about 10-20nm) provides it to have a large specific surface area and strong adsorption capacity. Also, it is a highly porous and hydrophilic material with high stability and low toxicity. Nano-silica is of two types based on the structure; P-type and S-type.

Table -1: Physical properties of nano-silica particles

Physical Properties:	Silica Nanoparticles:
Appearance	White-colored, fluffy textured powder.
Structure	Amorphous, crystalline.
Density	2.2 - 2.6 g/cm^3
Molar mass	59.96 g/mol
Particle size	10-20nm

1.1.2. Chemical Properties:

The chemical formula of the nano-silica particle is SiO_2, with group silicon-14 (46.8%) and oxygen- 16 (53.3%). The electronic configuration of silicon is [Ne] $3s^2$ $3p^2$ and oxygen is [He] $2s^2$ $2p^2$. It belongs to the acidic group, has low water solubility, and has high chemical reactivity.

Table -2: Chemical properties of nano-silica particles

Chemical Properties:	Silica Nanoparticles:
Chemical formula	SiO_2.
Group	Silicon-14, Oxygen- 16.
Electronic configuration	Silicon - [Ne] $3s^2$ $3p^2$ Oxygen - [He] $2s^2$ $2p^2$
Composition	Silicon - 46.8% Oxygen – 53.3%
Nature	Acidic.
Reactivity	Highly reactive.
Solubility in water	Low.

1.1.3. Thermal Properties:

The thermal properties of nano-silica include; boiling point and melting point. The boiling point of silica nanoparticles is 2230°C (4046°F) and the melting point is 1600°C (2912°F). Nano-silica particles, when added to a material, can increase its thermal stability.

Table -3: Thermal properties of nano-silica particles

Thermal Properties:	Silica Nanoparticles:
Boiling point	2230°C (4046°F).
Melting point	1600°C (2912°F).

Because of the large surface area and porosity of the nano-silica, it is used in products such as fillers, pharmaceuticals, catalysts, and chromatography. Adding nano-silica particles to paints can increase their stability, strength, finishing, etc. They are also used in plastic production to provide strength, toughness, etc. When nano-silica particles are added to rubber, it increases its toughness, strength, elongation, bending, anti-aging performance, etc. 2-5% of nano-silica is added to concrete during the manufacturing of cement which can enhance the performance of concrete. Nano-silica is also used in semiconductors.

1.2. Rice Husk Ash:

Fig -3: Rice husk

Fig -4: Rice husk ash

Rice husk ash (RHA) is obtained by the burning of rice husk, which is an abundantly available agricultural waste. As rice is one of the common food crops with an annual gross production of about 510 million metric tons in the world its production is mainly dominated across Asia (roughly around 112 million metric tons) therefore, there is a large availability of rice husk. It is composed of cellulose (50%), silica (15-20%), lignin group (25-30%), and moisture content (10-15%). Rice husk's bulk density is low and has a range of 90–150 kg/m^3. Its calorific value is 50% of the calorific value of coal. Rice husk has been considered a great source of silica. When this rice husk is burnt at a certain temperature (below 800°C) for a required period, rice husk ash (RHA) is obtained. In this burning, only one-fifth to one-quarter of the initial weight is obtained as rice husk ash. The silica proportion in this rice husk ash is greater than 80%, which is the highest proportion of silica as compared to other plant residues. Along with silica, it also contains a very small proportion of impurities like; K_2O, Na_2O, and Fe_2O_3. These impurities can be removed by acid leaching. The silica obtained from rice husk ash is mainly in amorphous and powdered form and is about 99.9% pure. The rice husk ash characteristics depend upon the following;

(i). the rice husk's composition,

(ii). its burning temperature, and

(iii). It's burning time.

Some properties of rice husk ash are mentioned below;

1.2.1. Physical Properties:

Rice husk ash is a greyish-black colored, lightweight, amorphous powder. It is a highly porous material with a high specific surface area. RHA has a high microporous structure due to which its specific surface area can be determined by the Brunauer-Emmett-Teller (BET) adsorption method, this can range from 20 m^2/gm to 250 m^2/gm. Its particle size can vary up to 10 μm. The specific gravity of rice husk ash is about 2.11 to 2.27.

Table -4: Physical properties of rice husk ash

Physical Properties:	Rice Husk Ash:
Appearance	Greyish-black color, lightweight powder.
Structure	Amorphous.
Surface area	20 m^2/gm - 250 m^2/gm.
Particle size	vary up to 10 μm.
Specific gravity	2.11 - 2.27.

1.2.2. Chemical Properties:

The composition of rice husk ash depends upon its combustion conditions. The composition of some chemical compounds is; SiO_2 (87%), Al_2O_3 (0.15%), Fe_2O_3 (0.16%), CaO (0.5%), MgO (0.3%), SO_3 (0.2%), and carbon (5.91%). A well-burnt rice husk ash will contain about 90% of amorphous silica, 5% of carbon, and 2% of K_2O.

Table -4: Chemical properties of rice husk ash

Chemical Properties:	Rice Husk Ash:
Chemical composition	SiO_2 - 87%, Al_2O_3 - 0.15%, Fe_2O_3 - 0.16%, CaO - 0.5%, MgO - 0.3%, SO_3 - 0.2%, and carbon - 5.91%.
Chemical composition (Well burnt RHA)	Amorphous silica – 90%, Carbon – 5%, and K_2O – 2%.

Rice husk ash (RHA) is used in the production of many construction materials such as; high-performance concrete, green concrete, refractory bricks, low-cost building blocks, lightweight construction material, insulating material, ceramic glaze, roofing shingles, etc. It is also used for the production of waterproofing chemicals and oil spill absorbents and many more.

2. LITERATURE REVIEW

Abstract-I: The nano-silica is extracted from agricultural waste, rice husk when it is burnt in the muffle furnace at a temperature 650°C for about 4 hours, the amorphous silica is obtained. For pure silica, rice husk ash is titrated using H_2SO_4 solution with continuous stirring. By this method, about 98% of pure nano-silica powder is obtained. The particles with dimension 80mm and in agglomeration form are obtained as a result in SEM. XRD test showed that the obtained nano-silica in powdered form is in amorphous nature. The presence of nano-silica is indicated by FTIR spectrum.

Abstract-II: The introduction of silica extraction technique from agricultural waste has significantly increased the use of silica in industries. Rice is considered as world's most abundant and important plants and also provides carbohydrates to us. Rice husk, the waste from rice is a main agricultural waste which is a good source of silica. In this chapter, the extraction of nano-silica from rice husk ash with techniques like thermal and chemical methods is reviewed. A more sustainable method of nano-silica extraction is described.

Abstract-III: Rice husk ash is an abundant agricultural waste from rice mills and is a great source of silica. This research is a study of nano-silica extraction by burning rice husk for about 2 hours at a temperature of 700°C. This rice husk ash undergoes methods of precipitation for the production of nano-silica. This method includes refluxing of rice husk ash with 1.0N - 2.0N NaOH to get silica. The Infrared spectral (IR) test is conducted which provides the information of the presence of silanol group with hydrogen-bonds and the siloxane groups in the obtained silica. A wide bandgap value and absorption peaks located in the UV region is shown by the optical properties through UV-visible spectroscopy of the Nano silica samples. The particles size is analyzed with the values of about 13.02, 16.83 and 23.96 nm for the samples 1NSiO2, 2NSiO2, and acid leaching were obtained.

Abstract-IV: Earth's crust has a lot of components one of which is silica and it has a lot of uses in technology. It is generally found commercially as alkoxysilane compounds which has a negative impact on health. Chance to find the source of silica which is safer, cheaper, and more environmentally friendly is avoidable. But the method of isolation and application of the silica from agricultural waste is restricted. The other sources of agricultural waste are rice husk, rice straw, corn cobs, and bagasse.

Abstract-V: Waste products are used to produce silica by different extraction methods. Agricultural wastes like rice husk or straw and wheat husk or straw contains very large amount of silica. The paper concludes that silica production can be done using two methods one is thermal and another one is chemical methods which also gives directions to form silica from other agricultural waste.

3. METHODS FOR EXTRACTION OF NANO-SILICA

There are two major methods that are used for nano-silica extraction namely;

i. Chemical Treatment Methods and,

ii. Thermal Treatment Methods.

3.1. Chemical Treatment Methods:

In chemical treatment methods, pure and high quantities of nano-silica are derived from the rice husk ash using chemical techniques such techniques include alkaline extraction technique. These methods take a little bit more time, about 24 to 28 hours therefore, they are costly. The rice husk is first converted into rice husk ash for which they have to go through a thermal treatment process (mentioned above) then they have proceeded with the chemical treatment methods. This treatment can be done with the help of;

(i). Alkaline Treatment Method, and

(ii). Acid Leaching Treatment Method.

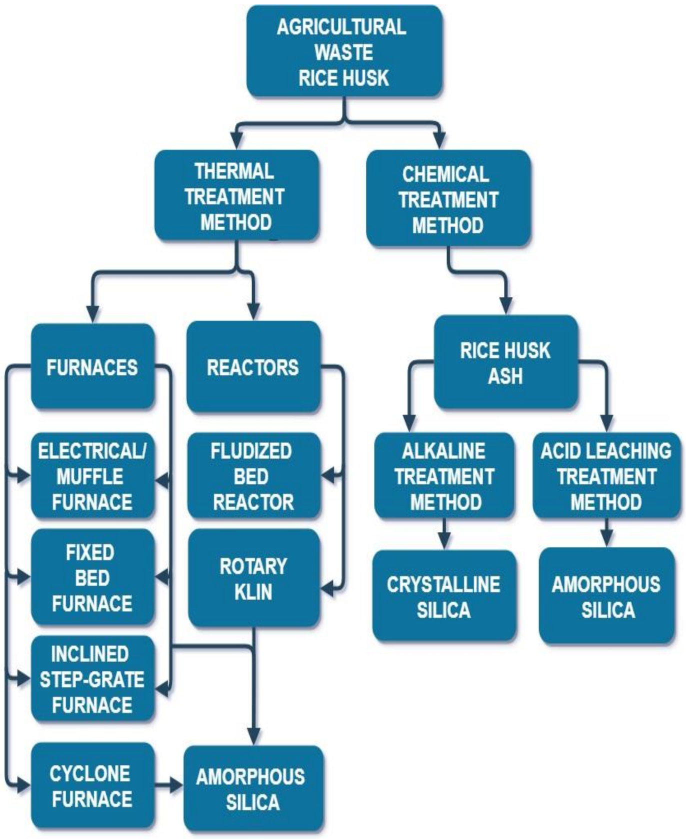

Fig -5: Methodology of preparation of silica nanoparticles from rice husk

3.1.1. Alkaline Treatment Method:

The alkaline treatment method generally includes alkaline extraction and acid neutralization is a simple and effective method for the extraction of the amorphous silica from agricultural waste rice husk. This method is used for the extraction of silica particles from rice husk ash, as it can effectively remove metallic impurities and can produce pure silica with high silica content. In this technique, rice husk ash is treated with a 2.0N to 3.0N solution of NaOH which can provide nano-silica with an approximation of 90.44%.

Advantages

(i). Simple and efficient method

(ii). It can effectively remove metallic impurities and can produce pure silica with high silica content.

(iii). Approximately 90.44% pure silica

Disadvantages

(i). Costly

(ii). High reaction rate (about 24 to 48 hours).

(iii). It requires the involvement of varying measures along with different chemicals.

3.1.2. Acid Leaching Treatment Method:

The acid leaching treatment method or acid extraction method is used to prepare nano-silica with different kinds of acidic solutions such as acetic acid, hydrochloric acid, phosphoric acid, citric acid, etc. by acid leaching the rice husk ash. High purity and high surface area of nano-silica are extracted using this technique.

Thermal treatment methods are preferred over chemical treatment methods for nano-silica extraction from rice husk ash as, these methods are costly, high reaction rate (about 24 to 48 hours), and require the involvement of varying measures along with different chemicals.

Advantages

(i). High purity of nano-silica

(ii). High surface area of nano-silica

Disadvantages

(i). Costly

(ii). High reaction rate (about 24 to 48 hours)

(iii). It requires the involvement of varying measures along with different chemicals

3.2. Thermal Treatment Methods:

Thermal treatment of rice husk is one of the initial methods for the extraction of nano-silica from rice husk. In thermal methods, the nano-silica is extracted by the heating of agricultural waste, which is rice husk in this case. This treatment can be done with the help of;

(i). Furnaces and

(ii). Reactors.

The furnace-thermal treatment methods include;

(i). Muffle/ Electric Furnace,

(ii). Fixed Bed Furnace,

(iii). Inclined Step-Grate Furnace, and

(iv). Cyclone Furnace

The reactor-thermal treatment methods include;

(i). Fluidized Bed Reactor and

(ii). Rotary Kiln

These furnaces produce silica in amorphous form. Thermal treatment methods are not generally preferred as they have certain limitations like;

(i). The reactions are time consuming

(ii). Hot spot formation

(iii). The free-flowing air required for complete oxidation of carbon is less, and many more

3.2.1. Muffle/ Electric Furnace:

In a muffle furnace, the rice husk ash is incinerated to form nano-silica. The extraction of nano-silica from agricultural waste (rice husk), at a laboratory scale is done with the help of a muffle or electric furnace. The rice husk is collected and burnt at about 500°C to 900°C for a required time period in a muffle furnace. When it is burnt at about 500°C-700°C, amorphous nano-silica is produced. While burning it at approximately 900°C will produce crystalline nano-silica. This process requires a lot of time which means that the rate of reaction is low as well as the production rate is also low. A muffle furnace keeps the heated material isolated from the contaminants of combustion which makes it an ideal choice for collecting ash samples. Chemical pre-treatment before the process is important as it avoids the unburned particles that can lead to reducing the purity of nano-silica.

Advantages

It keeps the heated material isolated from the contaminants of combustion which makes it an ideal choice for collecting ash samples.

Disadvantages

This process requires a lot of time which means that the rate of reaction is low as well as the production rate is also low.

3.2.2. Fixed Bed Furnace:

Nano-silica from rice husk can also be extracted by using a fixed bed furnace. In the furnace, rice husk is conducted at the temperature of about 600°C to 1200°C for the pyrolysis process. Burst nano size of amorphous silica is obtained as a product that will transform into crystalline silica at 1000°C. A fixed bed furnace provides an interaction between the material to be treated and the gas stream in the furnace. This interaction is more effective as compared to other furnaces also the product is uniformly finished. The major benefit of this process is the complete combustion of the carbon content. The heat loss that occurs during this process could affect the temperature and can lead to unstable silica production.

Advantages

(i). It provides an interaction between the material to be treated and the gas stream in the furnace.

(ii). This interaction is more effective as compared to other furnaces also the product is uniformly finished.

(iii). The major benefit of this process is the complete combustion of the carbon content.

Disadvantages

(i). The heat loss that occurs during this process could affect the temperature and can lead to unstable silica production.

3.2.3. Inclined Step-Grate Furnace:

The inclined step-grate furnace is mainly used for the production of rice husk ash (RHA). This process is simple in construction and process. Some of its components are; the feeding component, combustion chamber, and chamber of ash precipitation. The yield quantity of this process is low and the unburnt carbon content is high. The inlet for feed (rice husk) is provided at the top of the furnace and the air stream flows from the bottom part. Inclined step-grate is an inefficient process in the combustion and the separation of rice husk ash can result in smoke and spark that can partially draw into the dryer plenum.

Advantages

This process is simple in construction and process.

Disadvantages

(i). The yield quantity of this process is low and the unburnt carbon content is high.

(ii). It is an inefficient process in the combustion and the separation of rice husk ash can result in smoke and spark that can partially draw into the dryer plenum.

3.2.4. Cyclone Furnace:

The cyclone furnace is generally used for producing rice husk ash from rice husk. In this furnace, the air is supplied which keeps the rice husk spinning or rotating in a circular motion and accelerated combustion in the combustion chamber. The product made from this process has less carbon content. An additional preheater is given in the furnace which increases its efficiency. The waste product can be easily removed in the process.

Advantages

(i). An additional preheater is given in the furnace which increases its efficiency.

(ii). The waste product can be easily removed in the process.

Disadvantages

The product made from this process has less carbon content.

3.2.5. Fluidized Bed Reactor:

In the modern fluidized bed reactor process, less ash content pozzolanic rice husk ash is produced in the combustion chamber with fast residence time. The amorphous silica from rice husk ash is obtained with the help of fluidized bed reactors at varying temperatures and varying speeds.

Advantages

(i). Rate of reaction is high,

(ii). Uniformity in temperature,

(iii). The range of operating temperature is low,

(iv). High carbon conversion efficiency,

(v). Combustion intensity is high,

(vi). Highly effective mixing characteristics, and

(vii). The elevated reaction of gas-solid mixtures.

Disadvantages

High cost

3.2.6. Rotary Kiln:

A rotating kiln is a type of pyro-processing tool that is used to increase calcination materials in an ongoing process. For silica production, rice husk is firstly carbonized in an upstream rotary kiln and heated by electric heaters/ burners/other heat sources at a temperature of about 300°C to 400°C. This carbonized rice husk is again supplied to a second rotary kiln or rotating oven and is burnt at 600°C. These techniques produce rice husk ash very effectively. It produces white-colored rice husk ash which has an excellent chemical reactivity.

Advantages

(i). These techniques produce rice husk ash very effectively.

(ii). It produces white colored rice husk ash which has an excellent chemical reactivity.

Disadvantages

(i). Rate of reaction is high,

(ii). More energy is required, and

(iii). Some additional quantity of fuel is added to avoid the crystallization of rice husk ash.

4. PRODUCTION OF SILICA NANOPARTICLES FROM RICE HUSK BY SIMPLE CHEMICAL METHOD

The detailed methodology of the process of extraction of nano-silica using a simple chemical method. The experimental process procedure and the product analysis are well explained below;

4.1. Raw Material, Chemical and Equipment Required:

Raw Material: Rice husk.

Chemical: Distilled water, HCl, NaOH, and concentrated H_2SO_4.

Equipment: Crusher, Hot plate, Muffle furnace, Filter, and Reflux set-up.

4.2. Procedure:

(i). The raw material, rice husk, was obtained from a rice mill and washed thoroughly with distilled water to remove any adhering impurities.

(ii). The washed rice husk was air-dried at room temperature and then burnt at 973K (700°C) for about 3 hours in a muffle furnace (briefly explained in section 2.1.1.).

(iii). The obtained rice husk ash was washed with distilled water for the removal of the sand, dust, light empty grains, and fine dirt.

(iv). Hence, the repeated washing of rice husk ash removes the adhered impurities on the surface of silica.

(v). The rice husk ash was re-fluxed with 6N HCl for about one and a half hours and then filtered to remove metallic impurities and to extract pure nano-silica.

(vi). The filtered rice husk ash was thoroughly rinsed with hot water repeatedly and then boiled with 2.5N NaOH solution at 353K (80°C) for about one and a half hours while being stirred magnetically.

(vii). It was then filtered to obtain sodium silicates, after the reaction,

SiO_2 (ash) + NaOH $\rightarrow Na_2SiO_3 + H_2O$.

(viii). The obtained residue was repeatedly washed with hot water to make sure the complete extraction of sodium silicate.

(ix). The pH of the obtained sodium silicate was reduced to 2.0 using concentrated H_2SO_4 in a controlled manner.

(x). To extract the nano-silica precipitates, the chemical reaction that takes place during the above process is $Na_2SiO_3 + H_2SO_4 \rightarrow SiO_2 + Na_2SO_4 + H_2O$.

(xi). The obtained precipitate was washed thrice in warm distilled water and then filtered.

(xii). The obtained residues were sintered at 973K (700°C) and 1373K (1099.85°C) for 3 hours in a muffle furnace.

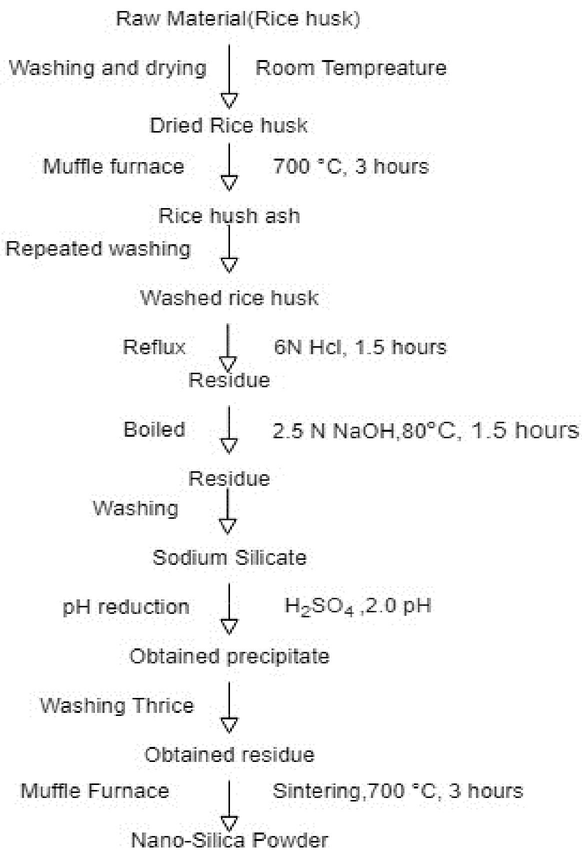

Fig -6: Preparation of Silica Nanoparticles from Rice Husk by Simple Chemical Method.

Fig -7: Washed Rice Husk

Fig -8: Rice Husk Ash (RHA)

Fig -9: RHA after HCl reflux

Fig -10: Residue burnt in muffle furnace

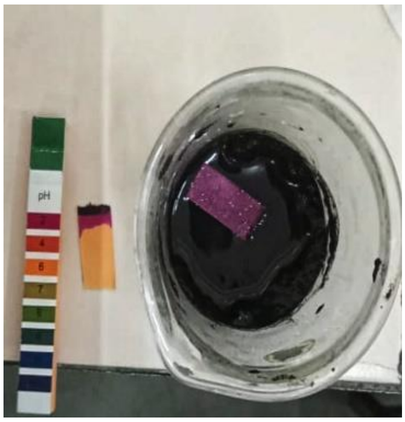

Fig -11: pH reduction to 2.0 with H_2SO_4

Fig -12: Boiled with 2.5N NaOH

5. RESULTS AND DISCUSSION

With the help of a transmission electron microscope (TEM), we can evaluate the size of nano-silica particles. The particle size of Sio2 nanoparticles of rice husk ash (RHA) that was obtained was between 20 to 40 nm range.

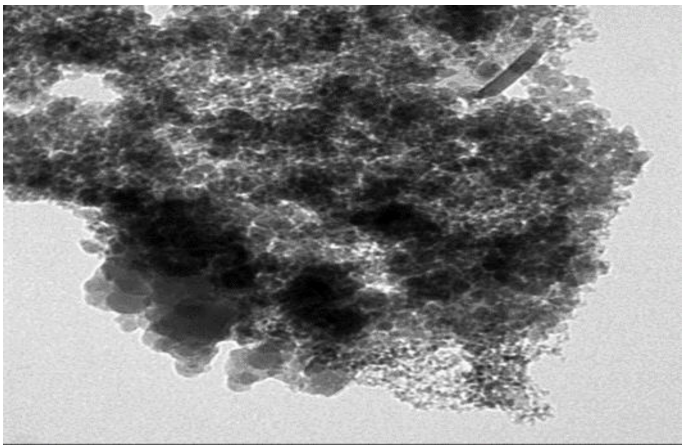

Fig -13: TEM image of Silica nanoparticles

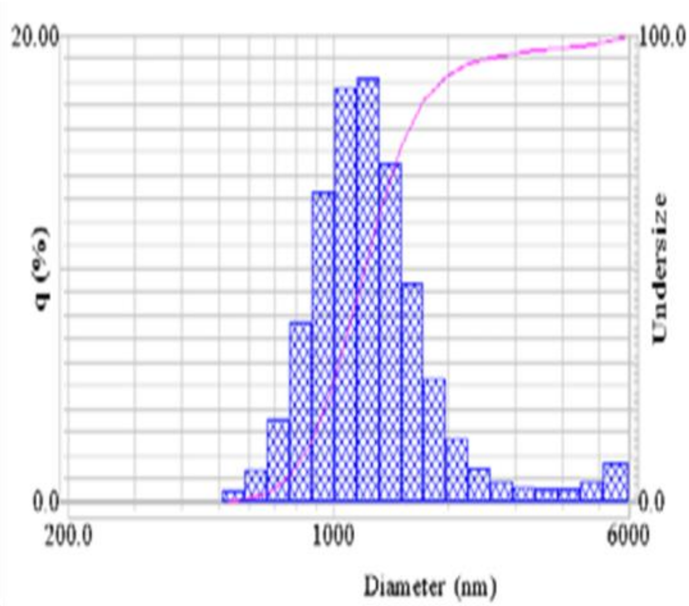

Fig -14: DLS image of Particle size distribution of Silica nanoparticles

Dynamic laser scattering (DLS) is a method that is used for finding the dynamic diameter of a particle. The size of the particle obtained from TEM is 20-40 nm, which is smaller than the size obtained from DLS. This figure provides us the information about the particle size of nano-silica, which is in the range of about 900 to1400 nm. The huge size difference between the TEM and the DLS is because silica nanoparticles are dispersed in water.

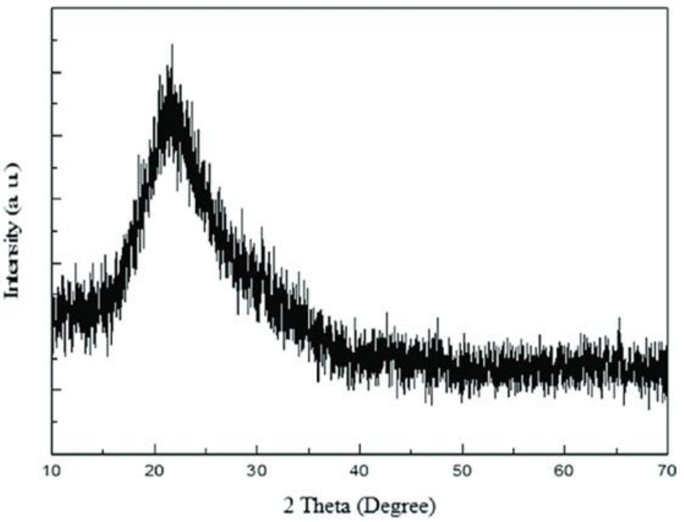

Fig -15: XRD pattern of SiO2 NPs from RHA

We use the X-ray diffraction (XRD) pattern to confirm the amorphous phase. Here in this figure, the pick at $2\theta \sim 23_0$ (approx.) confirms the amorphous phase of SiO_2 nanoparticles. The silica nanoparticle was obtained from the calcination of rice husk ash (RHA).

6. ALTERNATIVE AGRICULTURAL WASTE FOR SILICA NANOPARTICLES SYNTHESIS

As rice husk is a by-product of the rice milling industry and carries around 20% weight of the total rice produced, we used rice husk in the extraction process of nano-silica particles. On an alternate basis we can also use rice straw, corn cobs, bagasse, etc.

Rice straw is basically a stem part of a rice plant that is separated during the rice harvesting process and is therefore considered as an agricultural waste. As we know that rice husk is rich in silica, so is rice straw.

Rice straw has the highest amount of silica as compared to other plants. It contains various organic components in different proportions. The amount of ash that a certain type of rice will produce depends on which type of rice is considered, where and in what kind of atmosphere the rice is grown.

Corn cobs are basically agricultural waste products generated from corn. It contains a significant amount of silica, around 60%. It is used in silica production.

7. CONCLUSION

The waste material obtained from agriculture is rice husk which was heated in a controlled manner, then the obtained rice husk ash was re-fluxed with an acidic medium, then the residue was mixed with NaOH from there we obtained sodium silicate. To maintain the pH value of 2.0 we mixed H_2SO_4 with sodium silicate. Using the product obtained, the size of the nano-silica particles was determined by the TEM method. Dynamic diameter of nano-silica particle obtained from DLS method. And lastly, XRD confirms the amorphous phase of silica nanoparticles.

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