Recent Advance in Silica Production Technologies from Agricultural Waste Stream– Review

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***Abstract*—Substantial applications of silica materials in industrial products have caused development of silica extraction methodologies out of various waste products. Rice husk /straw and wheat husk/straw (agricultural waste) are one of the agricultural wastes which have a huge content of silicate materials. This review shows that silica production techniques by using thermal and chemical methods, and also provide directions to efficient, new, rapid microwave assisted method for silica extraction from agricultural waste stream.**

***Index Terms*—agricultural waste, nano amorphous silica, thermal treatment, chemical treatment, microwave technology**

I. Introduction

Rice husk/straw and wheat husk/straw are by products of rice and wheat milling process and they are major waste products of the agricultural industries. In recent years, agricultural products and by products (waste) attract towards on sustainable energy generation, it is considered as a potential raw material for preparing high-value products like silicon composite [1].

Silicate materials can be found in two forms amorphous and crystalline. Silica in amorphous form has huge industrial applications. It is used in rubber, plastics, cosmetic, electronics, coating, optics, refractories and also use as an abrasive agent. Silica in crystalline form is widely used in glass and ceramic industry [2].

The conventional methodology to produce amorphous silica from quartz sand requires high temperature and pressure [3]. Another method of silica production is sol-gel process but it is expensive due to high material cost.

Agricultural waste is one of the cheapest source for silica production. It contains 50%-90% of SiO_2 (Table I). The reasons which are responsible for agricultural waste to be considered as good silica source and have potential for the large scale production are [4], [5]:

1. Low cost of the raw material
2. High silica content in agricultural waste
3. Comparable silica quality
4. High energy content
5. Fine sized amorphous material

Scientist found many extraction techniques (Table II) and patents (Table III) are exist for the preparation of amorphous silica from agricultural waste stream. This paper provides an overview of the previous work and describes two main methodologies.

TABLE I. Chemical Composition of Rice Husk Ash and Wheat Straw Ash [6], [7]

Component	Rice husk ash (%)	Wheat straw ash (%)
SiO_2	80-90	50-55
CaO	0.67	10.6
Fe_2O_3	0.0	0.67
MgO	0.44	2.20
Al_2O_3	0.46	0.48
Na_2O_3	0.12	5.41
K_2O	2.91	11.4

II. Existing Methods for Preparation of Silica

Major methods for silica production from agricultural waste may be grouped into two methods (Fig. 1):

A. Thermal methods
B. Chemical methods

A. Thermal Methods

Thermal treatment includes the use of muffles furnace, fixed bed furnace, inclined step-grate furnace, cyclonic furnace, fluidized bed reactor, rotary kiln or tubular reactor and The thermal technology has drawbacks like longer reaction time, hot spot formation, lack of free-flowing air for complete oxidation of carbon etc.

1) Electric/ muffle furnace

For the extraction of silica from agricultural waste electric/ muffle furnace is used in laboratory scale. The major drawback of using this technology is long reaction time and less production rate. Patil *et al.* reported the highest amount of silica extraction from rice husk (RH) that consisted of thermal treatment at various temperatures by using electric furnace at 700°C for 6h. The product was characterized by XRD and FTIR. The XRD data indicated that the obtained silica was amorphous in nature [8]. 95.55% pure silica produced from RH ash by using acid leaching treatment followed

Manuscript received August 10, 2016; revised December 19, 2016.

doi: 10.18178/joaat.4.3.274-279

by thermal heating treatment at 600 ℃ using muffle furnace [9]. According to Bogeshwaran *et al.* silica from RH has high pozzolanic activity when it burns in muffle furnace [10]. Chen *et al.* has successfully synthesized nano-silica materials from the wheat straw by thermal treatment. Wheat straw ash was kept under the combustion at temperature 500 ℃ for 8 hours. After the combustion the obtained sample was washed with distilled water and calcined again in muffle furnace with a heating rate 10 ℃/min at 400 ℃, 500 ℃, 600 ℃ and 700 ℃. The characterization of nano amorphous silica was analyzed by X-ray diffractometry (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), energy-dispersive X-ray (EDX), the Fourier transform infrared spectrometer (FT-IR) for elemental analysis and the Brunauer–Emmett–Teller (BET) method with an N_2-adsorpmeter for surface area determination [11]. Alyosef *et al.* has reported meso /macroporous biogenic silica (3 to 1500nm) from miscanthus, cereal remnant pellets and wheat straw by using thermo chemical treatment. Wheat straw sample was leaching by 5M sulfuric acid. The ratio of wheat straw and sulfuric acid was 1:10 (gmL^{-1}). The leaching treatment was carried out with constant stirring at 1000 rpm at 353K for 24 hours. The silica ash was produced after heating treatment by furnace at different temperature and timing period [12]. Pure amorphous silica was produced by the combustion and the acid leaching treatment of rice husk. The husk was leached by HCl, H_2SO_4 and HNO_3 with various concentrations. After leaching treatment the sample of wheat husk ash placed in muffle furnace 300, 400, 500, 600 and 700 ℃ respectively for 24 hours. The study shows hydrochloric acid leaching treatment was most affective to removing metal ions rather than any other acid. Pure amorphous silica was obtained at 500-700 ℃ from acid treated wheat husk ash [13]. Yalçin *et al.* successfully produced amorphous silica from rice husk at 600°C in stainless steel tubular reactor placed in electronic laboratory scale muffle furnace for 4hrs [14].

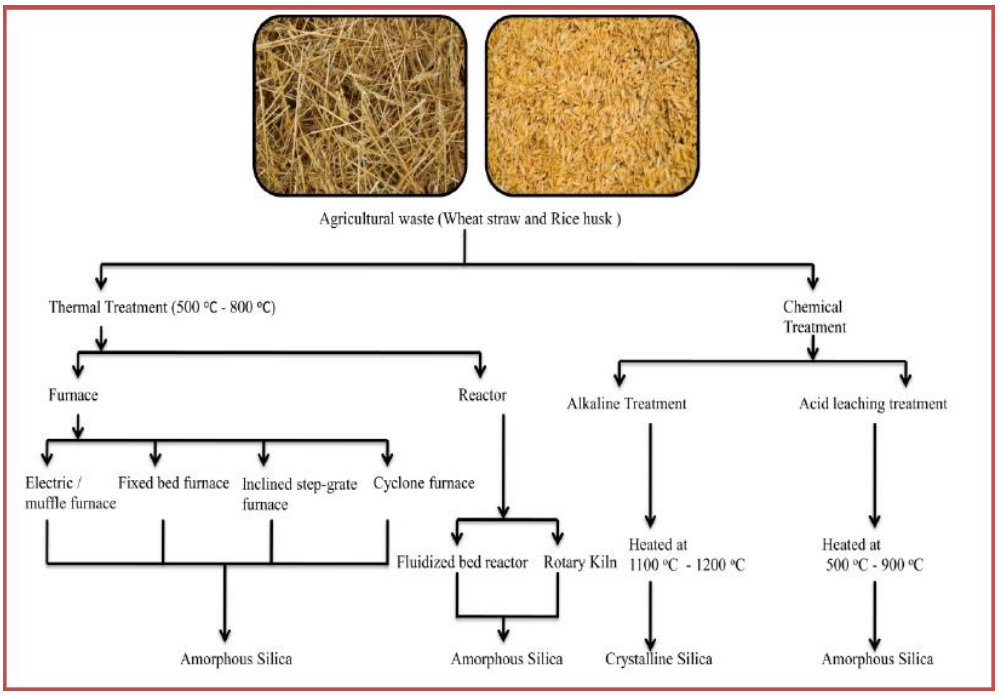

Figure 1. Different treatments, used for producing silica from agricultural waste with different structure

2) Fixed bed furnace

The production of silica from rice husk was also done by fixed bed furnace. Yang *et al.* produced burst nano sized amorphous silica by using fixed bed furnace. In this process raw and acid treated rice husk was carried out for pyrolysis at temperature 600-1200 ℃ in fixed bed furnace. The result showed that 1-10nm size amorphous silica was obtained. The amorphous silica converts in to crystalline at 1000 ℃ [15]. Hamad *et al.* successfully discovered silica from rice husk ash by using muffle furnace and fixed bed reactor at 500–1150 °[16].

3) Inclined step-grate furnace

Inclined step-grate furnace is common to use in rice husk ash production. It consists of feeding component, combustion chamber and ash precipitation chamber. The drawback of using this methodology for rice husk ash production is poor yield quality and high amount of unburnt carbon content. Rice husk is fed at the top of the grate reactor while the air flows from the bottom [17].

4) Cyclone furnace

Sing *et al.* designed cyclonic furnace. In this furnace air kept the husk rotating in circular motion and accelerated combustion in chamber. The advantage of using cyclone furnace for the production of husk ash is less carbon content product [18].

5) Fluidized bed reactor

The use of modern fluidized bed reactor process, pozzolanic RH ash was produced with less ash content with faster residence time in the combustion chamber was possible. The advantages of using fluidized bed

reactor is distribution of uniform temperature; reaction time is rapid, efficiency of carbon conversion is very high, Operating temperature range is less, there is high combustion intensity, high reaction rates of gas -solid mixtures and exceptional mixing characteristic [19], [20]. Huang *et al.* produced silica white from rice husk by using fluidized bed reactor [21]. Amorphous silica from RH is obtained by using bubbling fluidized bed pilot plant using various temperatures with various velocities. [22]. Geniva *et al.* produced silica material from RH which is product of rice milling process and are a major waste product of the agricultural industry by using Fluidized bed reactor in nitrogen atmosphere and characterized it [23]. Luan *et al.* discovered silica from the rice husk in the presence of pilot flame in a modified fluidized bed reactor. The result showed that the silica containing product with high activity was obtained [24].

TABLE II. EXTRACTION METHODOLOGIES OF SILICA FROM AGRICULTURAL WASTE

Sr. No	Technology/ Methodology	Descriptions	Advantages	Disadvantages	Ref.
1.	Electric/muffle Furnace	Amorphous Silica with specific surface area could be produced from rice husk after thermal treatment at 700 ℃ for 6 hours in air in an electric/ muffle furnace.	Simple operation	Poor yield, lower efficiency	[25]
2.	Inclined step-grate furnace	At the top of the grate furnace rice husk was fed while the air flowed from the bottom.	Simple, low cost maintenance	High required labor	[17]
3.	Cyclone furnace	Amorphous silica with a 12 – 14 wt% residual carbon produced from rice husk was burnt at a temperature of less than 700 ℃ by using cyclone furnace.	Automatic and continuous operating	High investment cost	[26]
4.	Rotary Kiln	For the silica production, rice husk was carbonized in a first rotary kiln and heated at 300 – 400 ℃, after charged into a second rotary kiln heated at 600 ℃.	Semi - automatic operating	Not developed for the higher capacity yet	[19]
5.	Alkaline extraction	NaOH solution was added in rice husk ash and this mixture was refluxed in a covered Erlenmeyer flask for 1 hour with constant stirring. The solution was filtered through ashless filter paper and the residue (carbon) was removed. The filtrate formed was sodium silicate solution, which was cooled to room temperature; Silica in gel form was produced after titration with acid (HCl or H_2SO_4) with constant stirring until pH 7. The solution was aged for 18 hours at room temperature to form gel (silica).	Efficient and simple method, good yield quality.	Expensive, longer reaction time requires, various steps with the use of various types of chemicals.	[27]

6) Rotary kiln

A rotary kiln is a pyro processing device used to raise materials for calcination in a continuous process. Active rice husk ash produced from rotary kiln was patented by 'Sugita' [28]. In this process rice husk was carbonized by upstream rotary kiln which is heated by electrical heater, burner or other heat sources at 300-400 ℃. After carbonization period, carbonized rice husk is fed in rotary kiln and burn at 600 ℃. The husk ash was successfully produced through these methods. The disadvantage of using this method was need to auxiliary fuel to prevent crystallization of ash, reaction time was very longer, high energy required etc.

B. Chemical Methods

Chemical methods include alkaline extraction method used to produce pure and high amount of silica but it is expensive because of significant longer reaction time (24-48 hrs), and also requires various steps with the use of various types of chemicals.

1) Alkaline extraction followed by acid neutralization

Alkaline extraction followed by acid neutralization method is very efficient and simple method to extract amorphous silica from agricultural waste (Fig. 2), Kalapathy *et al.* developed pure silica from rice husk ash with minimal mineral contaminants by using alkaline extraction followed by acid precipitation. In this procedure RH ash is mixed in 1N NaOH solution in 250 ml Erlenmeyer flask and is allowed to boil for 1h with constant stirring to dissolve silica and make sodium silicate solution. Sodium silicate solution titrated with 1N hydrochloric acid (HCl) solution for 18 h at 7.0 pH with constant stirring, Pure silica in gel form obtained after stirring period [29]. The Humidity Indicating Silica Gel from Rice Husk Ash obtained by based on alkaline extraction followed by acid precipitation produce by Nayak *et al.* [30]. Selvakumar *et al.* successfully produced silica from RH ash. In this study, RH ash was pretreatment by various acids (1, 3, 5 or 7 using 6 N HCl, HNO_3, H_2SO_4). Nano structured amorphous silica from RH ash was produce by alkaline extraction method used 2.0, 2.5 and 3.0 N NaOH solutions. The study revealed that treatment of 2.5 N NaOH resulted 90.44% silica content in RHA. [31].

Rungrodnimitchai *et al.* developed silicate materials from RH ash using microwave heat by using 2.0 M sodium hydroxide at microwave power of 800W for 10 minutes. The silicate material was produced after acid neutralization process [32]. Huq *et al.* produced silica from RH ash by NaOH solution with reflux condition for variable time period [33].

TABLE III. SILICA EXTRACTION PROCEDURES FROM WASTE AGRICULTURAL STREAM (PATENTS)

Patents Refrences	Descriptions	Ref No.
Chen *et al.* 2010	Nano-SiO_2 (40nm) from bio waste straw with high purity more than 99.7% obtained by using chemical method.	[34]
Tutsek *et al.* 1997	Method of producing low-carbon, white husk ash, Produce crystalline SiO_2 by using thermal treatment.	[35]
Turkay *et al.* 2007	Magnesium silicate produced from rice hull ash using NaOH alkaline extraction process. Magnesium sulfate added in to extract solution for the production of magnesium silicate.	[36]
Mukunda *et al.* 2004	A novel process and apparatus for the manufacture of precipitated silica from rice husk ash by using Alkaline extraction process.	[37]
Amick *et al.* 1980	Solar cell-grade silicon production from rice hulls by using acid treatment followed by thermal treatment.	[38]
Culter I. 1973	Production of silicon carbide from rice hulls by using thermal treatment at the temperature below 1700 ℃	[39]

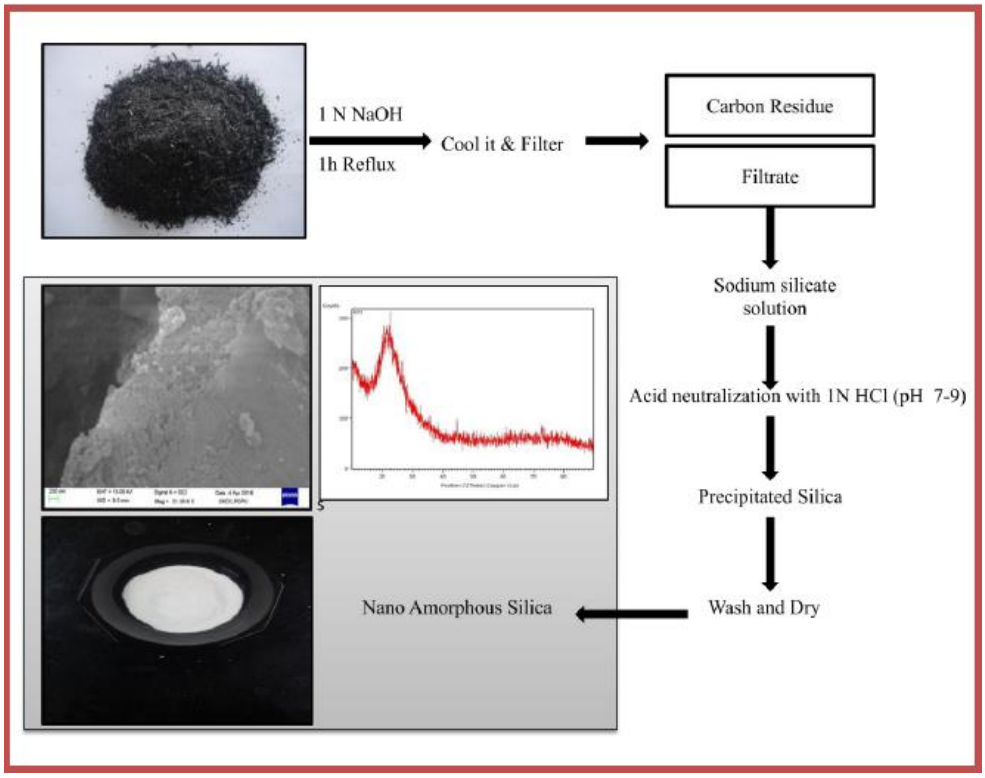

Figure 2. Extraction method for nano amorphous silica production from wheat straw.

The reaction mechanism of RH ash mixed with NaOH solution was:

$$SiO_2 + 2\,NaOH \longrightarrow Na_2SiO_3 + H_2O$$

(White Ash) (Sodium hydroxide) (Sodium silicate) (Water)

Silica was precipitated by acid neutralization from sodium-silicate [40].

$$Na_2SiO_3 + HCl \longrightarrow SiO_2 + NaCl + H_2O$$

(Sodium silicate) (Hydrochloric acid) (Silica (gel form)) (Sodium chloride) (Water)

Silica with low surface area microsphere can be successfully obtained from wheat husk ash with alkaline and acid precipitation process. After alkaline extraction process

with NaOH, it was nano amorphous silica (227nm) with specific surface area 8.23m^2/g was obtained [41] Mohamed *et al.* successfully synthesized Zeolite Y in sodium form (NaY) using silica source from rice husk ash [42]. Nano particles of silica were obtained from Vietnamese rice husk by using alkaline sol gel method by using NaOH. Silica was precipitated by using H_2SO_4 /water/butanol at pH-4. Nano silica particles with high specific surface area were successfully obtained by using method [40]. Awizar *et al.* produced nano silica by alkali ne extraction process and used it as a green corrosion inhibitor [43]. Adam *et al.* produced spherical nano sizes silica from rice husk by using Nitric acid (65%) and sodium hydroxide. In this treatment no calcination was required for the ash formation [44].

2) Other chemical extraction methods of silica from agricultural waste

Many other chemical treatments to produced silica material from bio waste. Hu *et al.* produced amorphous silica and activated carbon by using three efficient

processes toluene/ethanol, $NaClO_2$, and KOH. The calcination process was used to produce nano sphere amorphous silica (100-120nm) [45].

Amorphous silica production was done by using organic acid leaching treatment rather than using strong acid. [46]. Ionic liquid was also used for silica production from agricultural waste according to Chen *et al*. [47].

3) Microwave assisted reaction

One of the methods used by authors, for extraction of silica from wheat straw was trough microwave assisted reaction. Microwave assisted process gave faster reaction rate of gel production. Analysis revealed that product amorphous and particle size between 100-200 nm.

III. Applications

Silica in elemental form is used as a component for building material. Silica in amorphous form used as absorbent, catalyst, refining agent etc. In its vitreous (glass like) form, it is used in glass wires and optical element production. The other applications include the production of various silicate base materials like silicon carbide, soluble silicates, zeolite, silicon alloy etc. It is also used in ceramic, glass, rubber and refractory industries. It can also found used as a green corrosion inhibitor to prevent metal corrosion. Its use can be in cosmetic products as, thickening agent and an absorbent In toothpaste amorphous silica is used as an abrasive agent. In the production of inks silica is used as matting and thickening agent, silica in form of zeolite can be used as a catalyst in organo chemical synthesis [48].

IV. Conclusion

Due to a high demand of silica and silicate materials in industrial applications, it is necessary to discover new sources of production. Agricultural waste is one of the cheaper sources of production of silica. The purpose of this review was to introduce silica preparation from agricultural waste stream through thermal method and chemical extraction method. The alkaline extraction followed by acid neutralization treatment from ash by using microwave assisted process was found to be cost effective, rapid, easy and an economically viable method compared to other methods. This methodology can be used in future for production of pure, nano sized and high amount of silica from agricultural waste.

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Eco-friendly Extreme Pressure Lubricants for Water based Drilling Fluids' by Rakshith R. Shettigar, Nirendra M. Misra, Bhaskar Naik, Khushbu Patel; an International conference on Environment, Chemistry and Biology held on 19-21 November 2015 at Auckland, New Zealand.

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