Production and Characterization of Silica from Rice Husk: An Updated Review

ABSTRACT

Rice is regarded as one of the most valuable sources which is consumed by almost fifty percent of the world's population. Rice husk ash is a by-product substance obtained from the rice husk by thermal and chemical treatment. The non-crystalline nature of silica content in rice husk, which is converted to crystalline silica such as quartz, tridymite, and cristobalite on thermal treatment. It has been an important material which impacts not only enhances the betterment of industrialization but also protects the environment from pollution. The produced silica was characterized by various analyses such as Fourier transform infrared spectroscopy (FTIR), X-ray diffraction analysis (XRD), Scanning electron microscope (SEM), Transmission electron microscope (TEM), and Thermal analysis. This review is given attention to integrating and investigating processing, properties, characterization, reactivity, and application of rice husk (RH) and rice husk silica (RHS).

Keywords: Rice husk, Silica, Production, Characterization, Application

1. INTRODUCTION

Rice husk is an agricultural waste residue and a fibrous material extensively available in rice-producing countries. They are composite compounds of silica with organic materials and natural capsules that form on rice grains during the process of growing[1], [2]. Rice husk is an extensive by-product of rice processing industries and is used as fuel and animal feed in rural areas and used as animal feed[3], [4]. The RH is the commercially usable raw material for obtaining silica gels and powders [5]. RH is a fascinating source of decent levels of high quality and quantity of silica which has found much use in various purposes of applications such as adsorbent,[6] zeolites[7]– [11], aerogels[12], etc. The formation of RHA from RH and a high amount of hydrated amorphous SiO_2 and less amount of carbon is obtained. The components which are depending on the rice variety, particle size, furnace type, burning condition, climate change, soil types, fertilizers, and geographical area [13], [14]. The amorphous and crystalline silica has a great impact on the industry. Amorphous silica is used as a raw material to obtain plastics, cosmetics, coatings, rubber, optics, refractories, and electronics. In the glass and ceramic industry, crystalline silica is applied heavily [15]. At a lower temperature on rice husks, the amorphous nature of silica such as ß-quartz (573-870°C) was obtained. At higher temperatures, the amorphous silica converted to crystalline silica such as cristobalite (1470-1710°C) and tridymite (870-1470°C) [16]– [18]. In presence of a polar solvent, the shape of silica particles was well-ordered and spherical but when two different polar solvents were used, then no remarkable difference in the size of the silica particle [19]. Silica (SiO_2) is one of the valuable inorganic multiuse chemical compounds. It can exist in gel, crystalline, and amorphous forms. It is the most abundant material on the earth's crust [20]. The nature of amorphous silica is extremely permeable in its structure. The reactivity of the rice husk silica depends on the structure but not on the size of the particle. The reactivity of fume silica is lower than porous amorphous silica [21]. The amorphous silica formed as opal and semiprecious stone in the rice husk, but the crystalline silica existed in three phases such as quartz, tridymite, and cristobalite [22]. The crystalline structure of silica was obtained by thermal treatment [23]. The chemical structure of silica is shown in Fig. 1.

Fig. 1. Structure of Silica

In the countryside area of Bangladesh, RH is utilized as a source of energy for the boiling process. It is not an expensive source of energy for those purposes. When RH is burnt for generation, then the combustion product is not only a waste material but also provides an environmental problem. The main goal of the review is to extract silica from RHA and investigate its properties. This process plays an important role in producing silica powder at a very low cost and reducing disposal as well as pollution to protect the environment.

2. METHODS FOR PRODUCTION OF SILICA

2.1 Thermal method

In order to produce silica from agronomy-based waste residue, thermal treatment is necessary. Rice husk is transferred into RHA by the thermal treatment process. The RHA was used as the starting material for the extraction of silica [24]. To produce RHA, various thermal treatments such as muffle/electric furnaces, cyclonic furnaces, fixed bed furnaces, inclined step-grate furnaces, rotary kilns, and Fluidized bed reactors were used [25]. Seitkhanazat et al. have extracted silica materials from RH by thermal heating treatment. The RH was heated in a muffle furnace at 600°C for 4 hours [26]. It was reported that the high crystalline silica was produced from RH in a muffle furnace at 900°C for 6 hours [27]. Ghorbani et al. have synthesized amorphous silica from RH using acid leaching treatment in a muffle furnace at 600°C[28]. V.P. Della et al. have extracted amorphous silica with a heating rate of 10^{0}C/min in an electric furnace at different temperatures such as 400,500,600 or 700C for 1,3 or 6 hours [29]. The different methods carried out for the production of silica are shown in Table 1.

Table 1. Different methods for the production of silica

Serial No.	Methodology	Description	References

1	Muffle furnace	Amorphous silica is produced from RHA with thermal treatment at 500°C for 5 hr. and crystalline silica at 1100-1200°C.	[30]
2	Fluidized bed	A high energy content of silica obtained in rice husk ash by rapid analytical method and dense phase zone was greater than the precipitated form of silica.	[31]
3	Muffle furnace	Various size of crystalline silica was observed at different temperature 400-1200°C.	[32]
4	Electric furnace	Silica powder produced from the rice husk at 700°C at a heating rate of 5C$^{\circ}$/min	[33]
5	Muffle furnace	The Spherical size of amorphous silica consists of Si and O with a large surface area from the rice husk at the temperature of 700°C.	[34]
6	Muffle stove	The amorphous nature of silica with high purity and surface area by the acid leaching at 600°C for 3 hours.	[35]
7	Fluidized bed	The high-quality amorphous silica was extracted from rice husk ash under different temperatures with velocity.	[36]

The drawbacks of thermal treatment of RH to produce high-quality silica [37].

1. A huge amount of energy was consumed.
2. The production rate of rice husks silica was lower’
3. Longer reaction time.
4. Batch-like process
5. Sometimes explosions occur.

2.2 Chemical Method

In the chemical method, a strong acid was used on RH to eliminate organics and metallic impurities. Strong acids such as hydrochloric acid (HCl), Sulfuric acid (H_2SO_4), and nitric acid (HNO_3) were utilized in the leaching treatment [38]. S. Chandrasekhar et al. successfully produced silica from RHA using organic acid treatments such as acetic, oxalic, and inorganic acids such as hydrochloric and nitric acids of different concentrations [39]. Jinyoung Chun et al. extracted mesoporous silica from RH. In this procedure, the impurities of RH were removed using deionized water and dried in an oven. The dried RH was mixed with H_2SO_4(72%) for 1 hour. After adding water to the solution, the concentration of sulfuric acid converted to 4% at 121°C for 1 hour, and the separating sulfuric acid from the solution was washed with water and heated at 80°C for 24 hr. The white silica was obtained during RHA was burned at 900°C [40]. Dongmin An et al. derived silica powders from RH using hydrochloric acid (HCl). RHA was mixed with HCl in thermostat for a half an hour and washed with distilled water until acid-free solution and then it was dried at 120°C [41]. Farook Adam et al. produced silica from rice husks using nitric acid by the sol-gel technique [42]. V.B. Carmona et al. derived silica from RH by acid leaching process. In the procedure, the amount of RH was dissolved in an acid solution, then the solution was mixed with distilled water at high pressure and temperature for 1 hours [43]. Since Song et al. synthesized nano-silica from rice husk ash by the alkaline extraction method. In this procedure, about 300-900 mg RHA was added in 9 ml of 1.5 M sodium hydroxide, NaOH in a beaker at 90°C for 1 hours. The RHA was transformed into sodium silicate solution in NaOH then the solution was heated and diluted until 1M concentration. Sodium silicate solution was titrated with 1M hydrochloric acid (HCl) at 7.0 pH with constant stirring and the solution mixture was enveloped and placed in an oven at 70°C to stage for 1–7 days then silica nanoparticle was obtained [44]. Panasenko et al. produced silica from RHA using 1M NaOH by alkaline extraction technique (Scheme 1). In the initial step RH was converted into RHA, Then Sodium hydroxide solution was mixed with RHA and boiled for 5 hours in a round-bottom flask equipped with a condenser in a hemispherical heating mantle to dissolve the silica and to obtain a sodium silicate solution [45, 46]. In the second step, the silica was precipitated when an acid solution was added to dissolve the sodium silicate solution [47]. Table 2 shows the composition of rice husk varies with geological location.

Scheme 1. Reaction for the Production of Silica from Rice Husk Ash

Table 2.The composition of rice husks varies with geological location

Component	Rice husk ash %	Rice husk ash %	Rice husk ash%
SiO_2	94.60	76.66	81.4
CaO	0.40	1.07	1.60
Al_2O_3	0.30	0.07	1.80
Fe_2O_3	0.30	0.04	1.01
Na_2O	0.20	0.29	0.16
K_2O	1.30	2.47	2.35
MnO	-	0.13	-
TiO_2	-	0.01	-
MgO	0.30	0.65	2.25
P_2O_3	-	0.86	5.26
Others	2.60	17.75	4.17
References	[48]	[3]	[49]

The acid-leaching process was very hazardous to the environment, and humans and caused materials corrosion. The chemical method has been an economical problem due to utilizing of expansive materials and disposal treatment of used strong acids [50].

3. PROPERTIES AND CHARACTERIZATION

The development of many advanced instruments in the field of material characterization has facilitated structural studies of RH and its thermally produced products. Nowadays, many researchers all over the world have utilized sophisticated techniques such as IR, FTIR, XRD, SEM, TEM, TGA, DTA, and DSC[51].

3.1 Fourier Transform Infrared (FTIR) Analysis of Silica

The RHA with powder samples of interest has interacted with infrared spectroscopic analysis. These analyses were executed with the Fourier Transform Infrared Spectrometer in the range 400-4000 cm^{-1}.

The considerable amount of silica particles was mixed properly with KBr and continued to the sample cup of the diffuse Reflectance Accessory for scanning in the spectrum. A. Ananth et al. were used to identify functional groups in RHA and RHS by FTIR spectroscopic instrument. The range of isolated and separate silanol groups of Si-OH was indicated in the interval of 3670-3000 $cm^{-1}$and the intensity peaks of Si-O-Si in between 1200-1050 cm^{-1}[52]. D. An et al. reported that FTIR spectroscopic instrument is employed to recognize functional groups of produced silica. The appeared intensity peaks of stretching and bending mode of adsorbed water were at 3416 and 1639 cm^{-1} respectively. The obtaining peaks of different modes of SiO_2 such as asymmetric, symmetric, and bending were 1093, 788, and 466 cm^{-1} respectively [53]. Vlaev et al. observed different functional groups exist in RHA such as silanol Si-OH and siloxane Si-O-Si. The IR spectra of RH gave typical bonds of Si–O–Si stretching (very strong at 1096 cm^{-1} and strong at 798 cm^{-1}) and bending vibrations (very strong at 466 cm^{-1}). The intensity bands Si-OH vibrations at 3437 and 1633 cm^{-1} respectively [54]. Yan Liu et al. characterized silica and chemical groups were determined by FTIR spectrometer. The appearing absorbance peak of asymmetry stretching vibration (Si-O-Si) was 1105 cm^{-1} and symmetry stretching and bending vibration were at 797 cm^{-1} and 472 cm^{-1} respectively. The absorbance peak of silanol groups at 3363 cm^{-1} and 951 cm^{-1} for asymmetry stretching vibration and bending vibration respectively [55]. Table 3 shows the FTIR data for rice husk ash obtained from different sources.

Table 3. FTIR Data for Rice Husk Ash from Different Sources

Component	Silanol OH/cm^{-1}	Siloxane bonds (Si-O-Si)/cm^{-1}	References
Rice husk ash	3460	1039	[55]
	3426	1092	[56]
	3493	1091	[57]
	3431	937	[58]
	3429	1101	[59]
	3400	997	[60]
	3400	850-950	[61]

3.2 X-Ray Diffraction (XRD) Analysis of Silica

X-ray diffraction analysis is used to identify whether the obtained silica was crystalline or amorphous at a definite temperature [62]. Crystalline growth during heat treatment and silica ash production from RH and it has been studied by Ibrahim and Helmy using X-ray diffraction (XRD). It was shown that the nuclei of disordered cristobalite were present in the silica-containing ash. The X-ray diffraction pattern of RHS was determined using Bruker D8 Discovery X-ray Diffractometer by Rohani Abu Bakar et al. The maximum intensity of silica has appeared at 2θ = 22^{o} which indicates the amorphous nature of silica below temperature 900°C. After increasing temperature, amorphous silica converted to the crystallization phase [63]. Jan-Jezreel F. Saceda et al. reported that the crystallinity of RHS was observed by XRD pattern. At the higher temperature, the observed peaks of 2θ values of 20.9, 21.9, 26.6, 31.4, and 36.0° which indicate silica in crystalline form, and the interplanar spacing d values of 4.06, 3.35, 2.85, and 2.49 by Bragg's equation. The d values data indicated that These were tridymite and cristobalite reflections [10].Qudratun et al. reported that the amorphous and crystalline nature of silica was produced. The observed peaks of XRD 2ϴ angle of 22° with an intensity that stipulated for amorphous at 800°C and crystalline at 900°C [64]. Siti Haslina et al. characterized silica and compared it to commercial fumed silica. The intensity peaks of RHA and fumed silica of 2θ values at 15-30°C that indicating amorphous in nature [66]. The crystallinity of rice husk silica at different temperatures shown in Table 4.

Table 4. Crystallinity of Rice Husk Silica at Different Temperatures

Component	2θ (theta)/˚	Temperature/˚C	Crystal's characteristics	References
Rice husk silica	22	900	Crystalline	[66]
	36.541	700	Amorphous	[67]
	16-72	1000 – 1400	Crystalline	[68]
	18-30	850-900	Amorphous	[69]
	10-90	800	Amorphous	[70]
	22	700	Amorphous	[71]

3.3 Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) is a virtual technology that is used worldwide in the field of research. It's regarded as an operative method in the analysis of organic and inorganic materials and those materials converted nanometer(nm) to micrometer(µm) scale [72]. R.V. Krishnarao et al. obtained a morphological change of RHS as a consequence of heat. During crystallization at 1200˚C, the size of RHA converted from 50 to 20 µm and smooth surface area [73]. Ghassan Abood Habeeb et al. reported that RH samples were examined by scanning electron microscopy and obtained high surface area with multilayered, angular, microporous, and grey colors. When RHA was performed with the LA machine then the particle size minimized from 63.8 to 11.5 µm for a grinding time of 90 to 360 minutes respectively [74]. The porous cellular structure and irregularly shaped particles of RHS originated with SEM micrographs observedby Le Anh-Tuan Bui et al. They reported that the particle size of RHA could be converted to an 11-12 µm range in diameter [75]. Rosario Madrid et al. synthesized amorphous silica with a porous and high surface area which was characterized by SEM micrographs. The particle size of the RHA sample was observed at 30 µm [76]. R.M. Mohamed et al. synthesized zeolite NaY and NaP from RH. The uniform size of amorphous crystals NaY was produced through a two-step route and the particle size was 4 µm which was characterized by SEM analysis. After crystallization for 48h, the NaY crystal was transformed to NaP. The main drawback of SEM analysis was that it was not justified the shape of NaP crystals due to most of the crystals having been cracked through the center [9]. Tzong-Horng Liou et al. characterized nano-silica and the particle size was changed with pH which was determined by scanning electron microscopy. At pH 3, the size of an irregular-shaped silica powder of 50-60 µm converted to nano size and spherical silica with 5-10 nm [77]. To perform different treatments on RH, Iara Janaína Fernandes et al. identified silica from RHA samples by scanning electron microscopy (SEM). The observed shape of the silica particle was irregular and size below 10 µm. The chemical composition of silica was altered by different pretreatments, but the shape and size were not changed [78]. The shape and size of nano-silica particles were characterized by SEM and the shape of silica particles was influenced by the solvent. When there was no solvent/nonpolar used, then those were irregular, and with a polar solvent, the particles were spherical and well-ordered [79].

3.4 Transmission Electron Microscopy (TEM)

Transmission electron microscopes (TEM) are versatile techniques in which a particle beam of electrons is transmitted through an ultra-thin specimen. When the interaction of the electron with the specimen, then produced a highly magnified image.Kasim Mohammed Hello et al. synthesized solid ammonium sulfate using RHA and a rod-like structure was attained by the

TEM micrographs, also the determination of length is about various millimeters (mm) and the range of diameter of micro rods is 0.1 μm to 0.26 μm [81]. Noor Sheeraz et al. synthesized different silica particles from RH and analyzed them by the TEM micrographs. The accumulated and irregular shape of silica particles was obtained without ethanol and the Spherical and scattered particles appeared in the presence of ethanol. When the amount of ethanol was increasing then more scattered particles were observed and upgraded the formation of Si-OH and Si-O-Si more spherical [79].

3.5 Thermal Analysis

Eduardo J. Nassar et al produced functionalized silica from RHA by sol-gel method and analyzed silica components by thermal analysis. In this procedure, at the temperature range of 60-80°C, the weight of the sample decreased, and then a weight-loss water molecule was absorbed on the silica surface. In the range of 250-460°C, the decomposition of organic part and -OH groups on the surface. At above 400°C temperature, there was no loss of weight of the sample. In differential thermal analysis, two exothermic peaks were obtained the first peak was observed between 250°C and 280°C, with a maximum of 270°C, and the second peak was observed between 350°C and 460°C, with a maximum of 380°C. This data indicated that a non-crystalline polymer was obtained [82]. Mohammed Sabah Ali et al. characterized silica particles of RHA. In the thermal analysis of RHS, 6.8% weight loss was observed in the thermogravimetric analysis. The organic materials such as carbon and unburnt elements were separated at around 200 to 650°C. The highest weight loss has occurred at 300°C. This result was assigned to the presence of ceramic oxide [83]. Addis Lemessa Jembere et al. synthesized silica powder from RHA and determined the thermal stability of RHA and RHS by Differential Scanning calorimetry (DSC). The data reflected that the major peaks of the melting point of RHA and RHS were 462.31°C and 499.7°C respectively. During the extraction, the melting point of RHS was reduced due to the presence of impurities. The observation of two enthalpies of RHS was changed at 215°C and 250°C due to the loss of organic materials from the sample [84]. Yanping Zou and Tiankui Yang analyzed acid-leached and untreated RH by Thermogravimetric process (TG). In the analysis, the temperature range at 50-150°C, and the moisture was removed from RH. The volatile matter was removed from RH at the temperature of 240-360°C. The thermal stability of acid leaching of RH was smaller than unleached RH [85]. Azadeh Tadjarodi et al. characterized nano-porous silica by thermal analysis such as TGA, DTA, and DSC. In the TG curve, the first weight loss indicated that the water molecule was removed up to 100°C temperature and the second weight loss indicated that a polycondensation reaction occurred and organic groups were removed also due to oxidation reaction. In DTA curves there are two exothermic peaks were obtained at 370°C and 680°C respectively [86].

4. color

Rice husk is converted into RHA by the thermal and chemical treatment process. When introducing heat to RH, various colors of ash were obtained. The color change was associated with the total combustion process. The structure of RHS was converted into different phases. The white color of the ash was obtained due to the complete oxidation process. The white color of the ash indicated the amorphous nature of silica. A strong interaction between potassium and silica produced potassium polyciliate which was conjugated to carbon obtaining grey color ash. The grey color of the ash indicated the transition state of silica. During the acid leaching process, acid was treated with potassium polyciliate, Then the potassium ion as well as the grey color was removed from the ash. At higher temperatures, the transition phase of silica was converted to crystalline silica such as cristobalite and tridymite with pink color [88]– [90].

5. Application

Rice husk silica has been playing an important role in almost every field of life even a century after its discovery. In the earth's crust, silica is the amplest oxide yet despite this abundance, silica is predominantly made by synthetic means for its use in technological applications and it is one of the most relevant inorganic various chemical compounds [90], [91]. Nowadays, It has been dealing with nano silica has expanded significantly and includes various aspects in industries, catalysis,[92], [93], pharmaceutics, electronics, dental materials, ceramics [94], optical element production, chromatography[95], vegetable oil refining[96], and others [97]. The extracted silica materials are fascinating for biomedical, industrial, and environmental applications [4].

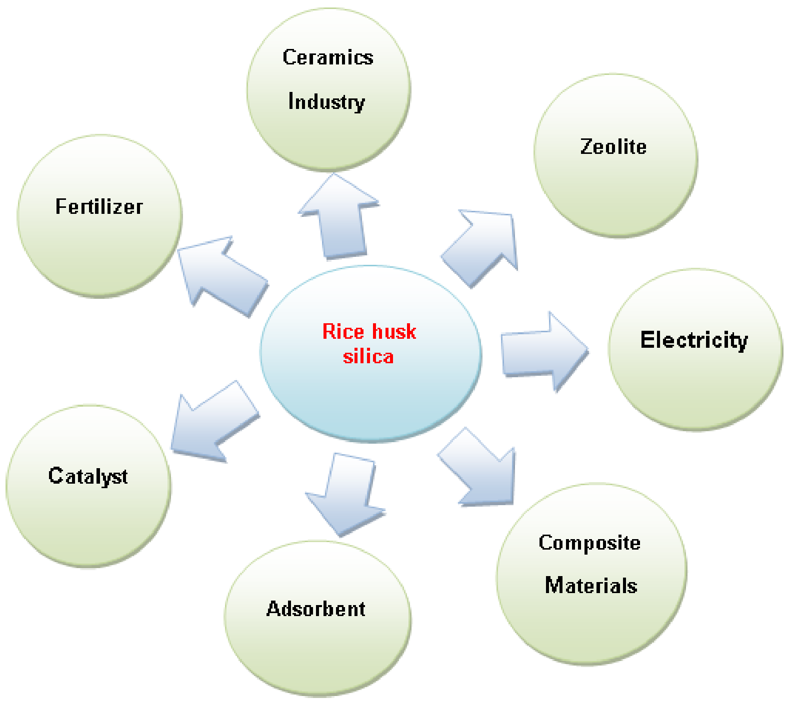

Fig 2.Application of Rice husk silica

The precipitated silica was applied in many industries such as cosmetics, tire industry, reinforcing agents in rubber, thickening agents in paints, and anti-caking agents in the food industry [98]. Several silicate-based substances such as zeolite, silicon carbide, silicon alloy, and soluble silicates from RH are applicable for different purposes [99]. RHA is a potential agro-waste residue that is used as fertilizer[100], and amorphous silica was utilized by plants and could not be absorbed as a crystalline form of silica [101]. RH as raw materials to produce electricity systematically to enhance local income and protect the environment [103]– [105]. In the presence of H_2O_2, the oxidation of benzene in acetonitrile solvent produces hydroquinone and 1,4-benzoquinone. In this reaction, the Cu–Ce silica plays an important role as the catalyst [94]. The oxidation of benzene with Cu-Ce silica catalyst is presented in Scheme 2.

Scheme 2. Oxidation of Benzene with Cu-Ce Silica Catalyst

Rice husk silica is used as composites and semiconductor materials [105], [106]. Silicon-carbide-reinforced composites have greater advantages than ceramic matrix composites [107]. Rice husk silica is a very pozzolanic characteristic that is used in cement industry. The cost-effective and high-grade cement was obtained due to the existence of RHA in cement [108, 109].

4. CONCLUSION

Now a day, the development of new products, waste management, and by-products has been playing a great role in interest. RH is one of the prospective agro residues which can be utilized as very cheap raw materials. It is regarded as a sustainable silica source to produce high quantities of amorphous and crystalline silica. In industrial applications, silica and silica materials are highly essential. The containing high amount of silica makes it an important raw material used in various industries like molecular sieve, zeolite, electronics, cement, absorbent, polymer industries, etc. The review highlighted that thermal and chemical treatment for production of silica and techniques such as FTIR, XRD, SEM, TEM, TGA, DTA, and DSC are used to characterize silica.

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