

Synthesis of Silica and Silica Compounds Based on Rice Husk Ash: Article Review

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Abstract: Rice husk (RH) is an agricultural solid waste residue. It is a byproduct in rice mills and creates disposal and pollution problems. Burning of RH in ambient atmosphere causes toxic air pollution also dumping causing a damage to the land and the surrounding area in which it is dumped. Therefore RH and rice husk ash (RHA) must be appropriately managed. In the present article synthesis methods for silica from RHA are reviewed. In addition basic properties, structure and uses in various industries of RH, RHA and silica have been lighted.

Keywords: Rice Husk, Ash, Silica, Leaching, Calcination, Alkali method.

1 Introduction

Rice cultivation covers about 1% of the earth's surface. More than half of the world's population depends on rice as a staple food and it ranks second to wheat in terms of cultivation area and production [1,2]. The rough rice is called paddy. Rice production continents are Asia, South and North America, Africa and Europe. The global production of rice in the world is close to 600 million tons per year [3]. China and India contribute 33% and 22% of global rice production respectively. An average of 20% of rice paddy is husk, giving an annual total production of 120 million ton. RHs are the hard protecting coverings of rice grains and removed from rice seed as a byproduct during the milling process. In majority of rice producing countries, most of the husk produced from processing of the rice is either burnt or damped as waste. Once RHs are formed, efficient utilization is urgently needed to avoid environmental pollution. It is attracting as value added material for domestic and industrial processing. Burning of RH in ambient atmosphere leaves a residue of 14 - 22% called rice husk ash (RHA) which represents a burden economically and environmentally on the country. It causes toxic air pollution also dumping causing a damage to the land and the surrounding area in which it is dumped. Therefore RH and RHA must be appropriately managed. The RHA contains from 90% to 95% silica based on rate of burning, temperature and time of calcination. The extracted silica is normally in the crystalline form, but under controlled burning conditions it gives amorphous silica, which is highly reactive due to its ultra fine size and high surface area. Silica (SiO_2) is one of the valuable inorganic multipurpose chemical compounds. It is an atom bonded to four surrounding O atoms by Si-O-Si bonds. It occurs in nature mainly in three crystalline forms namely quartz (hexagonal), cristobalite (white) and tridymite (hexagonal) and also in amorphous form like opal. Amorphous silica prepared by physical combustion with controlled temperature. Thus amorphous silica with specific properties such as high purity, good reactivity and large surface area can be produced from rice husk and the process can be economically viable if the fuel value of the material is also made use of. Although various research studies concerning the production and application of RHA have been already made, there are still gaps regarding the optimization of treatment conditions and its relationship with the properties of the final RHA. In this sense, for producing high reactive and pure silica, some conditions are crucial in order to obtain an amorphous structure and the absence of unreacted carbon. The present article review is concerned with published researches related to synthesis of amorphous silica, nano silica from RHA and their applications for a better knowledge about the appropriate conditions to achieve a good quality of silica product to be used for different purposes such as adsorption of wastes from wastewater, preparation of silica gel, silicon carbide and solar grade silicon.

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2 Rice Husk Properties

Value addition of any material including wastes is based on the understanding of their properties and composition. Chemical composition is one of such property, which determines the end use of any material. This is also important for pre-treatment to be given to RH for further processing. The physical properties of the RH as reported from different sources [1, 4,3,5,6,7,8] are: odorless, of particle size of 1-10 μm , length of 5-12 mm long and 2-3 mm thick, of bulk density 96-116 kg/m^3 , hardness 5-6 Mohr's scale, molecular weight 60.06. It's melting point is 1440 $^{\circ}\text{C}$ and boiling point is 2230 $^{\circ}\text{C}$, specific gravity is 2.650 at 20 $^{\circ}\text{C}$ and calorific value is 720 kJ/kg. Table 1. presents the composition of RH as reported from different published papers. It is remarkable that the average composition of RH is mainly 75-90% by weight organic compounds (cellulose, hemicellulose and lignin) and about 10-25% by weight silica and metal oxides. The elemental analyses of RH from different sources are presented in Table 2. It is noticeable that the elements percentages ranges are: 35-37C, 4-8.8H₂, 31-36.6O₂ and 0.23- 0.5N₂.

Table 1: Organic Composition of RH from different published papers

Constituents	Composition % Percentage										
	[2]	[3]	[6]	[7]	[8]	[9]	[10]	[11]	[12]	[13]	[14]
Cellulose	75-80	75-90	40-50	34.4	75-86	31.12	38	34.00	69-87	75-80	80-86
Hemi Cellulose				24.3		22.48	20	23.50			
Lignin			25-30	19.2		22.34	22	22.00			
Mineral Ash (SiO ₂ and metal oxides.	15-20	10-25	15-20	18.85	14-25	13.87	20	14.00	22-29	20-25	14-20
Water	10	15	8-15	3.25	11	7.86	--	4.50	9	5	4-6

Table 2: The elemental analyses of RH from different sources.

Element	Percentage			Average
	[5]	[1,3]	[7]	
Carbon	37.5	35	37.05	35-37.5
Hydrogen	5.4	4-5	8.8	4-8.8
Oxygen	36.6	31-37	35.03	31-37
Nitrogen	0.5	0.23-0.32	ND	0,23-0.5
Sulfur	traces	0.04-0.08	traces	0-0.8
Ash	20.0	22-29	19.01	19-29
Moisture	---	8-9	---	8-9

The properties and the constituents of the RH and consequently RHA are found to vary from sample to sample as presented in Tables 1 and 2. The reason of variation may be resulted from the type of paddy, climate, and geographical conditions, in addition to the sample preparation and methods of analysis. Table 3 represents the chemical analyses for different RHAs produced from different sources. It is noticed that the ranges of the oxides components as weight % are: silica 84-96% , CaO 0.25- 2.55%, MgO 0.01- 1.3%, Al₂O₃ 3.8- 021 %, Fe₂O₃ 0.15- 1.95 % , Mn₂O₃ 0.03- 5.64 % , K₂O₄ 0.2-2.6 % , others.

X-ray fluorescence (XRF) and X-ray diffraction (XRD) are used for chemical analysis and structure of the RH, RHA and silica. The structure and morphology of silica and SNPs can be examined by electron scanning microscopy (SEM) and the specific surface area of the SNPs can be evaluated using the BET method, and the pore size distribution is calculated by the BJH method. TGA.

Water data are not determined in some references as presented in table 1 and 2 and it can be calculated from the 100% of the total constituents. as shown in table-1

3 Applications of RHA

Suitability of RHA mainly depends on the chemical composition of ash, predominantly silica content in it. RHA has been widely used in various industrial applications such as: processing of steel during the production of high quality flat steel, insulator in ceramic and refractory industry, as silica source due to the presence of large silica

Table 3: The chemical composition or RHA from different sources.

References	Components wt %	SiO ₂	CaO	MgO	Al ₂ O ₃	Fe ₂ O ₃	Mn ₂ O ₃	K ₂ O	Na ₂ O	P ₂ O ₅ , SO ₄ and TiO
	[1]	94.5	0.48	0.23	0.21	0.54	1.09	Traces	Traces	Traces
	[5]	90.5	1.48	1.23	1.21	1.54	1.094		2.946	
	[6]	80-90	1.0-2.0	0.5-20	1.0-2.5	0.5	-	0.2	0.2-0.5	
	[7]	89	1		1.2	0.41	5.64	2.48	0.28	
	[8]	72.1	ND	0.7	0.3	0.15	0.15	0.72	0.5	0.06
	[9]	91.4	0.17	1.3	1.57	0.62	0.11	ND	ND	ND
	[9]	89.4	2.55	1.3	3.81	1.95	0.03	ND	ND	ND
	[9]	94.5	0.25	0.23	Traces	Traces	Traces	1.1	0.78	0.53
	[15]	96.51	0.66	0.77	0.15	0.17	0.21	Traces	Traces	0.21
	[16]	98.61	0.21	0.01	0.1	0.21	0.02	0.16	Traces	0.18
	[17]	84.3	1.4	0.5	0.3	0.6	Traces	0.2	0.4	traces
	[18]	89	0.68	0.86	0.21	-	Traces	2.6	Traces	1.5
	[19]	94.64	1.89	0.96	traces	0.23	Traces	0.58	0.39	Traces
[20]	73	0.52	0.89	0.35	0.25	Traces	2.2	Traces	1.4	
[21]	96.34	0.41	0.45	0.41	0.2	Traces	2.31	Traces	Traces	
[22]	93.26	1.96	0.77	0.63	0.51	Traces	2.85	0.01	Traces	

Nd: Not Determined

content in ash, in blending with cement, as mineral additive to improve performance of concrete, as a highly reactive pozzolan in manufacturing of low cost concrete block, as adsorbent and catalyst/support in fine chemical synthesis. Other applications such as improving residual soil properties by mixing RHA and cement in suitable proportions as stabilizing agents, water purification, in vulcanizing rubber, for synthesize high-performance phosphors, in control of insect pests in stored food stuffs, as flue gas desulfurization absorbents, as an oil spill absorbent, in waterproofing chemicals, flame retardants, and as a carrier for pesticides and insecticides. Uma et al [23] explored the adsorptive characteristics of Indigo Carmine (IC) dye from aqueous solution onto rice husk ash (RHA). Batch experiments were carried out to determine the influence initial pH, contact time, adsorbent dose and initial concentration on the removal of IC. The pseudo-second-order kinetic model represented the adsorption kinetics of IC on to RHA. Equilibrium isotherms were analyzed by Freundlich, Langmuir, Temkin and Redliche and Peterson models using a non-linear regression technique. Adsorption of IC on RHA was favorably influenced by an increase in the temperature of the operation. The positive values of the change in entropy and heat of adsorption and the negative value of change in Gibbs free energy indicate feasible and spontaneous adsorption of IC on to RHA. Sivakumar et al [24] deal with the removal of Cr (VI) in a tannery industry wastewater using rice husk silica powder as an adsorbent. The experimental investigations have been carried out by using rice husk silica powder for different adsorption dosage, different contact time and different pH against the initial Cr (VI) concentration of 292 mg/L. The maximum percentage removal of Cr (VI) in a tannery industrial wastewater (88.3 %) was found at an optimum adsorbent dosage of 15 g, contact time of 150 min., and pH of 4. The experimental data were fitted to Langmuir and Freundlich isotherm models. Isotherm models result indicated that the equilibrium data fitted well with the Langmuir isotherm than Freundlich isotherm. Ahmaruzzaman, et al [25] studied the removal of various pollutants from water and wastewaters such as dyes, phenols, organic compounds, pesticides, inorganic anions, and heavy metals with rice husk as an adsorbent.

4 Silica preparations from RHA

The ash resulted from burning RH contains various metal ions and unburned carbon which influences the purity and color of the ash. Controlled burning of the husk after removing these ions can produce silica in white amorphous or in crystalline silica form of high purity. Many researches for synthesize of silica in amorphous or crystalline or in nano-silica forms were carried out. Bogeshwaran K. et al [9] reported that RHA is obtained by burning of RH in a muffle furnace at a temperature of 500, 600 and 700°C for 2, 3, 4, 5 and 6 h. RHA then treated with HCL and NaOH where amorphous silica was produced at 700°C for 3 h. High content of amorphous silica was processed by Rosario M. et al [2] via washing, leaching with H₂SO₄ for 2 h, then calcination for 2 h at 540°C. Chandrasekhar et al [21] produced Silica of high purity, chemical reactivity and white color by controlling the heating conditions. Sideswaran et al [22] prepared pure white amorphous silica of high purity where small particle size and high surface area was produced by pretreating the husk with HNO₃ followed by calcination. Sathy et al [26] prepared amorphous and reactive silica form by treatment of RH using 0.1 N HCl acid and subjected to heat treatment at different temperatures from 500 °C to 1000 °C. Abhishek Mehta et al [27] reported that RH was calcinated at 500°C and 700°C, duration time of 2h and 3h. Farshid et al [28] leached RH with 1N HCL, 4.5N H₂SO₄, and 1N

HNO_3 . Silica extraction process was carried out under controlled calcination conditions in an electric furnace at 600°C . A purity of 95.55% amorphous silica was obtained. Chakraverty et al [29] studied effects of various acid treatments of rice husk to remove metallic ingredients. and in addition different combustion temperatures to produce amorphous white silica ash where 1N HCl was the optimum acid followed by calcination at 500°C for 5 h. Chandrasekhar et al [30] investigated leaching of RH with acetic, oxalic acids, hydrochloric and nitric acid. They found that organic acids improved the properties of ashes comparable to mineral acid leaching. Yalcin et al [31] prepared pure activated silica by pre-and post-treatment using 3% (v/v) HCl, 10% (v/v) H_2SO_4 and 3% (v/v) NaOH solutions for 24 h. Samples were incinerated for 500°C , 600°C , 700°C , 800°C under air, argon and oxygen for 4h. They concluded that 1N HCl removed most of the metallic ingredients and producing ash completely white in color at 500°C . Ghosh et. al [32] found that roasting of silica to attain amorphous structure at temperatures between 500 - 600°C and the presence of crystalline silica in RHA sintered at temperature range of 600 - 750°C . Amorphous silica is obtained at 540°C for 2 h. Thuadaj et al [33] prepared RHA by burning RH at 700°C for 3 and 6 h, respectively. 98.14%. of silica content obtained at 700°C for 6 h. Krishnarao R.V. et.al [34] investigated the calcination of RH at high temperature (800°C), the presence of potassium led to surface melting and carbon fixation in the lattice. Calcination of RHA must be calcinated in temperature less than 800°C to obtain amorphous silica. Sathy etal [14] reported that the rice husk as well as the acid treated husk was subjected to heat at different temperatures from 500°C to 1000°C . Reactive silica is formed at 700°C and soaking time 2 h. Shamle et al. [35] reported that RHA obtained at 500°C for 5 hours. The silica content was $88.55\pm 0.20\%$. Ezzat Rafiee1 [36] treated RH with an acidic solution (0.1-1N HCl and 0.1N HNO_3) for nearly 90 min by stirring frequently and dried in an oven at 110°C for 24 h. Thermal treatment at different temperatures (500°C , 700°C , and $1,000^\circ\text{C}$) and the rates of heating was $2^\circ\text{C}/\text{min}$, $5^\circ\text{C}/\text{min}$, and $10^\circ\text{C}/\text{min}$, were checked. RHA was obtained at 1N HCl for 1.5 h at calcination temperature of 700°C . Rozainee et al [37] reported that leaching of acetic and oxalic acids improved the properties of RH comparing with leaching with hydrochloric and nitric acids of different concentrations. Chandrasekhar S. et al [38] prepared amorphous, reactive and high purity silica with high surface area and pore volume and good optical properties in presence of "chemical activators" such as KOH, K_2CO_3 , and Na_2CO_3 . Patel et al [39] prepared RH by calcination up to 1000°C in air, oxygen, argon and non-oxidizing atmospheres. Chemical treatments consisted of HCl, H_2SO_4 , HNO_3 , NaOH and NH_4OH . Carbon and SiO_2 contents in rice husk, caked at different temperatures (200°C , 300°C , 400°C) and time. AC: SiO_2 ratio of 2:1 is required for the production of solar grade silicon at low temperature. SiO_2 of 99% purity can be obtained by acid leaching and calcination for 400°C for 2h. V.R. Shelke et al [40] obtained silica in amorphous form from RHA when RH is burnt in controlled temperatures below 700°C . The transformation of this amorphous state to crystalline state takes place if the ash is exposed to high temperatures of above 850°C . Rajesh et al [32] studied the precipitation of silica by using various parameters such as acid washing, carbonization temperature (550°C - 700°C), time and activating agent such as sodium hydroxide, zinc chloride and phosphoric acid. Optimum carbonization time was at 600°C for 1h. Rozainee et al [2] reported that amorphous silica is obtained after calcination at 700°C for 6 h.

From previous survey a process description for silica preparation is presented as shown in Fig.1

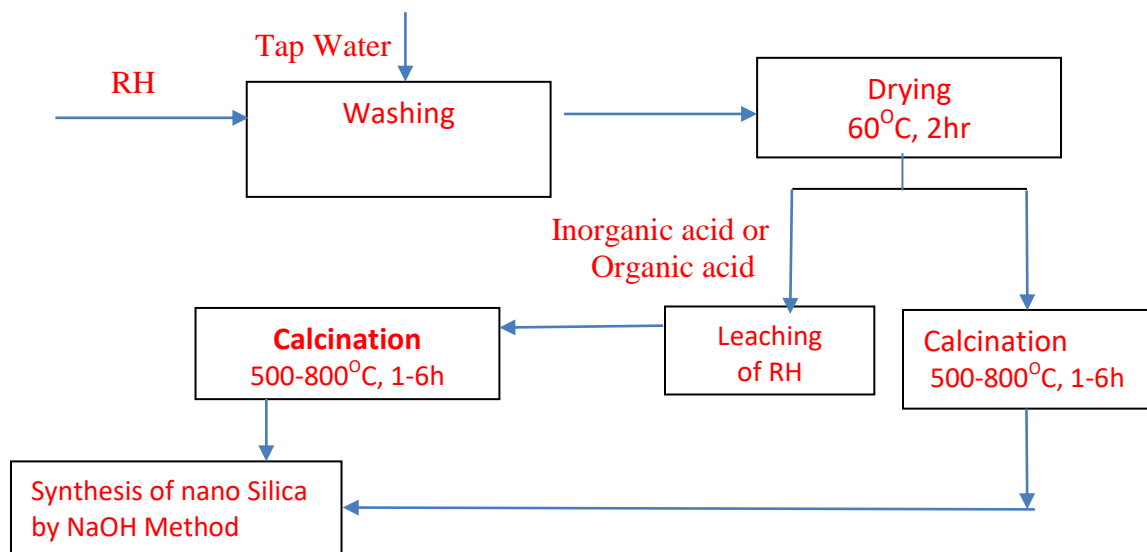


Fig.1: Scheme procedure of Silica preparation.

5 High Pure Silica Preparations

Many researches were carried out to improve the properties of extracted silica from RHA to be used for producing high quality products. Proctor et al [41] reported that amorphous silica from RHA can be extracted using low temperature at alkali extraction. Shelke V.R. et al. [42] produced sodium silicate by reacting RHA with aqueous NaOH, where silica is precipitated from the sodium silicate by acidification. The conversion of about 90% of silica in RHA into sodium silicate was achieved in an open system at temperatures of about 100 °C. Bogeshwaran .K et al [9] reported that formed RHA when treated with NaOH and HCl will produce sodium silicate (NaSiO_3) which can be used for water treatment. Thuadaij et al [43] prepared RHA by burning RH at 700 °C for 6 h, respectively. RHA was purified by alkaline extraction method by using 2.0, 2.5 and 3.0 N sodium hydroxide. Percent yield of silica extracted by 2.5 N NaOH, was 90.3%. Subsequently, the RHA was subjected to precipitation method in order to produce nanosilica. The precipitation was done by refluxing silica from RHA in boiling 2.0, 2.5 and 3.0 N NaOH, respectively. TEM results showed that 2.5 N NaOH for 10 h provided agglomerate particles with dimension of 5-10 nm. The specific surface area was found to be 656 m^2/g . Ghorbani et al [44] reported that nitric, sulfuric and hydrochloric acids were applied to evaluate the effect of different rice husk pretreatments. Silica extraction process was carried out under controlled calcination conditions in an electric furnace at 600°C. A purity of 95.55% amorphous silica with major impurities of K_2O , CaO and P_2O_5 were obtained indicating a relatively significant portion of it removed by acid treatment. This product was then subjected to chemical precipitation followed by slow gelation to obtain silica nano particles (SNPs). The BET specific surface area of 409 m^2/g , the average particle size of 200 ± 20 nm, the spherical particle shape and purity of 97% indicates the successful production of silica nanoparticles. Yuvakkumar et al [45] prepared a highly pure, small particle-sized and high surface area nano silica powder from rice husk using alkali extraction, followed by an acid precipitation method. High-purity nano silica powder was obtained by sodium hydroxide purification treatment of 0.5, 1, 1.5, 2 and 2.5 N. The high purity of silica (99.9%) was obtained at 2.5 N NaOH treatment. The pure nano silica powder that is obtained shows an average particle size of 25 nm with a high-specific surface area of 274 m^2/g , with an average pore diameter of 1.46 nm. Rafiee et al [46] investigated the production of nano silica, where acid treatment of RH followed by thermal combustion under controlled conditions gave 22.50% ash of which 90.94% was silica. Silica was extracted from RHA by using, 2.5-M sodium hydroxide solution. The obtained viscous, transparent, and colorless solution was allowed to cool down to room temperature, and 10 M H_2SO_4 was then added until pH 2; then NH_4OH was added up to pH 8.5. Nanosilica was prepared by reflux technique where 6.0 M HCl to make it acid free, then dissolved in 2.5 M sodium hydroxide followed by H_2SO_4 addition until it reached pH 8. The precipitated silica was washed to make it alkali free and then dried at 50°C for 48 h in the oven. Ezzat Rafiee [46] investigated various chemical treatments in varied conditions for controlled combustion to produce highly purified nanosilica. At optimized conditions, a nanosized, highly purified silica (98.8 mass percentage) was produced with a high surface area, high reactivity, and 99.9% amorphous in form. Della et al [47] reported that 95% silica powder could be produced after heat-treating at 700 °C for 6 h. The specific surface area of particles was increased after wet milling from 54 to 81 m^2/g . Rajesh et al [32] studied the precipitation of silica by using various parameters such as acid washing, carbonization temperature (550-700°C), time and activating agent such as sodium hydroxide, zinc chloride and phosphoric acid. Optimum carbonization time was at 600°C for 1h. Weixing Wang [48] synthesized silica nanoparticles using rice RHs as the raw material via controlled pyrolysis. The characterization results showed that the morphology of the synthesized silica was highly related to the pretreatment of RHs and the pyrolysis conditions. Particularly, potassium cations in RHs were found to catalyze the melting of silica, during which the amorphous silica were converted to crystalline phase. Two hours of pyrolysis at 700 °C appeared to be ideal to synthesize silica nanoparticles with a diameter of 20–30 nm. Higher temperature and longer duration of pyrolysis led to undesired melting of silica nano particles, while too low temperature cannot effectively remove carbonaceous residues. Singh D. et al synthesis and characterize of phosphoric acid activated ash nano silica [13]. N Setyawa [49] examined influences of various volume and concentration on silica extraction. The silica was extracted using a technical alkaline grade solvent (NaOH) with a variation in volume/ the ratio of RHA to the amount of solvents (1: 4, 1: 5, and 1: 6) and the solvent concentration (0.50, 0.75 and 1 N). The highest average yield was found in the concentration of 1 N solvent with the ratio of RHA to the number of solvents 1: 6 is 62.83%. Based on the physical characteristics of silica from RHA indicates that the use of technical alkaline solvents tends to decrease the purity of the proven silica also with the presence of major contaminants such as Na and Cl and other elements. In saline soils, the presence of contaminants in silica with an amorphous phase ($2\theta = 22.29^\circ$) especially Na and Cl may have a negative effect if used as raw material for fertilizers. J T Libreal et al. [50] investigated the effect of water and acid pre-treatment on the physicochemical properties of RH for silica extraction to minimize the impurities in the husks. Acid treatment was more effective in removing the sodium and potassium components compared to water treatment. Subash et al. [51] reported that aluminium nanocomposite extracted from the Chinese traditional joss paper using an alkalization procedure. Characterization of silica and aluminum nanocomposite extracted was

performed whereas 3D nano profiler has proven that the height of nanocrystal synthesized from joss paper waste is 234.37 μm .

6 Silica Applications

Silica can be used for silica aerogels to be used in super thermal insulators and dielectric materials. It is an economically viable raw material for the production of silicates and silica and in silicon chip in industry. Some of the uses of silica are in rubber industry as reinforcing agent, in cosmetics, in toothpastes as a cleansing agent and in the food industry as an anti-caking agent. It is also used for the preparation of inorganic materials like xylose, furfural, xylitol, ethanol, acetic acid and ligno sulfonic acid. Amorphous silica of high purity, small particle size and high surface area can be of use as an adsorbent or catalyst support in fine chemical synthesis. Zemnukhova et al [52] prepared silica containing specimens by the thermal method and deposition from rice production waste (straw and husk) which were evaluated for their ability to remove different strains of microorganisms from the water. In that study removal efficiency of SiO_2 specimens and aluminum silicate sample prepared from rice husk to different strains of microorganisms was higher than that of other specimens including commercial sorbent. The use of various processing schemes for rice production waste led to obtain efficient selective sorbents.

7 Preparations of some Silica Compounds

Following are some prepared silica compounds from high pure silica as reported from literature survey:

7.1 Preparation of Silica Gel

Supitcha. et al [53] separated silica gel from RHA by microwave heating. The experiments were performed by heating rice husk ash in sodium hydroxide solution with various concentrations in microwave oven for 5 or 10 minutes. The obtained sodium silicate was neutralized to give silica gel. The best condition for silica gel production was the reaction with 2.0 M sodium hydroxide at microwave power of 800 W for 10 minutes. The ability of silica gel as desiccant was investigated by adsorption test. The results showed that silica gel prepared by low concentration of sodium hydroxide solution had the highest ability of water adsorption. The power of microwave and volume of reactions seemed to have only little effect. Patil et al. [8] extracted silica by adding 1N NaOH to the washed and unwashed RHA samples and boiled for 1 h with constant stirring to dissolve the silica and produce a sodium silicate solution. The solutions were filtered. The filtrates and washings were allowed to cool to room temperature and were titrated with 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 h.

7.2. Preparation of xerogels

Kalapathy et al [54] reported an improved method to produce silica xerogel with lower sodium content by adding silicate solution to pH 1.5 hydrochloric, citric, or oxalic acid solutions until the pH 4.0 is reached. The aquagel was washed and dried to form silica xerogel. For comparison silica xerogels were also produced at pH 7.0. Silica xerogels produced by the improved method using citric and oxalic acid where it had sodium content of 0.52% and 0.22%, respectively. Kalapathy U. et al [55] developed a simple method based on alkaline extraction followed by acid precipitation to produce pure silica xerogels from RHA, with minimal mineral contaminants. The silica gels produced were heated to 80°C for 12 h to obtain xerogels of 93% silica and 2.6% moisture. The major impurities of silica produced from RHA at an extraction yield of 91% were Na, K, and Ca. Acid washing prior to extraction resulted in silica with a lower concentration of Ca (< 200 ppm). However final water washing of the xerogel was more effective in producing silica with lower overall mineral content (Na < 200 ppm and K < 400 ppm). Vinoda et al [56] prepared silica from RHA, where it was extracted by a developed an alkaline precipitation method to produce pure silica xerogel. Samantha P. et.al [57] presents a study on the production of silica gel in hydrothermal process using residual RH and the optimal silica gel was selected for use as a mineral additive in cement pastes. The results indicate that the mixtures containing silica gel showed improved mechanical behavior over all time periods evaluated.

7.3 Preparation of Silicon Carbide

Khalil Ahmad et al. [54] deals with the catalytic synthesis of silicon carbide from biomass husk of indigenous rice as raw materials. Rice husk samples were treated with different concentrations of sodium silicate, 40% hydrochloric acid and a mixture of 40% HCl and 2 g/l Na_2SiO_3 . Treated husk were pyrolyzed at 1350°C in argon atmosphere and then oxidized to eliminate unreacted carbon. Maximum yield was obtained in case of catalytic treatment with 2

gl-1 Na_2SiO_3 . Acid treatment, although showed no considerable increase in yield, fostered whiskers formation instead of particles.

7.4 Preparation of Solar Grade Silicon

Patil et al [7] investigated the thermal treatment of RH up to 1000°C in air, oxygen, argon and non-oxidizing atmospheres. Chemical treatments consisted of HCl, H_2SO_4 , HNO_3 , NaOH and NH_4OH . Purity, particle size distribution and SEM micrographs of chemically treated samples were carried out. Carbon and SiO_2 contents in RH, caked at different temperatures and time, have been determined to show that a C: SiO_2 ratio of 2:1, required for the production of solar grade silicon, can be achieved at low temperature. SiO_2 of 99% purity can be obtained by acid leaching. The results have been interpreted in terms of two types of bonding of silicon, a rigid structural framework, and selective sites for the preferential attack of acid and alkali in rice husk. Amorphous silica nanoparticles with narrow size distribution and high purity are expected to replace silica gel and fumed silica for various applications.

8 Conclusions

Silica extraction are carried out by washing, drying, leaching and calcination, where different parameters are tested such as acid leaching, calcination temperature and time, rate of heating, activating agent and time of activation. Leaching process for RH are carried out by using organic or inorganic acid such as HCl, H_2SO_4 , and HNO_3 , acetic and ceric acid for decreasing the metal oxides in RH. It can be concluded that the optimum acid used for leaching is in the range of 0.1-1N HCL, and the extracted silica is normally in the crystalline form, but under controlled burning conditions it gives amorphous silica. Calcination can be carried out at temperature in the range from $500 - 700^\circ\text{C}$ to produce amorphous silica. Crystalline silica is produced above 700°C .

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