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A Review on Recent Studies on Development of Rice Husk Silica and Its Application in Thin Film Growth

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Abstract

Rice husk based SiO_2 is considered as a non-toxic, environmental-friendly, sustainable, also suitable for large scale production and cost-effective material with a wide range of applications. Burning the fuel, rice husk to generate energy results in the waste product called rice husk ash (RHA). RHA is an abundant agricultural by-product. It is rich in silica (over 65%) and can be made into economically viable raw material which can be used for production of silica gels and powders. Recently its application in an adsorbent, catalyst support and thin film semiconductor material is being considered. This paper reviews the production of silica particles from rice husk ash and development of silica thin films using the spray pyrolysis technique.

Keywords

Rice Husk, Silica Gel, Leaching, Spray Pyrolysis, Environmental-Friendly, Agglomeration

1. Introduction

Ever increasing demand for cheap, safe and non-conventional silica as raw materials in industries, and also the importance of having a pollution-free environment has led to worldwide attention on the research and applications of silica. Agricultural wastes and by-products are cheap and reliable bio-sources of silica, which can be recycled to produce a high-end value material, thereby effectively reducing pollution problem that vast amounts of agricultural wastes constantly constitute to the environment.

Artificial sources of silica, which includes sodium silicate, tetraethylorthosilicate (TEOS) and smelting of quartz in high temperatures. Reasonably high percentages of silica can be found in clay and the ocean, which are natural sources of silica as well as plants which have accumulated silica through the process of biomineralization [1-3]. Silica from plants is a mostly amorphous, hydrated and usually polymerized material produced from silica acid [$Si(OH)_4$]. Agricultural wastes that are high in silica content are Cogon grass, Corn cob, Wheat husk, and Rice husk. 60% of silica present in pyrophyllite clay of which roughly 85% of this silica is amorphous [4]. An amorphous means array of molecules in silica is irregular. 36% silica can be extracted from corn cob by treating it with organic acids [5]. Grasses are known to contain silica biomaterials as a silica presence within plants is necessary for structural support and pathogen defense [3]. Silica can be found in cogon grass, though in relatively low quantity with its purity and non-crystalline influenced by the presence of potassium [6]. Wheat husk contains 9% of silica that can be retrieved under the right processing conditions [7]. Rice husk is a secondary product, which is also considered as waste materials in the courses of rice milling. It encapsulates the rice grain and when separated from the grain, produces one of the highest volumes of bio-residues. Rice husk is very rich in silica compared to the other bio-sources of silica and the chemical analysis of rice husk has shown that it contains 22.12% silica [8]. A higher percentage of amorphous silica can be gotten by burning these plants to ash in a controlled incineration. In terms of yield, the burnt rice husk known as rice husk ash (RHA) has higher yield of silica than both corn cob ash and cogon grass ash [9]. Burnt rice husk contains more than 90% silica in crystalline form. Silica from rice husk ash is a promising, cost-effective and safe substitute to silica synthesized from artificial sources.

Table 1. *Chemical composition of rice husk [8].*

Constituents	Weight in %
Moisture and Organic material	73.87
SiO_2	22.12
Fe_2O_3	1.28
CaO	1.24
Al_2O_3	1.23
MgO	0.21
MnO_2	0.074

World rice production is approximately 645 million tons [8] and the rice husk generated as waste material from such huge size of production will definitely contribute to environmental pollution hence, recycling is very essential. Rice husk is an agro industrial waste that contains a reasonably high percentage of silica content [10]. Rice husk is tough and its low biodegradability is due to high silica and organic content present, especially Lignin. Feeding domestic animals with rice husk cannot be sustained because of its low nutritive properties [11]. Rice husk is burnt for combustion in cooking stoves, and for heat generation leading to air pollution and considerable health risk. RHA obtained from this burning is also categorized as waste material. The target product in RHA is its silica which is a fundamental raw material utilized in production of ceramics, rubber, pharmaceutical drugs and a host of other materials [12]. Silica is a binary semiconductor compound which needs less demand in accurate atomic ratio. Silica has application in electronic industries as insulation materials in integrated circuits and also non-linear optics and electro-optics [13]. Silica is also widely used in photocatalytic process and it was reported that silica is able to boost photocatalytic reactivity in TiO_2 compounds [14-18]. Silica is now being tested as absorber layer in thin film semiconductor research and it can be recovered from rice husk through acid leaching and gasification methods [19, 20]. Amorphous silica can then be obtained by treating rice husk ash with alkaline material. In this review, we will consider the method of fabricating silica from rice husk and its application in semiconductor thin film. The industrial utilization of RHA silica will ultimately lead to reduction in cost of production of silica-based materials.

2. Methodology

2.1. Extraction of Silica from Rice Husk

Extraction of silica is a wet chemical process which has to be properly managed to significantly reduce the amount of impurities present in the final product and also to produce amorphous silica with high reactivity. Optimization of the extraction parameters is therefore important for the development of quality silica. The parameters are source of rice husk, pretreatment of rice husk before burning, temperature at which rice husk is burnt, resident time of rice husk in furnace, and acids used in mineral acid leaching of RHA. Mineral intake of rice plant differs depending on soil type; hence, the source of rice husk is also a factor that will determine the percentage of impurities and organic compounds bonded covalently to silica in rice husk [21]. Structural properties like specific surface area and pore volume vary under intense temperature needed for processing rice husk. Many authors have agreed that leaching of rice husk before thermal treatment with temperatures within a range of 500°C and 1400°C for various time intervals is very effective in eliminating metallic impurities and produces ash silica completely white in colour [8, 22]. Crystalline silica with low reactivity was found in untreated rice husk [23]. It has been reported that silica is well bonded to organic constituents within the rice husk by strong covalent bonds, thereby making that portion of bonded silica resistant to extreme temperatures and solubility in alkali. The implication is guaranteed extraction of pure silica after organic constituents of the rice husk has been removed. RHA was obtained from pyrolysis of grinded rice husk in vacuum or non vacuum environments at very high temperature (higher than 500°C in most literatures) at a definite time. Burning rice husk in vacuum is an energy-intensive process and efforts have been made to reduce the magnitude of energy consumed and increasing the resident time. It is however observed in literature that highly reactive amorphous silica content of the final product requires burning rice husk at lower temperatures at a long residence time for rice husk in the furnace. Burning rice husk at temperatures above 1000°C at a shorter resident time will produce crystalline silica. Amorphous silica was obtained by subjecting RHA to chemical treatment using various acid and alkaline solutions. A non-intensive energy method for producing pure silica from RHA with 91% yield was developed by Kalapathy et al., [24]. Pure silica was obtained by dissolving RHA silica with alkali solution to form sodium silicate solution and subsequently forming silica aqua-gel by adding (HCL) acid solution to lower the pH from 11.8 to 7.0 [24].

Silica was extracted by grinding rice husk (RH) followed by its chemical treatment with hydrochloric acid hour with constant stirring to make the organic matter soluble [25] although there have been cases where grinded rice husk did not receive any chemical treatment before burning [21]. The soluble RH is washed with ultrapure water and it was filtered under vacuum, obtaining a pulp H_2SO_4 and H_2O_2 were added to the pulp under constant stirring for one hour to promote oxidation of the organic matter. Finally, silica was obtained from the pulped RH by vacuum filtering and calcinations in oven at 600°C for 4 hours. In a study of silica gel, preparation of silica [26] by Tang and Wang in 2005, RHA was obtained from RH burnt at 600°C for 4 hours. Another study reported by Van Han Le *et al.,* [27], RH was pretreated with 10% HCL and 30 wt% sulfuric solution. The pretreated material was incinerated at 600°C at 4 hours to expel the hydrocarbons. Later, 10g of RHA was leached with 10% HCL and afterwards 30 wt% sulfuric solution at 100°C for 2 hours. Acid was added at a slow rate until pH was 7. Shelke and his groups produced silica RHA as sodium silicate source using aqueous sodium hydroxide. Silica was precipitated from sodium silicate by acidification using orthophosphoric acid. The addition of acid was done slowly till a pH of 6.5 was reached and white silica precipitate was obtained. The wet precipitate was dried in an oven for 24 hours at 110°C. Silica obtained was in form of white amorphous powder having 98% bulk density 1.25g/ml [28]. Amorphous silica powder was obtained from rice husk by applying thermal treatment at several temperatures. After burning the rice husk at 700°C for 6 hours, rice husk ash samples obtained presented the highest amount of silica compared to other samples [29]. Sixty ml portions of 1M NaOH were added to washed and unwashed rice husk ash samples and boiled in 250ml Erlenmeyer flask for an hour under constant stirring for dissolution of silica and production of sodium silicate solution. The solutions were filtered and the residue washed in boiling water. The filtrates and washings are allowed cool at room temperature and HCL was added with constant stirring to obtain silica gel precipitate. Daramola *et al.,* [30] prepared submicron silica gel from rice husk using water shaker bath. The concentration of NaOH as leaching agent was optimized and it was observed that chemical composition of silica increases with increase in NaOH.

Generally, the application of silica is in powder form, it is also important that Silica nanoparticles be immobilized on a heated substrate via thin film technology after dissolution of silica gel in an appropriate solvent, for example, aqueous alkali metal hydroxides. Thin film technology uses fewer materials and will therefore reduce the cost of materials. There are several processes through which rice husk Silica thin films have be produced, the most popular ones being sol-gel [31, 32] and chemical vapour deposition (CVD) [33]. Sol-gel is the most attractive because it is a cheap and simple process while greater film smoothness has been obtained from CVD. Recently, commercial silica thin films have been developed from chemical spray pyrolysis process (CSR) [34] but this method has not been extended to silica synthesized from rice husk. Spray pyrolysis technique is cost effective and the process is applicable to several substrate/film systems, whereby the retention of chemical properties and phase homogeneity of films deposited is guaranteed [34, 35]. There is increased adhesion of deposited films and it also has the advantage of complete decomposition of micro-sized droplets before it gets to the target as a solid particle [36].

2.2. Silica Thin Film Preparation

Economically viable RHA silica thin film can be fabricated using inexpensive and industrially scalable methods. Methods of preparing silica thin films require high temperatures especially the techniques involving wet chemistry. An alternative is physical methods of deposition of films which require a complex vacuum device but will increase fabrication cost as vacuum instruments are very expensive. The device structure for chemical deposition of thin film is simpler and cost effective.

Sol-gel method will make the readily hydrolysable metal compound react with water in certain solvents, forming Sol through the process of hydrolysis and polycondensation and make Sol form liquid film on substrate by dipping or spin coating method. After gelatinization, it can be transformed into amorphous or crystalline films by heat treatment. Thin films fabricated by spin coating method usually involves three steps; first, by preparing precursor solution (soluble compound of Silica) containing specific ion; second, spin-coating precursor solution on the glass substrate to form film; and third, annealing thin films in a proper atmosphere [37]. Sol-gel method has been mostly used in the preparation of Silica thin films/powder synthesized from TEOS and other commercial compounds [27, 14, 38] but has rarely been used in preparing thin film from RH silica. In 2012, an hybrid photocatalyst of SiO_2-TiO_2 thin film was prepared from rice husk silica [14] Van Hai Le *et al* [27] reported the synthesis of silica nanoparticles in from rice husk using sol-gel method [27].

Chemical vapour deposition (CVD) is a chemical process used to fabricate high-performance thin film semiconductor. In typical CVD, the substrate (target material) is exposed to one or more volatile precursors which react and decompose on the substrate surface to produce desired solid particles. CVD was first utilized in the production of SiO_2 thin films by Seitaro Matsuo and Mikiho Kiuchi in 1983. CVD method was used in depositing SiO_2 and Si_3N_4 without substrate heating utilizing electron cyclotron resonance plasma [39]. Nan Jianget *al* [40] reported the deposition of SiO_2 thin films using CVD method. The effects of reactant gas mixture composition (O_2 and SiH_4) on the dielectric behavior of SiO_2 were studied [40]. The electrical performances of SiO_2 films in metal oxides semiconductor structures were assessed by several characterization techniques. Another method is spray pyrolysis. Spray pyrolysis is an aerosol process that atomizes a solution and heats the droplets to produce solid particles. A spray pyrolysis unit consists of an atomizer which sprays micro-sized droplets of precursor mixture on heated substrate. The spray pyrolysis method of deposition is scalable and applicable over a large area. It is a good technique for low cost deposition.-The spray pyrolysis technique has an advantage of producing fine micro-sized droplets that react with the heated substrate over CVD method. Unlike the CVD, there are many cases which large droplets of the solution do not vapourize before reacting to deposit on the substrate [41]. SiO_2 thin films have been grown by spray pyrolysis [34] but the method has not been employed in the fabrication of silica thin films from silica obtained from rice husk. Silica gel prepared from rice husk with SiO_2 concentration can be used as starting precursor and micro-sized liquid droplets of the precursor are generated by an atomizer. The droplets are converted to solid particles on target through a process of evaporation, precipitation, drying and decomposition. The solvent evaporates leaving a precipitate which decomposes on drying. The precursor must be low in solubility and solvent evaporation rate must be low for perfect aggregation of submicron particles. It should be noted that spray pyrolysis technique is an empirical process with some variables involved. The variables are;

1. Solute concentration is the amount of **solutes**/particles that are dissolved in a solution.
2. Atomization technique which is the type of atomizer used. Atomization refers to the process of breaking up bulk liquids into droplets. Atomizers can be a nebulizer or ultrasonic and pressure-based atomizer. Choice of atomizer will affect parameters like droplet size, atomization rate and droplet velocity.
3. Temperature during drying or sintering.
4. Residence time is how long the droplets move in the furnace. Modification on the droplet velocity will affect the residence time of the droplets in the furnace.
5. Carrier gases: type of carrier gas is a non reactive gas. The carrier gas can be varied from argon to pure nitrogen and other carrier gases including helium and air.

These variables have to be optimized to change the distribution of particle size, the size of the particle and the nature of the particle in the final product.

3. Results and Discussion

Energy dispersive X-ray spectrometric studies on RHA bulk silica have shown reduction of impurities in the final product by increasing the concentration of sodium hydroxide during the treatment of burnt husks [30]. Structural characterization of bulk silica by X-ray diffraction have indicated the amorphous nature of silica derived from rice husk burnt at temperatures below 1073 K. Rice husk burnt above this temperature will produce crystalline silica containing crystoballite and trydimite phases [42]. XRD results from Awizar et al [43] indicate crystalline phases in silica are noticed with increase in temperature [43]. We have mentioned that pretreatment of rice husk with chemicals before burning will produce amorphous silica and this was reported by Javed et al., [23]. Most reported morphological studies using SEM shows agglomeration and spherical morphology in RHA silica [8, 44]. At present, the methods of preparing rice husk silica gel require high temperatures and conditions of heating and it is possible this might lead to poor reproducibility of the preparation process. Abinitio studies has shown that manufacturing process of rice husk silica is accompanied by presence of metallic impurities which includes Ca, Na and K. Purity and yield Silica is highly dependent on the leaching agent used. The impurities can be removed by washing silica gel with deionized water after drying or by acid leaching. This will lead to increase in the cost of industrial production. Impurities impinge seriously on the quality of RHA silica whose effect may not go unnoticed in its applications, especially on thin film opto-electronic applications. Impurities are responsible for recombination centers that are known to interfere with sustainability and efficiency of thin films.

4. Conclusion

Agricultural waste rice husk is rich in silica from which silicon oxide based materials were synthesized under controlled conditions using acid leaching technique. Few works have been carried out on the fabrication of SiO_2 thin films by spray pyrolysis method and characterization of such films have not gone beyond structural. Silica thin film has been fabricated from silica sols by spray pyrolysis method and the film Structure, morphology and optical properties were investigated. Work should be done on the UV-vis characterization of SiO_2 as the results will pave way for photovoltaic device fabrication. Growing silica thin films from rice husk silica precursor will also save cost.

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