

Short Review: Biowaste as A Source of Silica and Its Application as A Filler to Fabricate The Superhydrophobic Silica-Based Coating

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ABSTRACT

Silica (SiO₂) plays a major key ingredient in producing products such as toothpaste, ceramics, and paints, to name a few, as it acts as a stabilizing agent, filler and additive. Due to its excellent properties, the use of high-purity SiO₂ in industrial applications is favourable. Unfortunately, high-purity SiO₂ is expensive. Tetraethyl Orthosilicate (TEOS) is an example of a SiO₂ precursor that is costly and harmful, yet frequently employed. This paper provides a short review of the advantages of biowaste materials as SiO₂ precursors and their role as fillers in the fabrication of superhydrophobic coating. Researchers nowadays are attempting to lower the expense of employing high-purity SiO₂ by extracting silica from biowaste using many methods such as acid leaching and alkali treatment as this option is highly sustainable. The growth of agricultural industries is exponential due to the increase in biowaste production. Therefore, this is one of the ways to utilize the use of biowaste in combatting the environmental issues regarding excess biowaste and receding pure resources. SiO₂ from biowaste also can be utilized as filler and used to develop superhydrophobic coating, providing numerous potential applications.

Keywords: *Biowaste, Silica, Superhydrophobic coating*

1. INTRODUCTION

Biowaste materials such as agricultural, lignocellulosic, and wood waste are part of the biomass. However, these organic residues need not only to be converted into biomass energy but can also be utilized to contribute to various aspects of human life. According to the Department of Statistics Malaysia, about 1.2 million tonnes and more than 900 million tonnes of biowaste were annually and globally discarded into landfills, respectively. The dumping of biowaste has intrigued scientists to investigate suitable solutions to ensure that biowaste can be utilized as a renewable energy source and an important ingredient in making various materials in various branches of science.

Plants such as sugarcane bagasse, corn, wheat straw, bamboo leaf, rice husk, and palm trees are some of the main biowaste sources. To be specific, biowaste serves as an enormous source of silica (SiO₂) production. Although SiO₂ is plentiful, especially in the crust of the earth, SiO₂ can still be obtained in some of the biowaste materials, which are highly environmentally friendly and leave fewer carbon footprints. In plants, SiO₂ enhances plant metabolism and increases resilience to pathogens and pests by assisting

plants in developing resistance to biotic and abiotic stress [1]. After harvesting, plants still contain SiO₂ along with other elements. The majority of the biowaste ash is made up of SiO₂ as pyrolysis and combustion processes are conducted to remove impurities and, at the same time, increase SiO₂ content. Moreover, ash from rice husks, sugarcane leaves, corn husks, and other sources varies from 98% to 36%. Other metal oxides, such as calcium oxide (CaO), magnesium oxide (MgO), and potassium oxide (K₂O), as well as a few trace elements, are also discovered in addition to SiO₂ [2].

SiO₂ is an inorganic compound of a typical diamagnetic with a low density, an excellent insulator and a photocatalyst semiconductor [3]. It is also the second most abundant mineral on earth [4] after oxygen. Figure 1 depicts the amazing properties of silica that make it a much demand compound. For example, a research study by Wang *et al.*, [5] in 2019 exploited the hydrophilicity nature of SiO₂ to impede the liquid formation in methane/liquid systems. In another research, the thermal properties of SiO₂ promoted the ability of microencapsulation of salt to be stabilized in high melting temperature was proved by Zhang *et al.*, (2018) [6]. In many industrial fields, SiO₂ plays

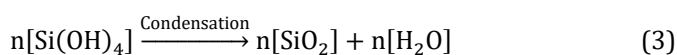
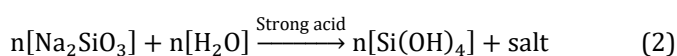
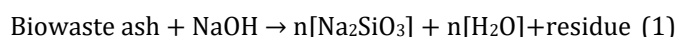
an important raw component in the production of materials such as coatings [7,8,9], composites, ceramics [2], insulations [10], cosmetics, electronics, and many more. Thus, in this short review, we attempt to discuss the synthesis of SiO₂ from biowaste as well as the application of silica as a filler to fabricate superhydrophobic coating.

2. SYNTHESIS OF SILICA FROM BIOWASTE

Many researchers reported the SiO₂ extraction process (Table 1) from biowaste, including the sol-gel method [11], thermal treatment, alkali treatment [10] and acid leaching [4], or a combination of any of these processes [12]. There have also been reports of various precursor materials other than biowaste to extract SiO₂. This includes Tetraethyl Orthosilicate (TEOS) and Hexadecyl Trimethoxysilane (HDTMS). Both chemicals are famously used as precursors to obtain SiO₂ nanoparticles by the Stöber method in the presence of catalysts and solvents. Unfortunately, due to the expensive, complex and time-consuming manufacturing process of SiO₂ extraction from TEOS and HDTMS, metal silicate solutions have been replaced as a low-cost and friendly alternative to obtain SiO₂ [13].

Alkali treatment is one of the most common chemical methods to obtain SiO₂ from biowaste due to its low cost and simplicity. Using an alkali to treat waste materials high in silica, a metal silicate solution such as Sodium Silicate

(Na₂SiO₃) solution is produced using strong oxidizing agents such as Sodium Hydroxide (NaOH) or Potassium Hydroxide (KOH), as shown in the Eqs. (1) – (3). Subsequently, the Na₂SiO₃ solution is acidified with an aqueous solution of strong acid or carbon dioxide gas to precipitate SiO₂ [13] or to obtain SiO₂ in gel form. The above treatment was conducted by Sreekantan *et al.* [9] in 2020, where they extracted the SiO₂ solution from Palm Oil Fuel Ash (POFA). They obtained POFA by combusting the palm kernel in the muffle furnace for a long period at 700°C before sieving with a 300 µm sieve. Subsequently, NaOH, a strong alkali solution, was used to treat 10 g of POFA. Afterward, the Na₂SiO₃ solution was let to cool down and filtered by Whatmann filter paper of pore size 11µm. To obtain the SiO₂ solution, Sulfuric Acid (H₂SO₄) was added dropwise until pH 3.



Another research study was also performed by Bakar *et al.* (2020) [20]. They utilized the rice straw as the main source of SiO₂ by performing the alkaline treatment of rice straw ash followed by precipitation by strong acid to obtain amorphous SiO₂ at the highest percentage of 74.11. Furthermore, the experiment was conducted by varying the concentration of Soda Anthraquinone (Soda AQ) solution to NaOH solution as well as the different temperatures for digestion of rice straw in the aforementioned solution to produce Na₂SiO₃ solution. Then, H₂SO₄ was used to neutralize the alkaline solution of Na₂SiO₃ until it reached pH 7. The SiO₂ gel was collected and dried at room temperature for further analysis.

Another method commonly used to obtain SiO₂ from biowaste is acid leaching. The process of extracting material from a solid using a liquid extraction medium is known as leaching. From its native solid state, the required component disperses into the solvent [4]. Other contaminants typically associated with agricultural ash, such as iron oxide and potassium oxide, to name a few, ought to be removed using acid leaching treatment [21]. Moreover, hazardous acids such as Hydrochloric Acid (HCl), Nitric Acid (HNO₃), and H₂SO₄ pose several economic challenges, which in turn causes major concern among

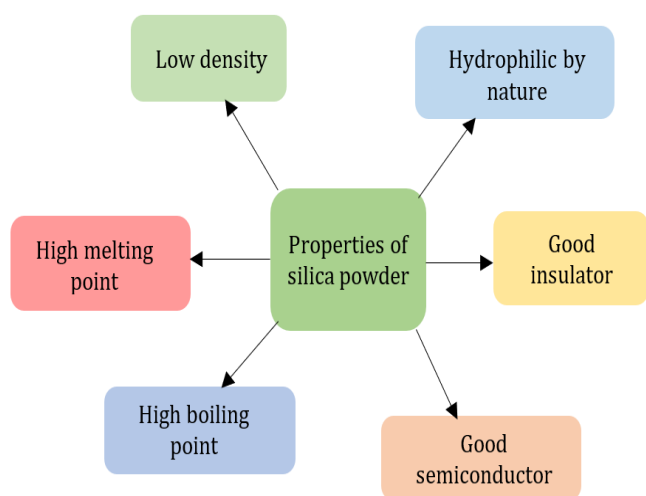


Figure 1. Properties of SiO₂

Table 1. A comparison of SiO₂ yield for different biowastes and extraction methods between a few studies

Biowaste	Synthesis method	SiO ₂ yield (%)	Reference
Rice husk	Sol-gel	86.52	[14]
Rice straw	Carbonization/ acid leaching/ reflux	85.9 (KOH as activating agent) 84.4 (K ₂ CO ₃ as activating agent)	[15]
Bamboo leaves	Sol-gel	85.57	[16]
Oil palm frond	Acid leaching/ sol-gel	67-68	[17]
Sugarcane bagasse	Acid leaching/ alkali treatment	66.3	[18]
Sugarcane bagasse	Acid leaching	78.61	[19]

researchers since the used strong acids require specific and proper disposal. Hence, it is important to employ specific materials that are usually expensive to resist corrosion phenomena due to strong acids [4,22].

Therefore, the best alternative is to use weaker organic acids, such as citric acid ($C_6H_8O_7$), which is non-toxic, inexpensive, and highly safe for long-term use, as conducted by Osman and Sapawe (2020) [21]. They achieved a high percentage of SiO_2 content by applying acid leaching treatment of Oil Palm Frond Ash (OPFA) followed by the sol-gel method to extract SiO_2 . They used Oil Palm Frond (OPF) as raw biowaste to obtain OPFA by combustion of OPF at $600^\circ C$ for 3 hours. Subsequently, the OPFA was mixed with citric acid for 60 minutes at $70^\circ C$ before rinsing with distilled water and calcinating at $800^\circ C$ in a muffle furnace. Afterward, the leached OPFA was added into the NaOH solution under constant stirring to produce a Na_2SiO_3 solution. The Na_2SiO_3 solution was adjusted to neutral pH, forming SiO_2 gel. The gel was dried in an oven for 11 hours at $80^\circ C$ after setting the gel to age for 18 hours in the refrigerator. As a result, the yield of SiO_2 was approximately 68.4%.

Some researchers used two different methods, which produced a high yield of SiO_2 as reported by [15], [17], [18], and [22]. For example, a different percentage of SiO_2 yield was revealed by Anuar *et al.* (2018) [23], who utilized coconut husk as a raw biowaste to synthesize SiO_2 by acid and alkali treatment. The yield percentage of SiO_2 for acid leaching and alkali treatment was 91.76% and 90.01%, respectively. This high yield percentage revealed by the treatment by acid leaching proved that this method greatly removed the impurities compared to alkali treatment,

Natarajan *et al.*, (2019) [24] used sugarcane bagasse ash to synthesize SiO_2 by alkali treatment to obtain Na_2SiO_3 solution before treating with H_2SO_4 acid to further remove the impurities which resulted in 94.2% of SiO_2 yield. A recent study by Porrang *et al.*, (2021) [25] revealed maximum percentage yield of SiO_2 of more than 90% after synthesizing the compound from rice husk and wheat husk. However, the Si element in rice husks recorded a higher percentage (71.02%) than in wheat husks (70.91%). The difference in yield percentage may be influenced by the extraction parameters such as temperature, time and molarity. Last but not least, Falk *et al.*, (2019) [26] also employed acid leading by HCl acid and alkali treatment by NaOH solution to acquire SiO_2 nanoparticles from sugarcane bagasse ash. They are able to obtain SiO_2 nanoparticles at yield percentages as high as 95.2%.

From those mentioned studies, we can conclude that biowaste is a potential source of SiO_2 . Different methods of synthesis revealed different SiO_2 yields, yet the percentage of SiO_2 yield is still high, although some methods may procure SiO_2 at a percentage below 50%. In addition, most researchers obtained raw biowaste before converting the waste into ash to increase the SiO_2 composition, followed by the necessary treatment to procure SiO_2 with little trace of other elements.

3. FABRICATION OF SILICA-BASED SUPERHYDROPHOBIC COATINGS

Essentially, the superhydrophobic coating is defined as a material that displays a Water Contact Angle (WCA) of more than 150° and a Sliding Angle (SA) of less than 10° [27, 28]. The unique behavior of water is a result of poor adhesion between water and substrate due to the formation of air pockets, therefore, contributing to the numerous applications of a superhydrophobic coating such as anti-bacterial [7], self-cleaning [29], anti-fouling [30] and anti-corrosion [31]. Note that the fabrication of superhydrophobic coating involves basic materials such as polymer matrix, filler, solvents, and modifiers. Ultimately, using biowaste as the raw filler is highly favorable among scientists as it is non-toxic, low cost, green, and readily available compared to commercial filler. When added to the polymer matrix, SiO_2 serves as a reinforcing filler, improving the coating's thermal, chemical, and mechanical properties, making it stable and environmentally friendly by nature [32]. Hence, the addition of a surface modifier is necessary to improve the hydrophobicity, as SiO_2 powder or silica solution is hydrophilic by nature.

For example, Sreekantan *et al.*, (2020) [9] fabricated a viable SiO_2 -based coating by combining the silica solution extracted from POFA and Polydimethylsiloxane (PDMS) and coating it onto the glass substrate. In this case, PDMS is the surface modifier for the SiO_2 solution. The coating exhibited a WCA of $151 \pm 1^\circ$ and SA of $7 \pm 1^\circ$ compared to a bare glass substrate. Based on the Atomic Force Microscopy (AFM) and Field-Emission Scanning Electron Microscopy (FESEM) images, the coating revealed a surface roughness of 21.80 nm. Meanwhile, the surface morphology displayed the existence of hills and valleys, which played a role in creating air pockets, thus, reducing the contact between water and substrate.

Another research work by Saharudin *et al.*, [27] in 2018 displayed a durable superhydrophobic SiO_2 -based coating derived from POFA with the addition of PDMS as a surface modifying agent. They prepared the coating by varying the ratio of PDMS to SiO_2 solution to improve the hydrophobicity of SiO_2 . The employed spraying technique was to coat the hydrophobic solution lass slides onto glass slides before drying in the oven for 5 minutes at $80^\circ C$. The spraying was repeated five times, followed by a curing process in the oven. By varying the PDMS to SiO_2 solution ratio, they obtained a coating with the highest WCA of $171 \pm 2^\circ$. In addition, the superhydrophobic coating exhibited excellent resistance against acid and base. The immersion of coating in three different pH of the solution (4, 7, 10) was conducted for 10 days. As expected, the superhydrophobic coating can retain its WCA above 150° , especially in a pH 10 solution. The X-Ray Diffraction (XRD) results for different types of silica in POFA revealed the existence of crystalline SiO_2 in α -quartz and cristobalite with percentages of 67% and 22%, respectively.

A study on the exploitation of bamboo leaf, a source of SiO_2 , to fabricate cost-efficient superhydrophobic coatings using

three different types of low-toxicity solvents and Trimethylchlorosilane (TMCS) as surface modifying agents using the spray coating method was reported by Silviana *et al.* (2021) [33]. They prepared and purified the SiO₂ from the bamboo leaf by calcinating the biowaste for 2 hours in a furnace at 650°C to produce bamboo leaf ash, then leaching it by 1N of HCl for 3 hours under constant stirring. Afterward, the cake from the filtered slurry was dried in the oven before NaOH solution was added to produce Na₂SiO₃. By employing the sol-gel method, they gained the SiO₂ in gel form and continued the aging process for 18 hours. Subsequently, the gel was dried in the oven for 12 hours at 80°C to obtain a white SiO₂ powder. The SiO₂ yield pre- and post-acid treatment were 94.2% and 99.4%, respectively. Employing HCl acid as a bleaching agent greatly improved the purity of SiO₂ as only 0.6% of impurities remained.

After that, the superhydrophobic coating was prepared by varying the ratio of SiO₂ to TMCS in the solvent. The solution was sprayed onto a zinc substrate and left for the curing process for 3 h at 150°C. For all of the solvents, it was revealed that an increase in SiO₂ to TMCS ratio would increase WCA up to 180°. The dramatic increase in WCA was due to the difference in solvent polarity and dielectric constant and the addition of SiO₂ improved the adhesion property between coating and substrate.

A recent study on utilizing corn husk as a SiO₂ source to fabricate an almost-superhydrophobic silica coating was prepared by Dahliyanti *et al.* (2022) [34]. They synthesized amorphous SiO₂ nanoparticles by acid leaching and thermal decomposition methods before ball milling to obtain an average particle size of 127 nm. The leaching process was conducted after the corn husk was transformed into powder form. Note that the corn husk powder was introduced into 10% HCl for 2 hours. This process was to ensure that most of the inorganic impurities were removed. The powder was then left to dry in the oven at 150°C. Following acid treatment was the decomposition of leached powder in the atmospheric furnace at a high temperature of 700°C for 2 hours to obtain SiO₂ powder, which then was ball milled to obtain SiO₂ nanoparticles for 3 hours at 500 rpm. XRD results revealed that the SiO₂ extracted from corn husk is in an amorphous phase with little presence of quartz.

To fabricate the almost superhydrophobic coating, they used Methyltrichlorosilane (MCTS) as a surface modifying agent to modify the hydrophilic surface of SiO₂. In toluene, 1 g of SiO₂ nanoparticles was dispersed for 15 minutes, followed by the dropwise addition of MCTS for 2 hours, both steps by ultrasonication. Employing dip coating as the technique to coat the coating onto the substrate for 5 seconds under constant velocity along with repetition for several dips with a few minutes pause in between. Subsequently, the almost superhydrophobic silica coating was fried at room temperature for 1 hour and continued the drying process for 5 hours in a vacuum oven at 150°C. The coating exhibited the highest WCA of 141.448° and the lowest SA of 17° due to the increase in the number of dips.

In 2017, Husni *et al.* [35] developed a superhydrophobic coating sprayed onto concrete cubes by utilizing rice husk ash as a source of SiO₂ filler. They started the coating development by preparing the concrete cubes using water, cement, and fresh concrete as the main materials and let the concrete cure for 28 days. At the same time, rice husk ash was produced by acid leaching using citric and calcination processes in a furnace for 30 minutes at 800°C. After that, the ash was mixed into the HFDS/ethanol mixture for 1 hour before spraying the solution onto concrete cubes at a distance of 10 cm. The superhydrophobic coating recorded the highest WCA and silica composition percentage of 152.3±0.5° and 97.56%, respectively. Along with hydrophobic radical groups like (Difluoromethyl) CF₂ and (Trifluoromethyl) CF₃, SiO₂ particles helped the superhydrophobic coating to exhibit the Cassie-Baxter wetting state. In this state, the air remains trapped between ash particles to further lessen the interaction between water droplets and coatings.

Table 2 summarizes a few experimental evidence of utilizing SiO₂ from biowaste as a source of filler to develop a coating that repels water. Thus, by looking at all the aforementioned literature examples, we can conclude that the presence of SiO₂ compound as a filler in fabricating superhydrophobic coating contributed to the formation of surface roughness which in turn improved the adhesion of the coating on substrate and reduced the wettability. At the same, the addition of SiO₂ enhances the properties of superhydrophobic coating such as robustness and stability of coating in harsh environments.

Table 2. A summary of a few studies on utilizing biowaste as a source of SiO₂ to develop superhydrophobic coating

Biowaste	Treatment method	Maximum WCA (°)	Application	Reference
Rice husk/ cabbage	Alkali treatment/ acid leaching	155±	Self-cleaning, resistance to pH	[36]
Corn straw fiber	Alkali treatment/ acid leaching/ sol-gel	152	Oil and water separation	[37]
Rice husk ash	Thermal treatment/ acid leaching	162	Self-cleaning, high weathering resistance	[38]
Rice husk	Acid leaching/ pyrolysis	161	Oil and water separation	[39]
Bamboo leaf	Acid leaching/ calcination/ alkali treatment	< 150	Sorption isotherm	[40]
Corn straw biogas	Acid leaching/ thermal treatment/ alkali treatment	154.49	Anti-icing, acid and alkali resistance	[41]

4. CONCLUSION

This review paper set out to overview the potential application of biowastes as a source of SiO₂ and its application in superhydrophobic coating. It is highly relevant to always discover the potential source of SiO₂ production as most commercial SiO₂ is highly expensive. Note that biowaste is a green and affordable source of SiO₂. A few SiO₂ extraction methods from different types of biowaste suggested a high percentage yield of SiO₂.

Although there is an extraction method that uses dangerous chemicals, another economical and safe alternative is being implemented. The extracted silica also becomes a sustainable and cheap filler for fabricating superhydrophobic coating.

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