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From bamboo leaf to aerogel: Preparation of water glass as a precursor

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ABSTRACT

In this study, water glass was synthesized from bamboo leaf as a precursor to produce silica aerogel. Bamboo leaf was combusted to produce bamboo leaf silica (BLS) and reacted with sodium hydroxide to form a water glass solution. The effects of the processing parameters such as the temperature, the time, and the agitation speed on the silica yield in water glass were studied. These processing parameters were optimized based on the regressed correlation to synthesize water glass. It was found that bamboo leaf contains approximately 20 wt.% of silica, which is higher than the silica content in rice husk. Characterizations of BLS confirmed that it has identical purity, amorphicity and chemical nature as rice husk silica. Optimization study also showed that BLS can be completely dissolved in NaOH in at least 2 h at 30 °C and an agitation speed of 20 rpm, depending on the combination of the parameters.

1. Introduction

Silica aerogel is well known as the lightest solid that consists of amorphous 3-dimensional silica network with air occupying >96% of its volume [1]. Because of its high porosity, silica aerogel exhibits many attractive properties including high specific surface area (500–1200 m^2 g^{-1}) [2], low thermal conductivity (0.005–0.015 Wm^{-1} K^{-1}) [2,3], ultra-low dielectric constant ($k = 1.0$–2.0) [4–6] and low refractive index (1–1.08) [2,7]. These special properties make aerogel a suitable material for thermal insulation (because of the low thermal conductivity), acoustic barriers (because it is highly porous), super capacitors (because of the low dielectric constant), catalytic support (because of the high specific surface area) and Cherenkov detector in high-energy physics (because of the low refractive index) [8].

Silica aerogel synthesis commonly involves either water glass or alkoxides as the precursor. The former was originally used by Samuel Kistler [9] to produce the first aerogel, whereas the latter is currently widely used because it requires fewer solvent exchange steps than the former. Water glass is conventionally manufactured by reacting sodium carbonate with silicon dioxide in the molten state. Because of the high temperature involved in this process, the market price of water glass makes it unattractive as a precursor. Furthermore, using alkoxides as a precursor also has drawbacks. In addition to being costly, alkoxides such as tetramethoxysilane (TMOS) and tetraethoxysilane (TEOS) are hazardous; in particular, TMOS can cause blindness [2].

Amorphous silica can be synthesized by sputtering [10–12] and bio-extraction [13–16]. Bamboo leaf was previously reported by other researchers [17,18] to contain 13–41 wt.% of silica depending on the bamboo species, the climate and the geographical influences. Extraction of silica from bamboo leaves using acids was previously attempted by other researchers [19]. Hence, in this work, water glass was prepared from bamboo leaves as a cheap source to compensate the cost of additional solvent exchange in the aerogel synthesis. Bamboo leaf is an agricultural waste that is commonly disposed in paper-pulp production. In Brazil, this waste contributes to 190 kT of waste per year and is normally burnt in open landfill [20], which directly causes airborne pollutants. In Asia, approximately 10 million tons of bamboo is harvested every year in China, India and Japan [21], which generates large quantities of bamboo leaf as waste. Thus using bamboo leaf as the precursor to synthesize aerogel can substitute the use of expensive raw materials, and helps to reduce environmental pollution. The synthesis of aerogel from bamboo leaf is not new because other researchers have previously attempted a similar approach by using rice husk [22–24] i.e., another agriculture waste that is known to have high silica content. Hence, rice husk was used throughout this work as a comparison to bamboo leaf.

2. Methods

2.1. Materials

The bamboo leaf used in this study belongs to the *Bambusa heterostachya* species, which is locally known as *Buluh galah*. The bamboo leaves were obtained from the forest park in the University of Malaya and the Forestry Research Institute of Malaysia (FRIM), Kuala Lumpur. The rice husk was obtained from the Padiberas Nasional (BERNAS) Berhad rice mill, Malaysia. Hydrochloric acid (HCl, 2 mol L^{-1}) and sodium hydroxide (NaOH, 2 mol L^{-1}) were purchased from R&M Chemicals (Malaysia).

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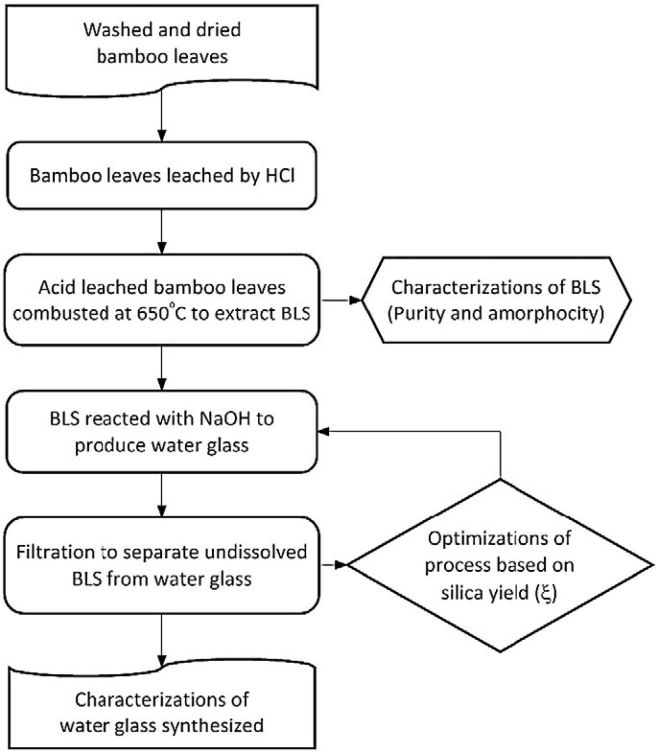

Fig. 1. Process flow of the synthesis of water glass from bamboo leaf.

Table 1
Selected processing parameters that may affect the silica yield (ξ) in water glass.

Parameter	Range
Time, t (h)	24 - 72
Temperature, T (°C)	30 - 60
NaOH concentration, C (mol L^{-1})	2 - 6
m_{BLS}/V_{NaOH} ratio, R (g L^{-1})	12 - 60
Agitation speed, A (rpm)	0 - 200

2.2. Experimental procedure

Fig. 1 shows the workflow of this study, which includes synthesis, characterizations and optimization of the processing parameters in the water glass synthesis. Withered bamboo leaves (length > 20 cm) were initially washed with deionized water and dried in the oven at 90 °C for 72 h. Then, all dried leaves were cut into smaller pieces (approximately 8 cm) for easy leaching and combustion. Each sample (50 g) was combusted in air using muffle furnace at 15 °C min^{-1} from room temperature to 650 °C.

This process was followed by isothermal heating at 650 °C for 4 h to extract the silica. The silica produced in the combustion are termed bamboo leaf silica (BLS) and rice husk silica (RHS). All samples were combusted at 650 °C to prevent crystallization of silica to form cristobalite and tridymite [16,25]. The weight percentages of BLS and RHS in biomass were calculated and reconfirmed using a thermogravimetric analysis (TGA, TA Instrument Q500). The TGA was performed in nitrogen atmosphere with three heating rates of 5, 10 and 20 °C min^{-1}; then isothermal heating was performed at 650 °C for 4 h. Finally, the samples were heated up to 1000 °C at 10 °C min^{-1}.

The purity of the BLS was determined using an energy dispersive X-ray analysis (EDX, INCA Energy 400) coupled with FESEM (FEI Quanta 200F). An X-ray diffractometer (XRD, PANalytical Empyrean) with a 2θ range of 10°–90° (step size 0.026°, $K_{\alpha 1} = 1.5406$ A, Cu anode) was also used to detect any formation of combustion-induced crystallized silica in the BLS. Then, water glass was synthesized by reacting BLS with the sodium hydroxide (NaOH) solution. The silica yield in the formed water glass was determined using inductively coupled plasma-atomic emission spectroscopy (ICP-AES, Perkin Elmer Optima 7000 DV); the silica yield (ξ) is defined as in Eq. (1):

$$\text{Yield}, \xi = \frac{C_{Si} V_{NaOH}}{\phi m_{BLS}} \left(\frac{M_{SiO_2}}{M_{Si}} \right) \quad (1)$$

where C_{Si} is the silica concentration in water glass, which is determined using ICP and measured in g L^{-1}; V_{NaOH} is the NaOH volume reacted with BLS, measured in L; ϕ is the purity of BLS, which is determined using EDX and measured in wt.%; m_{BLS} is BLS mass in the reaction, measured in g; M_{SiO_2} is the molar mass of silica i.e., 60.084 g mol^{-1}; and M_{Si} is molar mass of silicon i.e., 28.086 g mol^{-1}.

To identify the processing parameters that affected the silica yield in water glass, the reactions of BLS with NaOH were repeated by varying the parameters for the range stated in Table 1. Then, the processing condition was optimized to achieve a high silica yield in water glass. The water glass with optimized silica yield (ξ) was characterized to study its elemental composition, density and pH.

3. Results

3.1. Bamboo leaf silica (BLS)

The distribution of silica in bamboo leaf can be clearly observed in the remaining ash after combustion, which is shown in Fig. 2b. The FESEM image in Fig. 2c shows that skeleton of the bamboo leaf ash remains intact after combustion.

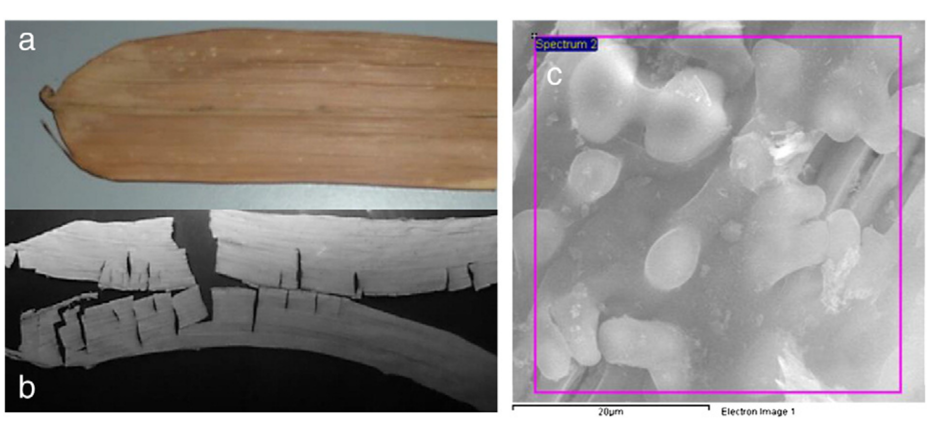

Fig. 2. Visual comparison of a bamboo leaf (a) before and (b) after combustion, and (c) under FESEM after combustion.