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Submission date: 22-Feb-2024 06:10PM (UTC+0700)

Submission ID: 2301498189

File name: Artikel-20_Pasymi.pdf (671.93K)

Word count: 4617

Character count: 24277

ACID WASH INFLUENCES ON PHYSICOCHEMICAL CHARACTERISTICS OF BAMBOO LEAVES ASH

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Article history

Received

7 December 2021

Received in revised form

4 May 2023

Accepted

4 August 2023

Published Online

21 August 2023

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Graphical abstract



Abstract

Silica production from sandstone requires tremendous fossil resources that threaten our environment. Hence, greener resources from biomass should be utilized. One of them is from bamboo leaves which contain rich silica in ash. Even so, acid wash should be applied to achieve higher silica purity. This work aims to investigate the acid wash influences on the physicochemical characteristics of bamboo leaves ash (BLA). The treatment used 1 mol/L HCl under 1-h, 2-h, and no acid wash as a control. The BLA crystallography indicates a fully amorphous phase of silica. Interestingly, 1-h acid wash precisely reduces the silica purity from 95.35% to 94.74%, but it then increases to 96.06% under 2-h acid wash. It is also notified that the longer acid wash duration could alleviate leaves' mechanical strength. After calcination, consequently, a smaller average particle size of BLA was nominated under 2-h acid wash (6.32 μm). It was then followed by 1-h acid wash (21.32 μm) and no acid wash (149.44 μm). The 2-h acid wash is concluded able to intensify silica purity as well as reduce the particle size of BLA. Finally, acid wash treatment becomes important to facilitate further BLA extraction in order to achieve high purity of silica.

Keywords: Amorphous, biomass, crystalline, mesoporous, renewable

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1.0 INTRODUCTION

Silica is the most abundant oxide in the earth's crust [1, 2] with a proportion of 47-60% [3-6]. It is an essential material for many industrial purposes, i.e. glass, adsorbent, catalyst support, cement, building, electronics, health care products, medical or drugs, and silica gel [7-12]. Silica is also widely utilized for its non-conductive, diamagnetic, and stable properties [2, 4, 13]. Unfortunately, silica production still depends on non-renewable raw materials which conventionally employ sandstone [14-17]. Moreover, the production spends a lot of fossil fuels because of the operation condition of up to 1500°C and 4 GPa [2]. The extravagant utilization of fossil fuels is proven harmful and threatens the environment as well as human beings [18-22]. Thus, silica production from sustainable and renewable resources, such as biomass, should be explored. The other advantages of biomass lie in its green and mild processes as well as less energy consumption [23-27].

The silica from biomass is hereinafter referred to as bio-silica. One potential biomass for silica source is derived from bamboo leaves. Bamboo itself is one of the most commonly-grown woody plants in Indonesia [28]. Currently, the well-utilized part is rods which are used for construction needs, handicrafts, furniture, paper industries, and angklung [29, 30].

On the other hand, fallen and yellowish bamboo leaves on the ground are used for solid bio-fertilizer, planting media, or animal feed, but most of them are often neglected and still not utilized [31-34]. If bamboo leaves are appropriately exploited through combustion and chemical extraction [35, 36], they can serve as a bio-silica source since they contain ash and also contain rich silica in the ash [12, 37].

The use of bamboo leaves ash (BLA) for silica from the synthesis of aluminosilicate zeolite was explored by Ng et al. (2017), but the process used hydrothermal [8]. Likewise, BLA extraction followed by magnesiothermic reduction was carried out to acquire silicon for battery purposes [13]. Other than that, the research on thermal and ash characterization of bamboo leaves from Purbasari et al. (2016) revealed that bamboo leaves have the potential for solid fuel, building material, and other engineering purposes [37]. Meanwhile, Rangaraj and Venkatachalam (2016) have succeeded to synthesize amorphous silica nanoparticles from thermal and chemical processing of bamboo leaves [12]. However, they directly calcinate the bamboo leaves without studying the effects of acid wash treatment.

Actually, acid wash is believed can eliminate alkaline impurities in ash [38] and consequently rectify the silica purity. Up to now, the existing studies regarding the acid wash treatment were mainly subjected to ash from rice husk, sweet sorghum bagasse, white pine, and wheat straw, as served in Table 1. Referring to the mentioned studies, acid wash has been proven to effectively remove oxides from K and Na in ash. Besides, it also can enhance ash pore volume, alleviate ash pore diameter, and alter ash particle size and morphology.

Nevertheless, the research on bamboo leaves acid wash is still limited to be disclosed. Therefore, this work aims to investigate the effects of acid wash on the physicochemical characteristics of BLA. The examined parameters comprise morphology, silica purity, particle size, crystallography, surface area, pore diameter, and pore volume.

Table 1 Existing studies on the influences of acid wash on several biomass ashes

Biomass	Acid Wash Treatment	Results	Ref.
Rice husk	Using: 1. 72% H ₂ SO ₄ at 30°C for 1 h and 4% H ₂ SO ₄ at 121°C for 1 h 2. 10% HCl at 90°C for 1 h 3. 1 mol/L H ₂ C ₂ O ₄ at 200°C for 1 h under CO ₂ ambiance 4. 100% 1-butyl-3-methylimidazolium hydrogen sulfate at 130°C for 36 h	Intensified silica purity from 94.7% to: 1. 99.6% 2. 98.0% 3. 98.0% 4. 99.5%	[39]
Sweet sorghum bagasse	Using 0.1 mol/L HCl at 25°C for 4 h with continuous stirring at 800 rpm	Removed alkaline impurities (oxides from K, Na, Mg) > 90%	[40]
White pine	Using 7% HCl (1), 7% H ₃ PO ₄ (2), and 7% H ₂ SO ₄ (3) at room temperature for 2 h	Removed alkaline impurities up to 75% (1), 72% (2), and 71% (3)	[41]
Rice husk	Using 7% HCl (1), 7% H ₃ PO ₄ (2), and 7% H ₂ SO ₄ (3) at room temperature for 2 h	Removed alkaline impurities up to 83% (1), 76% (2), and 82% (3)	[41]
Wheat straw	Using 5% H ₂ SO ₄ at room temperature for 2 h	1. Decreased surface area from 1.19 to 1.12 m ² /g 2. Alleviated pore diameter from 3.44 to 3.22 nm 3. Increased pore volume from 0.12 to 0.26 cc/g 4. Enhanced yield from 8.34 to 17.25%-wt	[42]
Bamboo leaves	Using 1 mol/L HCl for 1-h, 2-h, and no acid wash as a control		This Work

2.0 METHODOLOGY

2.1 Materials

Fallen and yellowish bamboo leaves from bamboo plants at Institut Teknologi Bandung (ITB) Jatinangor campus, West Java, Indonesia were collected (Figure 1a). An analytical grade of HCl for acid wash was purchased from Merck. The demineralized water for rinsing purposes was acquired from the ITB Bioenergy and Chemurgy Instructional Laboratory. A Thermo Scientific Heratherm OMH180 laboratory oven was applied for drying.

2.2 Bamboo Leaves Ash (BLA) Production

The collected bamboo leaves (Figure 1b) were first ground to form bamboo leaves powder (BLP), as shown in Figure 1c. Afterward, 100 g of BLP was treated through acid wash using 1 mol/L of HCl for 1 h, 2 h, and no acid wash as a control. Subsequently, it was filtered, rinsed with demineralized water, and then dried in the oven. Finally, treated BLP was calcined in a muffle furnace at 700°C for 2 h to produce BLA. The procedure is given in Figure 2.

2.3 Characterizations methods

As much as 2 g of bamboo leaves were subjected to Apollo L thermogravimetry analysis (TGA) instrument

with operating temperature of 900°C, gas flowrate of 3 L/min, and heating rate of 10°C/min. The morphology, silica purity, and particle image of BLA were assayed under JEOL JSM 6510 LA scanning electron microscope energy dispersive spectroscopy (SEM-EDS) apparatus which employed working distance from 9–13 mm, 15 kV of voltage, and 1000X of magnification.

The particle size determination was quantitatively measured using ImageJ software. The particle image from SEM results was initially imported into the software. Subsequently, the image was scanned and the line was created on that image for measurement scale. The particle size was then calculated by comparison principle and the replication was done 8X for every treatment.

In addition, the BLA crystallography was recorded under Rigaku MiniFlex 600 X-ray diffraction (XRD) instrument which utilized Cu- α and diffraction angles of 5–90°. Afterward, a Quantachrome TouchWin v1.2 instrument following Brunauer-Emmett-Teller (BET) and Barret-Joyner-Halenda (BJH) methods were utilized for BLA surface area, pore diameter, and pore volume analyses. The sample was vacuum-degassed prior to liquid nitrogen injection. The nitrogen was adsorbed through Langmuir isotherm mechanism at 77.35 K under relative pressures of 0.02–0.3.



Figure 1 (a) Bamboo plants; (b) Collected bamboo leaves; (c) Ground bamboo leaves

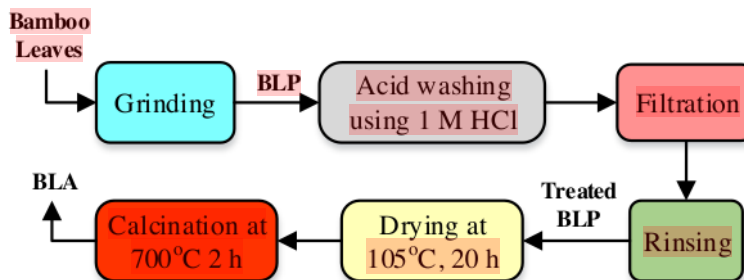


Figure 2 Bamboo leaves ash production

3.0 RESULTS AND DISCUSSION

3.1 Bamboo Leaves Proximate Analysis

The bamboo leaves proximate analysis results are presented in Table 2. The ash proportion in the biomass is measured as much as 25.86%-wt. The observed ash content from bamboo leaves is relatively high compared to rice husk and rice straw which are in the range of 18.67-20.26%-wt [43,44]. In accordance with the EDS result, the silica in BLA occupies 95.35%-wt and the rest are recorded as 3.67 %-wt CaO and 0.98% K₂O. Therefore, it is proven that bamboo leaves become a potential bio-silica resource due to their high ash content and rich silica in ash.

Table 2 Bamboo leaves proximate analysis (%-wt dry basis)

Parameters	Value
Moisture Content	-
Volatile Matter	60.60
Ash	25.86
Fixed Carbon	13.54

3.2 BLA Crystallography

The crystallography for BLA under 2-h acid wash reveals a peak appearance at a diffraction angle of 21.8°, as in Figure 3, which interprets amorphous silica. Amorphous silica is silica with random lattice order whereas crystalline silica lattices are well arranged [45]. It is also known as silica without crystalline phase in 3D particle structure as defined by no appearance on definitive line in XRD measurements [46, 47].

Furthermore, it is exhibited that the BLA has a low amount of oxides or mineral impurities such as NaCl, SiO₂, K₂O, Fe₂O₃, and CaO. Amorphous silica in BLA reflects a suitable raw material for producing bio-silica because it is more harmless than crystalline silica, which is highly toxic and could cause silicosis [12, 45, 48].

3.4 Acid Wash Influence on BLA Particle Size

According to this work, BLA morphology has more fracture lines and wrinkled structures under longer duration of acid wash as depicted in Figure 4. This happens due to longer acid wash having high possibility of surface erosion in BLA particle which

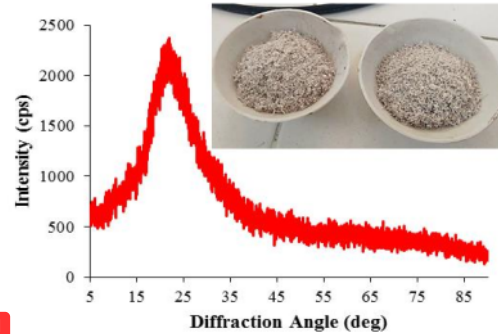


Figure 3 XRD result of bamboo leaves ash [inserted figure: appearance of BLA for 2-h acid wash]

3.3 Acid Wash Influence on Silica Purity

The BLA produced was dominated by white color as shown in Figure 3. The appearance of the BLA color qualitatively indicated high silica content. The purity was then determined quantitatively by EDS analysis and the results of which are tabulated in Table 3. The silica purity in BLA ranged from 94.74 to 96.06% which again strengthens that BLA is lucrative as a silica source from biomass.

Based on the results in Table 3, there is no significant difference in silica purity for all treatments. However, the purity increases to 96.06% under 2-h acid wash. It is caused by most of the alkaline impurities being successfully dissolved in acid [12,16,38,49]. Compared with other biomass sources, under no acid wash, BLA with is quite competitive with rice husk ash (94.7% of silica purity) [39] and sugarcane ash (88.68% of silica purity) [48].

Table 3 Silica purity under varying acid wash durations

Acid Wash Duration (h)	Silica Purity (%-wt)
0 (no acid wash)	95.35
1	94.74
2	96.06

results in a smaller size of obtained BLA particle [42]. This qualitative explanation is reinforced by the BLA particle size, as outlined in Table 4. For each treatment given, intensifying acid wash duration from 0 h to 2 h results in a smaller BLA particle size for both ranged size and averaged size.

Table 4 BLA particles size under varying acid wash durations

Acid Wash Duration (h)	Ranged Particle Size, 8X Replications (µm)	Averaged Particle Size (µm)
0 (no acid wash)	20.69-168.97	149.44
1	10.47-109.30	21.32
2	2.56-56.41	6.32

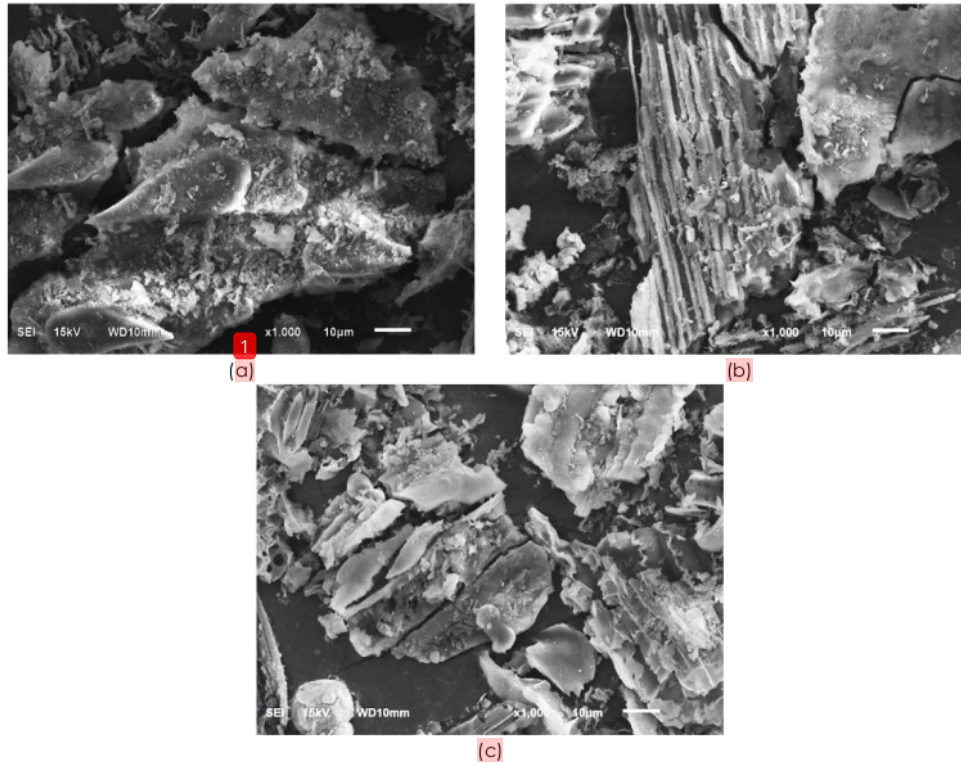


Figure 4 Bamboo leaves ash morphology: (a) without acid wash; (b) 1-h acid wash; (c) 2-h acid wash

3.5 Acid Wash Influence on BLA Surface Area, Pore Diameter, and Pore Volume

The acid wash has also an effect on producing BLA particles with higher surface area compared with no acid wash [42]. It is reflected by BLA with higher surface area, 211.1–257.5 m^2/g vs. 60.9 m^2/g . This implies acid wash is proven to eradicate major alkaline impurities that are trapped in the pore structure [16, 38, 40].

Additionally, the higher surface area has a consequence of mesoporous particles with a smaller pore diameter [20, 50]. In consequence, the BLA pore diameter with acid wash (2.77–3.26 nm) is smaller than BLA without acid wash (5.22 nm), as given in Table 5.

The BLA particle surface erosion due to acid wash turns out to wrinkle BLA structure and leads to the enhancement of pore volume. These conditions

make BLA possesses more pores thus shrinking their pore diameter [42]. As the number of pores increases, the pore volume and surface area of the BLA is also larger [20, 51].

Following this work, the acquired BLA under 2-h acid wash treatment is nominated to achieve the desired physicochemical characteristics in terms of mesoporous, highest purity, highest surface area, smallest pore diameter, and largest pore volume.

For comparison, another work on rice husk ash acid wash with HCl 10%-w at 90°C for 1 h slightly increased the pore volume from 0.17 cc/g to 0.178 cc/g but decreased the surface area from 99.2 m^2/g to 65.6 m^2/g . Based on their work, the acid wash with HCl still can not completely remove alkaline impurities and this evidence was strengthened by the XRD pattern that has other peaks beyond 22° [39].

Table 5 Surface area, pore diameter, and pore volume of BLA under varying acid wash durations

Acid Wash Duration (h)	Surface Area (m^2/g)	Pore Diameter (nm)	Pore Volume (cc/g)
no acid wash	60.9	5.22	0.16
1	211.1	3.26	0.34
2	257.5	2.77	0.36

4.0 CONCLUSION

The produced bamboo leaves ash (BLA) is mesoporous and amorphous with silica purity of up to 96.06%-wt. The longer duration of acid wash treatment decreases the particle size and pore diameter, and also increases the surface area and pore volume of BLA. Under 2-h of acid wash, the average particle size reduces from 149.44 μm to 6.32 μm , the pore diameter decrease from 5.22 nm to 2.77 nm, the surface area increase from 60.9 m^2/g to 257.5 m^2/g , and the pore diameter increase from 0.16 cc/g to 0.36 cc/g . It is concluded that acid wash treatment has positive influences that facilitate further BLA extraction to obtain high purity of bio-silica.

Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

Acknowledgements

This work was financially supported by P3MI ITB. The authors also wish to thank the Faculty of Mathematics and Natural Sciences, ITB for SEM-EDS analysis and the physical chemistry laboratory for XRD analysis.

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