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Influences of pretreatment, extraction variables, and post treatment on bench-scale rice husk black ash (RHBA) processing to bio-silica

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Abstract

Abundant availability and large silica content in rice husk black ash (RHBA) make the use of it very interesting to study. Many works only deal with lab-scale rice husk ash extraction while the studies on bench-scale RHBA extraction are still limited. This study, hence, presents the influence of pretreatment, extraction variables, and posttreatment on bench-scale RHBA processing to bio-silica. The pretreatment through acid leaching was carried out using HCl. The extraction was implemented under varying process variables such as alkaline-to-feed ratio ($R_{A/F}$), extraction duration, and acid precipitation agent. According to this study, the highest extraction yield up to 98% was gained under $R_{A/F}$ 6 g/g and 1-h extraction. The amorphous bio-silica had an asymmetric siloxane bond and white appearance with a purity exceeding 95% and surface area up to 406.98 m^2/g. Meanwhile, precipitation under HCl and H_2SO_4 had little impact on product purity and surface area. This study exhibits that acid leaching is executed to release mineral impurities but is still not sufficient to remove the remaining carbon content in bio-silica. However, the contribution of refining process is able to do so. Moreover, the produced bio-silica is suitable for adsorbent purposes which could adsorb up to 83.5% of 3-monochloropropanediol compound.

KEYWORDS

adsorption, extraction, furnaces, mixing, precipitation, production

1 | INTRODUCTION

Rice husk is one of the rice processing residues, which usually occupies 14–25 wt% from straw-free rice plant.[1–3] To date, the abundant amount of rice husk is only considered as an agricultural waste that is usually disposed of and burned in the landfill to reduce accumulation.[4–6] This treatment can actually harm the environment, animals, and human[2,7]; so further rice husk utilization should be well formulated. Untreated rice husk burning often produces ash with high unburnt carbon content due to incomplete burning as well as the melting of alkaline minerals in ash which entrap and complicate the fixed carbon to undergo further oxidization.[8,9] This kind of ash is quite different from common rice husk ash (RHA), so it is then called rice husk black ash (RHBA),

which is still rare to be exploited.[10–14] Simply, RHA has a grayish to white color while RHBA's color is black to grayish.

On the other side, excessive fossil fuel consumption is imperative to be prevented, and it becomes urgent to utilize the biomass for renewable energy as well as green chemicals.[3,6,15] Nowadays, commercial silica production still consistently depends on it and involves an extremely high temperature process, up to 1500 K, which threatens the environment due to vast CO_2 and NO_x formation.[15–18] Interestingly, rice husk as agricultural biomass contains approximately 15–25 wt% of ash with 80–98 wt% silica content in it.[1,2,19] This makes rice husk utilization to bio-silica become potential for versatile industrial purposes, that is, crystalline silica for ceramic reinforcement,[1] composite reinforcement,[7] cement and concrete,[11] ceramic processing,[7,11,16,20,21] battery,[10,22] adsorbent,[19,21,23,24] and silica monolith for catalyst support or nanocomposites.[24–26] Throughout this fact, RHBA becomes a promising green feedstock candidate to produce bio-silica which is more renewable, eco-friendly, and sustainable compared to the commercial one.[15]

An earlier study on RHA extraction with NaOH yielded 65% silica.[9] Subsequently, a simple method for obtaining silica from RHA acid leaching followed by NaOH extraction showed an increase in amorphous silica recovery from 89% to 91%.[27] Another RHA extraction study under various alkaline-to-feed ratio ($R_{A/F}$) and extraction duration gave the highest silica yield of 82% under $R_{A/F}$ 5 g/g of 1 mol/L NaOH for a 2-h extraction.[28] In contrast, the yield increased from 66.11% to 71.15% at 2 mol/L NaOH of RHA extraction.[29] In 2016, a study still used a similar method to obtain silica from RHA and gave a silica recovery of more than 90%.[21] On the other hand, the silica source from RHA for upgrading the wettability between RHA and aluminum alloy in the advanced ceramic synthesis was also successfully conducted.[1] Meanwhile, the RHA extraction experiment with alkaline under varying acid leaching sequences which was performed by Steven et al.[19] resulted in amorphous and mesoporous green silica with 96.44% of purity and 400.69 m^2/g of surface area.

The silica aerogel synthesis with surface area of 322.5 m^2/g was achieved from RHA under various acid types for precipitation and the addition of tetraethyl orthosilicate (TEOS), ethanol, and n-heptane solvents in gelation process followed by various drying techniques (air, oven, freeze, and vacuum).[30] The preparation of silica aerogel from RHA was also conducted under alkaline extraction and then followed by cation exchange, pH adjustment with NaOH, ethanol solvent replacement, and supercritical drying resulting in uniform silica particle with 729.82 m^2/g of surface area.[31] Aside from that, rice husk acid leaching, calcination, and freeze casting which was carried out by Bahrami et al.[24] succeeded in producing an environmentally friendly amorphous silica monolith with 1.5 m^2/g of surface area and crystalline silica monolith with 151 m^2/g of surface area as further applied for catalyst support or sorption.

Several existing works, as aforementioned, mainly deal with lab-scale RHA extraction, but bench-scale RHBA utilization to produce bio-silica still needs to be discussed as shown in the state of the arts in Table 1. The current study, therefore, proposes to communicate the influence of pretreatment, extraction variables, and posttreatment on bench-scale RHBA processing to bio-silica. The influence of RHBA pretreatment is investigated. The effects of alkaline-to-feed ratio ($R_{A/F}$), extraction duration, and acid precipitation agent on extraction yield and product purity are evaluated. In the end, the importance of posttreatment in the form of refining to meet the silica quality and further product application is also pointed out.

2 | EXPERIMENTAL SECTION

2.1 | Materials and chemicals

RHBA was provided by ITB Bioenergy and Chemurgy Workshop, West Java, Indonesia. Sodium hydroxide was purchased from the local chemical market, while hydrochloric acid and sulfuric acid were procured from Merck. All chemicals used in the study were of analytical grade. Deionized water was acquired from the ITB instructional laboratory.

2.2 | Main process procedures

The experiment was held in triplicate. As much as 200 g of RHBA was initially pretreated using 1 mol/L HCl at a temperature of 100°C for 1 h. The treated RHBA was rinsed with deionized water and was then extracted using 1 mol/L NaOH at a temperature of 120°C under vigorous stirring. The $R_{A/F}$ was varied at 4, 5, and 6 g/g, whereas extraction duration was varied in 1 and 2 h. The extraction solution was then cooled and filtered to remove the residue. The next step was acid precipitation of residue-free extraction solution using HCl and H_2SO_4 without stirring until gel was formed. Afterwards, gel aging occurred for 2 h and the aged gel was then washed and oven dried. Finally, the posttreatment process was applied by using a box-type resistance furnace at 700°C for 2 h. Extraction yield (E_y) is evaluated from measured product mass (m_p), RHBA ash content (Y_A), and RHBA amount (m_RHBA) with the following relation:

TABLE 1 State of the arts for silica production from rice husk derivatives

Raw material	Process	Highest yield (%)	Purity (%)	Surface area (m²/g)	Ref.
Rice husk	Acid leaching + calcination + freeze casting	-	-	1.5 (crystalline) 151 (amorphous)	Bahrami et al.[24]
Rice husk ash (RHA)	Alkaline extraction	65.00	>96	-	Mittal[9]
	Alkaline extraction	82.00	-	-	Ul-Haq et al.[28]
	Alkaline extraction	71.15	-	-	Mehta and Ugwekar[29]
	Alkaline extraction	>90.00	-	-	Todkar et al.[21]
	Pretreatment + alkaline extraction	91.00	93	-	Kalapathy et al.[27]
	Acid leaching + alkaline extraction	-	96.44	400.69	Mysen and Richet[18]
	Pretreatment + alkaline extraction + various solvent replacement + drying technique	-	>97	322.5	Temel et al.[30]
	Alkaline extraction + cation exchange + solvent replacement + supercritical drying	-	-	729.89	Cui et al.[31]
Rice husk black ash (RHBA)	Pretreatment + alkaline extraction + posttreatment (refining)	To be investigated			This study

$$E_y = \frac{m_p}{Y_A \times m_{RHBA}} \times 100\%. \quad (1)$$

2.3 | Analytical procedures

The proximate analysis using ASTM D3172-13 standard method is applied to investigate the ash content. It was done in triplicate for RHBA and once for rice husk. Another simple method for RHBA ash content determination was also conducted by gravimetric analysis to ensure the proximate analysis result. The RHBA samples were initially weighed, and then the mass after calcination at 700°C for 2 h was also measured. The mass difference before and after calcination is defined as RHBA ash content, and the mass loss is reflected as loss on ignition (LOI).[7,32,33]

The X-ray diffraction (XRD) pattern measurement was assisted with a Bruker D8 Advance X-Ray Diffractometer, Cu-Kα radiation for $\lambda = 1.5406$ Å, and 40 kV to 35 mA of generator. The sample should be ground first until 200 mesh and was then scanned with diffraction angles from 5° to 90°. Product purity was estimated by semiquantitative analysis using Rigaku ZSX Primus III⁺ wavelength dispersive X-ray fluorescent (WD-XRF) spectrometer which is equipped with 50 kV and 4 mA of palladium material X-ray generator. All samples were

pretreated first by a polyvynil chloride (PVC) ring pelletizer and were then dried using a Myllar oven.

An Alpha Bruker Fourier transform infrared (FTIR) spectrometer with attenuated total reflection (ATR) and KBr method was used to identify the product functional group and chemical bonding behavior. Product morphology was captured with Jeol JSM-6510A analytical scanning electron microscope (SEM), and the sample preparation was employed under Jeol EC-32010CC vacuum carbon coater for 5 min to enhance the conductivity. Finally, product surface area was characterized under Brunauer–Emmett–Teller (BET) Quantachrome NovaWin v11.0 instrument at 77.3 K with a relative pressure P/P_0 ranging from 0.02 to 0.32.

3 | RESULTS AND DISCUSSION

3.1 | Raw material analysis

The proximate analysis, as tabulated in Table 2, implied that rice husk transformation to RHBA exhibits a steep decrease from 58.31% to about 6% in the volatile matter. However, the fixed carbon only changes gently, and in consequence, RHBA has a blackish appearance because it still contains the remaining carbon. This happens when untreated rice husk is not completely burned which is predominantly caused by the melting of alkaline minerals in ash and poor mixing in combustion.[8,9,17,34]

TABLE 2 Proximate analysis result

Proximate analysis	% mass, air-dried basis (a.d.b)			
	Rice husk	RHBA		
Moisture content	9.13	5.36	5.14	7.70
Volatile matter	58.31	5.98	5.68	6.50
Fixed carbon	12.12	10.83	14.07	18.24
Ash	20.44	77.83	75.11	67.56

Abbreviation: RHBA, rice husk black ash.
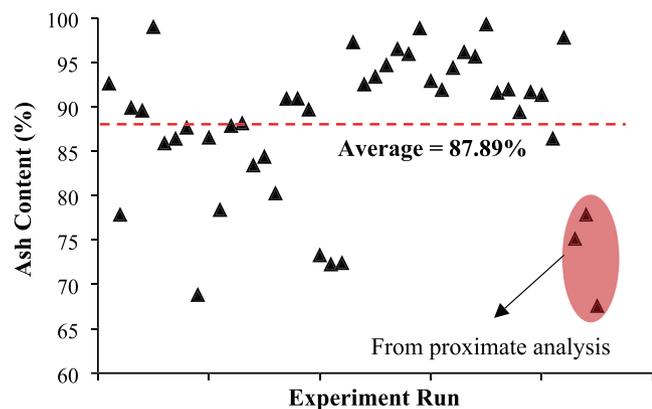
Figure 1. RHBA ash content

FIGURE 1 Rice husk black ash (RHBA) ash content

agitated and allows the NaOH infiltration into the RHBA porous matrix.[19] This condition provides the perfect and intense contact of RHBA and NaOH, which could enrich the silica extracted amount. RHBA extraction under NaOH has the superiorities of cheaper price compare with extraction under KOH[35] and lower hazard risk compares with extraction under ammonium fluoride.[36] The average extraction yield was depicted in the histogram as served in Figure 2.

Herein can be seen that the highest extraction yield of about 97%–98% is achieved at $R_{A/F}$ 6 g/g and 1-h extraction duration, which is relatively higher than other studies.[9,21,27–29] The acquisition of the silica is influenced by $R_{A/F}$ and extraction duration. Meanwhile, the use of HCl or H_2SO_4 as a precipitation agent does not give a significant difference in extraction yield. This extraction produces a sodium silicate solution which follows the reaction in Equation 2. Sodium silicate is a limiting reactant that is fully consumed when complete precipitation is reached. The sodium silicate is then precipitated with acid until pH below 10 to form silica in gel form.[2,37] The reaction follows Equation 3 for precipitation with HCl and Equation 4 for precipitation with H_2SO_4.[21,23,28] Both reactions are thermodynamically spontaneous, and the neutral solution pH indicates the complete precipitation.

$$2NaOH_{(aq)} + SiO_{2\,in\,ash} \rightarrow Na_2SiO_{3\,(aq)} + H_2O_{(l)} \qquad \Delta G = -83.4\,kJ/mol, \qquad (2)$$

$$Na_2SiO_{3\,(aq)} + 2HCl_{(aq)} \rightarrow 2NaCl_{(aq)} + \mathbf{SiO_{2\,(gel)}} + H_2O_{(l)} \qquad \Delta G = -209.8\,kJ/mol, \qquad (3)$$

$$Na_2SiO_{3\,(aq)} + H_2SO_{4\,(aq)} \rightarrow Na_2SO_{4\,(aq)} + \mathbf{SiO_{2\,(gel)}} + H_2O_{(l)} \qquad \Delta G = -109.2\,kJ/mol. \qquad (4)$$
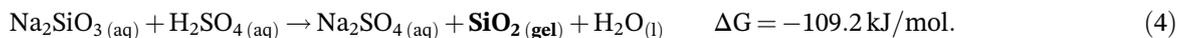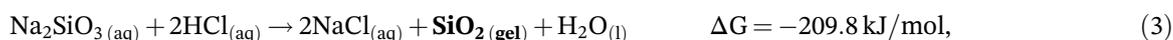
To convince the RHBA ash content, an addition of 42 gravimetric experiments was held, and the result is depicted in Figure 1. The average ash content for RHBA that is used in this study (Y_A) is 87.89%, which supports the evidence of blackish appearance.

3.2 | Influence of pretreatment and extraction variables

Besides impurities removal, acid leaching pretreatment facilitates RHBA particle disruption, which is easier to be

For $R_{A/F}$ 4 g/g, increasing the extraction duration from 1 to 2 h increases the extraction yield from 32.43% to 39.17% for HCl precipitation and from 33.11% to 39.48% for H_2SO_4 precipitation. A similar trend under $R_{A/F}$ 5 g/g gives an enhancing extraction yield from 68.97% to 85.89% for HCl precipitation and from 67.38% to 84.42% for H_2SO_4 precipitation. Referring to Equation 2, more NaOH could shift the reaction to produce more sodium silicate. This phenomenon inherently enhances the bio-silica extracted as informed by reactions in Equations 3 and 4. On the other hand, the longer extraction duration promotes more intense contact

FIGURE 2 Extraction yield on various $R_{A/F}$ and extraction duration for HCl and H_2SO_4 precipitation
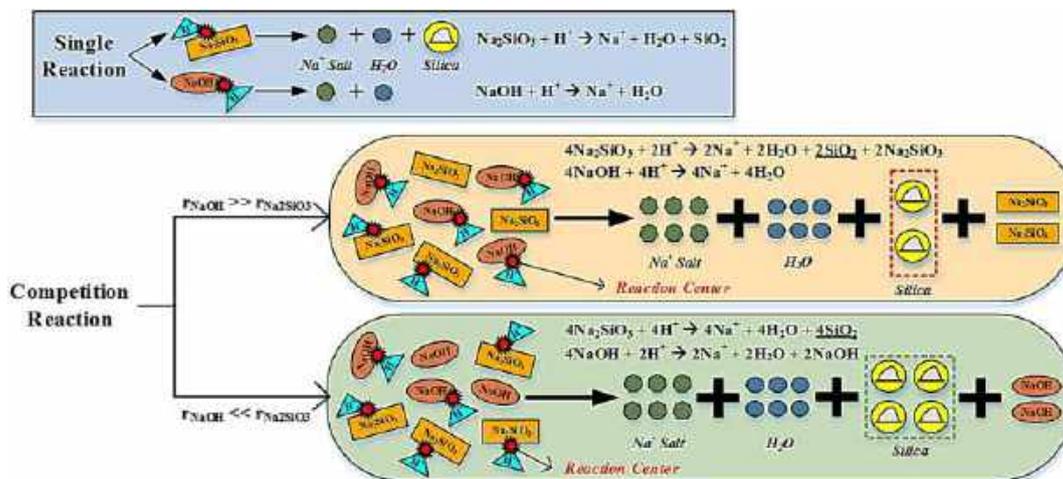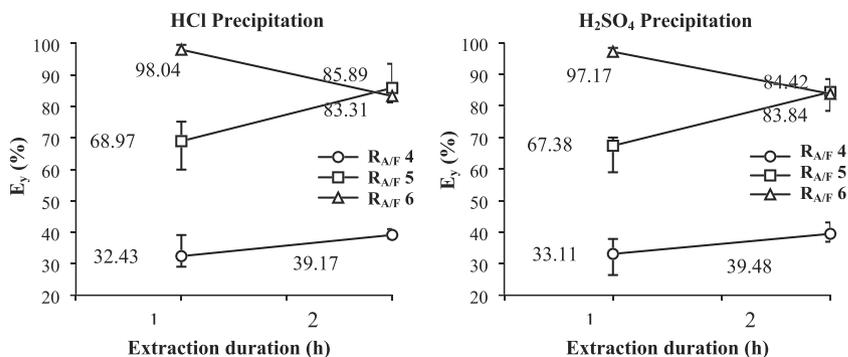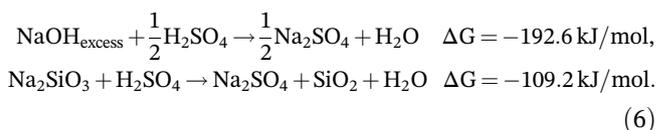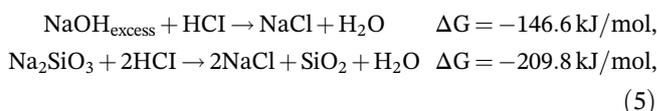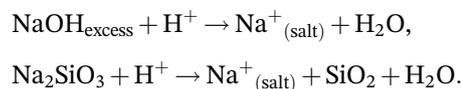

FIGURE 3 Illustration for the competition reaction mechanism

between RHBA and NaOH which also allows more bio-silica to be extracted.[38] An opposite phenomenon occurs in extraction under $R_{A/F}$ 6 g/g. It shows a lesser extraction yield by 15% from a 1- to 2-h extraction. Actually, only a few reports are addressed on decreasing yield phenomena. According to the experiment from Ma et al.,[39] there is a slight decreasing yield when the extraction is performed with 4 mol/L NH_4F and the extraction duration is longer than 2 h. They explain that silica in RHA at least needs 2 h to dissolve away.

In accordance with the result, this study found that the extraction under $R_{A/F}$ 4 and 5 g/g needs at least 2 h while the extraction under $R_{A/F}$ 6 g/g needs at least 1 h. The extraction with $R_{A/F}$ 6 g/g for 2 h gives a slightly similar extraction yield as under $R_{A/F}$ 5 g/g for 2 h. The proposed hypothesis on the decreasing yield phenomenon lies in the excess NaOH in the sodium silicate solution. In such a way, there might be a competition reaction between acid with either excess NaOH ($r_{NaOH} \gg r_{Na2SiO3}$) or sodium silicate ($r_{NaOH} \ll r_{Na2SiO3}$), which follows the mechanism in Equation 5 for HCl precipitation and Equation 6 for H_2SO_4 precipitation.

$$NaOH_{excess} + HCl \rightarrow NaCl + H_2O \quad \Delta G = -146.6\,kJ/mol,$$
$$Na_2SiO_3 + 2HCl \rightarrow 2NaCl + SiO_2 + H_2O \quad \Delta G = -209.8\,kJ/mol,$$
$$(5)$$

$$NaOH_{excess} + \frac{1}{2}H_2SO_4 \rightarrow \frac{1}{2}Na_2SO_4 + H_2O \quad \Delta G = -192.6\,kJ/mol,$$
$$Na_2SiO_3 + H_2SO_4 \rightarrow Na_2SO_4 + SiO_2 + H_2O \quad \Delta G = -109.2\,kJ/mol.$$
$$(6)$$

In general,

$$NaOH_{excess} + H^+ \rightarrow Na^+_{(salt)} + H_2O,$$
$$Na_2SiO_3 + H^+ \rightarrow Na^+_{(salt)} + SiO_2 + H_2O.$$

Both reactions are spontaneous at the precipitation temperature. If the excess NaOH undergoes the reaction with acid, the remaining acid in the solution becomes low and inadequate to precipitate all sodium silicates. This condition might decrease the bio-silica extracted amount. The competition reaction mechanism is illustrated in Figure 3.

3.3 | Effects of posttreatment on product characteristics

Acid leaching is an important pretreatment process to suppress the content of impurities, but this still could not drain out the carbon content so the bio-silica appearance is different from the usual.[9] Carbon is an inert element and could solely be removed by thermal oxidization as long as it is not entrapped in the melted alkaline minerals which often begins to occur at 350°C.[8] This posttreatment is executed at 700°C for 2 h and is able to transform the black product to white, as shown in Figure 4. It means that the carbon has been completely oxidized because the alkaline minerals in the RHBA have been mostly eliminated by pretreatment.[8] Refining under higher temperature and longer duration should be avoided for it is believed to provoke crystalline silica formation[2,19,24,25] which could harm human health.[4,5,12,16,17,34,40]

The XRD pattern for RHBA and bench-scale bio-silica product from $R_{A/F}$ 6 g/g for a 1-h extraction is shown in Figure 5. The RHBA peaks that appear on $2\theta = 35°$, $49°$, and $58°$ indicate the presence of aluminum oxide, potassium oxide, and sodium oxide.[1,41–46] In addition, extracted bio-silica from this study is amorphous and absent from the crystalline phase, which is expressed from the wide broad peak attributed at diffraction angle $22°$.[1,2,19,47] The product has a silica content exceeding 95% under HCl and H_2SO_4 precipitation, whereas previously only 76.59% in RHBA, as outlined in Table 3. Based on XRF analysis, the product has major impurities of K_2O and Na_2O with a value below 1.5%, and this is in line with the XRD pattern analysis. The content of low impurities in the product is due to the contribution of RHBA acid leaching.[5,19,44,47] It is also observed that there is no significant difference in product purity between HCl and H_2SO_4 precipitation.

Moreover, the recorded FTIR peaks for RHBA and bio-silica are almost similar, as seen in Figure 6. The most dominant functional group observed in the bio-silica obtained is Si–O bonding.[27,47–49] An asymmetric siloxane bond, Si–O–Si, is attributed to the absorption band at wavenumber 1100 cm^{-1}, and yet, symmetric siloxane bond with vibration and stretching are also found at 800 and 950 cm^{-1}, successively.[2,5,20,22,50–52] Again, the FTIR result confirms that the product is in absence of crystalline phase because no peak appears at 620 cm^{-1}.[53] The FTIR spectrum of the bio-silica from this study is also in good agreement with the silica spectrum obtained from other studies.[54,55]
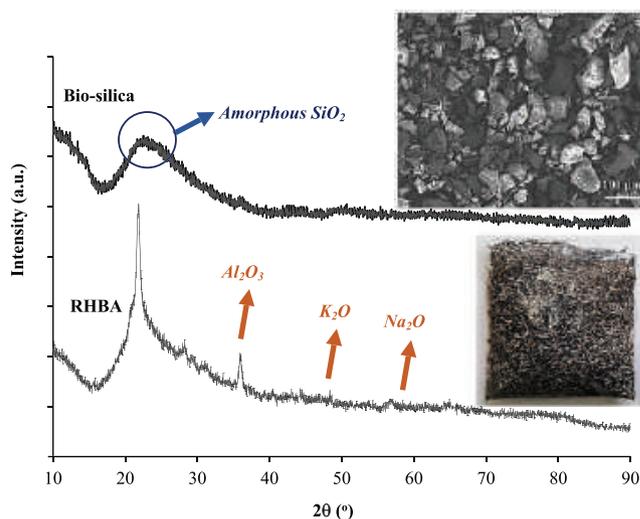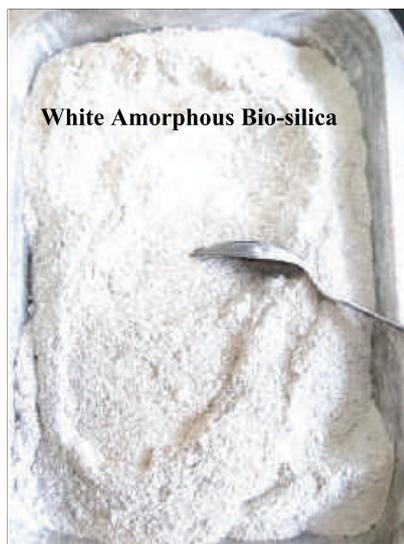

FIGURE 5 X-ray diffraction (XRD) profile for rice husk black ash (RHBA) and bio-silica

(a) (b)

FIGURE 4 Bio-silica from rice husk black ash (RHBA): (a) before refining and (b) after refining

TABLE 3 X-ray fluorescent analysis for RHBA and bio-silica under $R_{A/F}$ 6 g/g and 1-h extraction

Mineral	Composition (% w/w)		
	RHBA	Bio-silica from HCl precipitation	Bio-silica from H_2SO_4 precipitation
SiO_2	76.59	95.67	95.77
Na_2O	11.40	1.22	1.18
K_2O	2.59	1.49	1.48
SO_3	1.89	0.57	0.55
Cl	0.61	0.39	0.39
P_2O_5	3.03	0.32	0.32
Al_2O_3	1.62	0.23	0.19
CaO	1.82	0.10	0.11
Trace (hematite, zinc oxide)	0.45	0.01	0.01
Loss on ignition (LOI)	12.11	-	-

Abbreviation: RHBA, rice husk black ash.
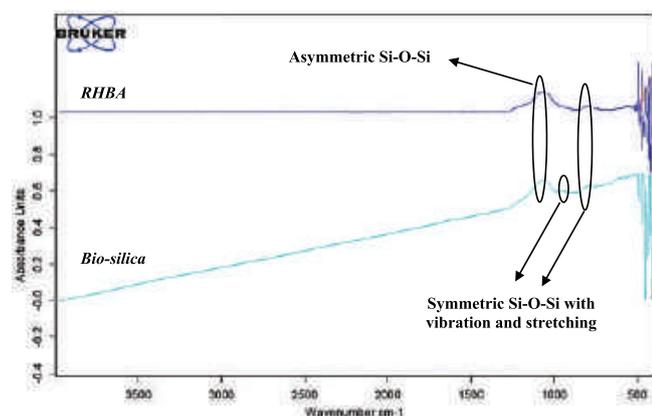
FIGURE 6 Rice husk black ash (RHBA) and bio-silica Fourier transform infrared (FTIR) spectrogram
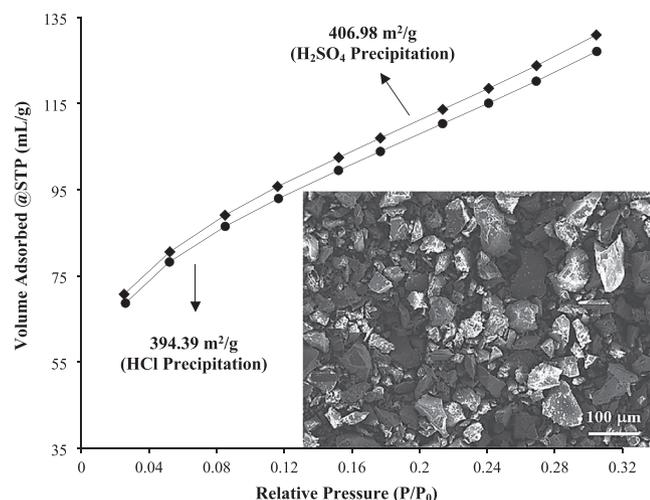

FIGURE 7 Bio-silica scanning electron microscope (SEM) and adsorption isotherm curve

In addition, the extracted bio-silica has a dominant prism shape with a slightly uniform particle size below 100 μm and has a surface area of 394.39–406.98 m^2/g as seen in Figure 7. Vacuum carbon coating is proven capable to intensify the particle conductivity, which is observed as the bright spot emersion. The brighter spot means more conducted particle, whereas the image and particle shape are difficult to be identified when in the absence of carbon coating. The bio-silica surface area from this study is quite higher than silica aerogel from RHA which is synthesized by Temel et al.,[30] but still far lower than silica aerogel preparation which is carried out by Cui et al.[31] Nevertheless, bio-silica from this study has the advantage of cheaper process production because it does not involve additional chemicals like organic solvents, does not utilize supercritical drying as in silica aerogel synthesis, and involves lesser chemicals. Likewise, the bio-silica morphology is consistent with Trysil 300 particles, a commercial silica product.[56]

This method is able to obtain bio-silica from the green feedstock with an equal product characteristic as commercial silica. Besides no excessive fossil fuel is required, the process condition is also far milder than the common commercial silica production. Hereafter, the CO_2 and NO_x emissions' suppression makes this process greener.[2,15] The high surface area of bio-silica from RHBA is suitable for adsorption interests[19] and was successfully applied in another study to adsorb up to 83.5% of 3-monochloropropanediol (3-MCPD), a carcinogenic compound in food (Restiawaty, 2020. The percentage of 3-MCPD Removal. Data not published yet). As a future outlook, bio-silica production could replace

commercial silica with the superiorities of being a cheaper process, more renewable, eco-friendly, and sustainable.

4 | CONCLUSIONS AND PROSPECT

The bench-scale amorphous bio-silica production from RHBA through pretreatment, alkaline extraction, and posttreatment has been successfully performed. Pretreatment by acid leaching is well proven to reduce the mineral impurities, resulting in the product purity exceeding 95%. The 1-h extraction under $R_{A/F}$ 6 g/g showed the highest extraction yield of 98.04% for HCl precipitation and 97.17% for H_2SO_4 precipitation. Furthermore, precipitation by HCl or H_2SO_4 provides a slight distinction for both extraction yield and product purity. To this point, the bio-silica obtained still seems black because pretreatment by acid leaching is unable to remove the carbon, while carbon removal by refining is successful in turning it white. This implies RHBA pretreatment has succeeded to remove most of the alkaline minerals content and consequently carbon content in RHBA could be completely oxidized through refining. The result of this study convinces that RHBA is able to be transformed to white amorphous bio-silica by combining the production process through the addition of posttreatment. The study also reveals that the bio-silica from RHBA had the superiorities of being a cheaper process, more renewable, eco-friendly, and sustainable compared to the commercial silica, as well as proper for 3-monochloropropanediol compound adsorption purposes.

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