

Chemeca 2018

Chemical Engineering in Australasia









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Abstract: The Chemeca 2018 conference was held in Queenstown, New Zealand. The theme for 2018 was Chemical Engineering in Australasia. The conference explored the emerging opportunities and challenges for the chemical engineering profession and process industries throughout the region, and included sustainability, process safety, education, chemical engineering fundamentals and systems, collaboration along with the 'bio' dimension.


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






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Biosilica recovery from pulped rice husk by acid precipitation

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Abstract: Rice husk is amongst the abundant agricultural wastes in rice-producing countries, such as Indonesia. About 20% mass of the whole rice is rice husk. Furthermore, silica content in rice husk is quite high of about 20%. Therefore, rice husk has a potential to be used as an alternative source of biosilica which could be further used as adsorbent or catalyst support. In this research, 2 types of acids, i.e. HCl and H₂SO₄ were compared in terms of their effectiveness during biosilica recovery from pulped rice husk. Rice husk was first pulped with 1 M NaOH at elevated temperature to dissolve silica and then filtered. Precipitation of silica was induced by the addition of acids until pH of 7 was reached. The mixture was aged for 72 hours prior to silica recovery. The results showed that HCl treatment was more effective compared to H₂SO₄ treatment. The silica obtained from was whiter in color and has a higher purity based on SEM/ EDX analysis. Furthermore, the silica was proved to be amorphous while the silica obtained from H₂SO₄ treatment showed the onset of crystalline silica. Additionally, the concentration of silica in the particle obtained from HCl treatment was ~24% based on SEM/ EDX analysis, which was almost double of that obtained from H₂SO₄ treatment. This process enabled biosilica recovery which has an added value out of low cost rice husk wastes.

Keywords: HCl, H₂SO₄, precipitation, pulping, rice husk, silica.

1 Introduction

Rice is the main staple food in Indonesia and its demand has been continually increasing every year leading to the increasing agricultural wastes such as rice husks during rice processing. Total rice production in the world in 2016 reached to about 745.5 million tons (FAO, 2016) and 79.141 million tons was produced in Indonesia (BPS, 2016). Rice husks were byproducts of paddy milling and accounts for about 12% of the total wastes which is still underutilized. Rice husks have been usually used as planting media and animal beddings. Rice husks were usually simply removed out by burning. In fact, rice husks contain many useful components such as 18% xylan, 22% lignin, 38% cellulose, 20% amorphous silica, and 2% of other organics (Zhang *et al.*, 2014). Silica has been widely used in many industries such as cement, concrete, ceramics, adsorbent, and catalyst supports (Liou and Jung-Wu, 2011). Biosilica is defined as silica accumulated in living creatures, not harmful, and has an amorphous form, thus it is quite useful for being used in food or medical applications. In contrast, silica found in the sands and rocks is crystalline and could cause silicosis when accumulated in the lungs. Biosilica has potentials to be used as emulsifiers for the oil-water systems from squalene, n-heptane, and paraffin oil (Linden, 2012). Biosilica could be used in medical applications as well as drug delivery vehicle and materials for dental composites (Aguiar *et al.*, 2017).

Lignocellulosic materials could have been used as raw materials for paper making. Lignocellulosic materials derived from both wood and non-wood materials were used to be treated with basic solution in order to dissolve lignin during the pulping process and alkaline black liquor was produced as a byproduct.

Minu *et al.* (2012) investigated rice straws pulping as alternative raw materials for paper making. Rice straws contain ashes of 15.5% which is rich in silica that could be further isolated to obtain pure amorphous silica. Minu *et al.* (2012) recovered silica out of black liquor by acid precipitation. The black liquor was produced from extraction of acid leached silica ashes using basic solution. Precipitation of silica was carried out at pH 6-7 by the addition of several acids such as H₂SO₄, HCl, H₃PO₄, and HNO₃ of 2% concentrations. Silica was precipitated in the form of silica gel after 24 hour aging process.

Majumder *et. al.* (2014) extracted silica from acid leached rice husk ashes using 1 N NaOH at 80°C for 2 hours under stirring. The solution was then titrated by HCl 2 M until pH 7 was reached. Precipitation of silica gels was allowed to settle for 24 hours. The obtained silica gels were diluted with water and centrifuged at 6000 rpm for 15 minutes. Silica powder could be obtained after drying of silica gel in the oven at 80°C for 12 hours followed by cooling in the dessicator.

Ghorbani *et. al.* (2015) investigated the recovery of silica from rice husk using alkaline treatment followed by acid precipitation. Rice husk was leached by 1 N of several acid solutions, such as H₂SO₄, HNO₃, and HCl followed by thermal treatment at 600°C in order to obtain silica ash. Silica ash was then subjected to alkaline extraction using 0.5 M NaOH solution and heated at 100°C for 4 hours with stirring during the formation of sodium silicate solution. Sulfuric acid solution of 10% was added until pH 7 to precipitate SiO₂. The mixture was then aged for 48 hours to complete the precipitation forming silica gel prior to drying in order to obtain silica powder. Simanjuntak *et. al.* (2016) used liquid smoke as a substitute for nitric acid for extraction of amorphous silica from rice husk through sol-gel route. Silica nanoparticles obtained using liquid smoke were much smaller within the range of 194-680 nm in contrast with those obtained using nitric acid of which sizes were in the range of 1142-3712 nm.

Non thermal method of silica extraction from rice husk using basic solution followed by acid precipitation required less energy compared to the thermal method which was operated at high temperatures to burn the organics. In addition, the non thermal method reduced air pollution thus considered to be environmentally friendly process (Todkar *et. al.*, 2016) and was able to produce silica with higher purity (Yuvakkumar *et. al.*, 2014). This research objective was to investigate the characteristics of biosilica directly recovered from pulped rice husk by acid precipitation using HCl and H₂SO₄ solutions. H₂SO₄ is an oxidizing acid which requires a specific storage area, though both H₂SO₄ and HCl acids are considered to be corrosive. The effectiveness of those acids was compared in terms of yields and characteristics of the recovered biosilica.

2 Materials and Methods

2.1 Materials

Rice husks were derived from a paddy milling in Mojokerto, East Java, Indonesia; HCl 37% (Merck, Germany); H₂SO₄ (Mallinckrodt, USA); NaOH flakes (Bratachem, Indonesia); demineralized water.

2.2 Silica extraction from rice husks

Silica in rice husks were extracted during the pulping process of rice husks by the addition of NaOH solution due to its solubility in basic solution. Rice husks were previously leached by 5% citric acid prior to pulping process in order to remove the inorganic impurities. Extraction process was carried out by adding 10 g rice husks in a beaker glass containing 500 ml NaOH 1 M. The mixtures were heated until boiling for 2 hours in a hotplate (Mirak, Germany) while being stirred with a magnetic stirrer at 600 rpm. The beaker was covered by *aluminium foil* during heating to prevent the solvent loss due to evaporation. Silica was extracted from rice husk and form sodium silicate with NaOH solution. The pulped rice husks were separated from the black liquor via filtration. The filtrate containing sodium silicate was cooled down to room temperature prior to further treatment.

2.3 Silica precipitation using acids

The black liquor containing sodium silicate was treated by acids for the rice husk biosilica recovery. Sodium silicate solution of about 200 ml was put in a beaker glass and slowly heated to 40°C and mixed at 400 rpm with a magnetic stirrer. The temperature was held at 40°C during the dropwise addition of acid solutions (HCl 3% or H₂SO₄ 3%) until pH 7 measured by pH-Meter CG 85 (Schott, Germany) was reached. The temperature was continuously held at 40°C while mixing for 90 minutes before cooling down to room temperature. The biosilica would start to gel. The aging time was set at 72 hours to allow the complete precipitation of biosilica.

2.4 Recovery of rice husk biosilica

The precipitated silica gel was filtered using a buchner filter (Schott, Germany). Silica gel with brownish color was rinsed using a demineralised water for several times to get rid of the impurities such as salts containing sulphates (Ghorbani *et. al.*, 2015). The silica gel color was getting transparent after rinsing. Afterwards, the silica gel was dried in an oven (Memmert, Germany) at 150°C for 24 hours to obtain biosilica. The dried biosilica was milled and subjected to thermal treatment at 750°C for 5 hours to remove the organic impurities of mainly lignin in the furnace (Ney VULCAN D-550, Denstply Ceramco, USA). Biosilica powder was whiter in color after the thermal treatment. The biosilica powder was cooled down to room temperature prior to further characterisation. The mass of recovered biosilica powder was weighed in order to estimate the biosilica yield according to Equation 1.

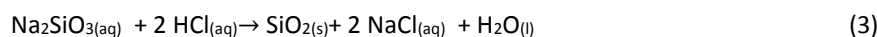
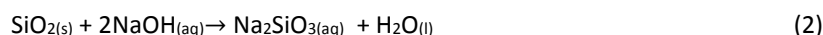
$$\% \text{ biosilica yield} = \frac{\text{mass of final silica powder}}{\text{mass of initial rice husks}} \times 100\% \quad (1)$$

2.5 Characterisation of biosilica

The recovered biosilica powders were characterised using several techniques such as FTIR (Fourier Transform Infrared Spectroscopy), XRD (X-ray Diffraction), and SEM/ EDX (*Scanning Electron Microscopy/ Energy Dispersive X-Ray Spectroscopy*). The functional groups of chemical constituents of the ashes were analysed using FTIR (Shimadzu, Japan) at Chemistry Department, Institut Teknologi Sepuluh November, Surabaya. Samples were prepared using a KBr pellet at dry atmosphere at 25°C. Each spectrum was derived from an average of 32 scans with a resolution of 64 cm⁻¹ within the wavenumber range of 400–4000 cm⁻¹. XRD (X'Pert Pro PACAnalytical, Netherlands) measurement was conducted within 2θ = 5–70° at Energy Laboratory, Institut Teknologi Sepuluh November, Surabaya in order to study the crystallinity of the biosilica powder. Finally, the morphology of biosilica powder and the semi-quantitative Si content in the particles were determined using SEM/ EDX (Quantachrome, USA) at Energy Laboratory, Institut Teknologi Sepuluh November, Surabaya.

3 Results and discussion

Alkaline extraction of biosilica during the pulping process of rice husk took place according to reaction equation (2) whereby silica reacted with sodium hydroxide forming sodium metasilicate (Todkar et. al., 2016). Biosilica precipitation was then induced upon the addition of acid solution according to reaction equation (3). Silica started to gel when pH < 8. However, lignin started to precipitate when pH < 5, thus optimum pH for silica precipitation at pH 7 was maintained (Minu et. al., 2012)



In this experiment, the black liquor derived from pulped rice husk was treated by 3% acids (HCl and H₂SO₄) until pH 7 was reached to induced silica gel precipitation. The biosilica recovered using HCl has a whiter color compared to that obtained using H₂SO₄ based on the observation during the experiments (Fig. 1). Furthermore, the biosilica yields obtained using HCl and H₂SO₄ during the precipitation process were 6.821% and 7.206%, respectively. The yield of biosilica derived from H₂SO₄ treatment was slightly higher due to the incorporation of carbon impurities in the silica powder indicated by their grayish color.

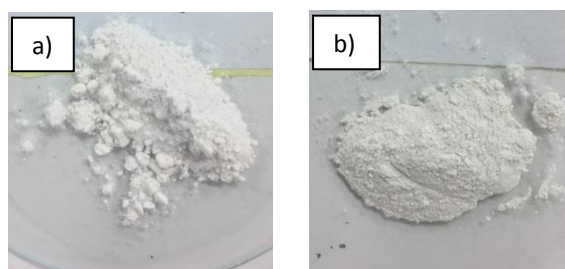


Figure 1: Physical appearance of recovered biosilica from sodium silicate solutions treated by different acids. a) HCl; b) H₂SO₄

The FTIR spectra of both biosilica precipitated by HCl and H₂SO₄ were depicted in Fig. 2. All spectra represented fingerprints of pure amorphous silica indicated by several peaks found at ~ 1100, 800, and 460 cm⁻¹. The biosilica spectrum derived from HCl precipitation seemed to be very similar with that obtained from H₂SO₄ precipitation. The trace of organic impurities presumably found in the biosilica obtained from H₂SO₄ precipitation was hardly seen and could be evaluated using another method.

The XRD patterns of both biosilica recovered using HCl and H₂SO₄ were depicted in Fig. 3. In general, both showed the patterns of amorphous silica which was the typical form of silica found in biological creatures. However, those recovered using H₂SO₄ showed the onset of crystalline silica shown by a conspicuous small sharp peaks at 2θ ~22° attributed to cristobalite. Most probably, some inorganic impurities were also precipitated upon the

addition of H₂SO₄ producing some salts such as K₂SO₄ and Na₂SO₄ which could act as catalysts accelerating the amorphous phase into biosilica during the furnace.

crystalline phase of combustion in the

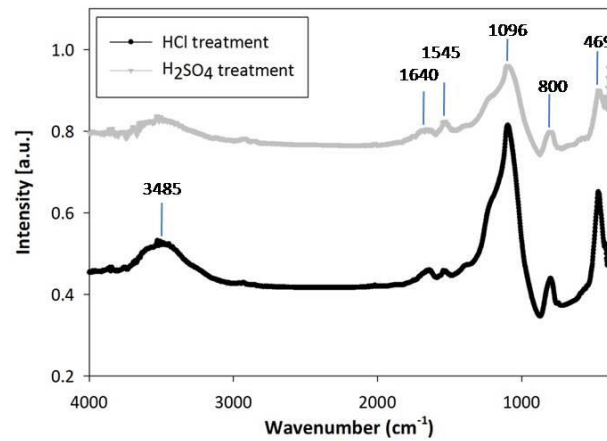


Figure 2: FTIR spectra of

from sodium silicate solutions treated by different acids

recovered biosilica

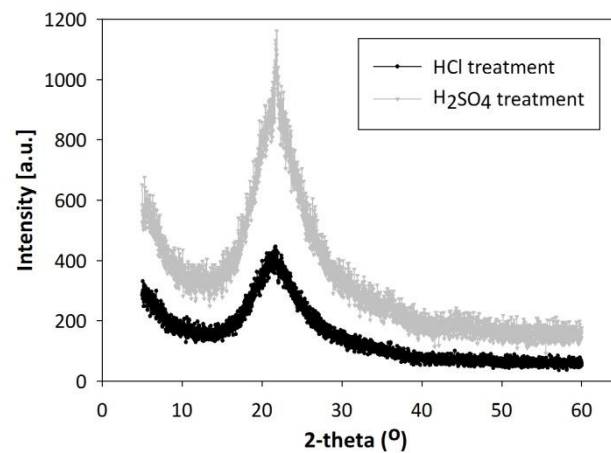


Figure 3: XRD patterns of recovered biosilica from sodium silicate solutions treated by different acids

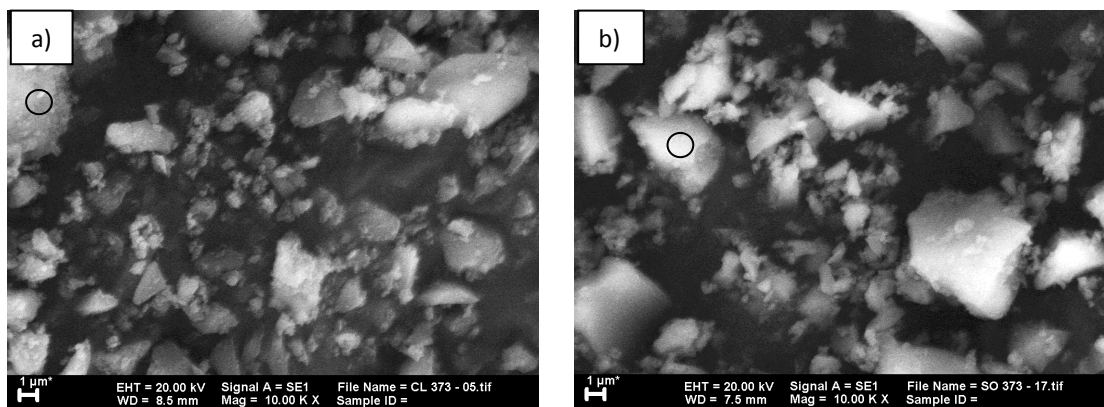


Figure 4: SEM of recovered biosilica from sodium silicate solutions treated by different acids. a) HCl; b) H₂SO₄. The black cycles indicated the location where EDX measurements were conducted.

Table 1: EDX results of biosilica particle derived from sodium silicate solutions treated by different acids

Acid treatment	Si	O	Na	K	C	Al
HCl	23.71	75.41	0.39	0.23	-	0.26
H ₂ SO ₄	11.73	53.99	0.37	-	33.82	0.09

The SEM/ EDX images of biosilica recovered by HCl and H₂SO₄ could be seen in Fig. 4. The morphology of biosilica particles appeared to be irregular with the highest Si concentration of ~24% was locally found in the particle obtained from HCl precipitation as depicted in Table 1. The Si content in silica particle obtained from HCl precipitation was almost doubled than that obtained from H₂SO₄ precipitation. The carbon content in biosilica particle obtained from H₂SO₄ treatment was quite high of ~34% which was not found in the biosilica obtained from HCl treatment. These results implied that the amorphous biosilica derived from HCl treatment was higher in purity and thus had better overall preferred characteristics.

4 Conclusions and recommendations

Amorphous biosilica particles were recovered from pulped rice husk by precipitation process induced by acids. The black liquor containing sodium silicate solutions and soluble organics was further treated by 3% HCl and 3% H₂SO₄ until pH 7 where silica gels started to form. The aging time of 72 hours was set to allow complete precipitation of silica gels. Silica gels were filtered followed by thermally treatment to remove the accompanied organic impurities in order to obtain biosilica powder. It turned out that the biosilica derived from HCl treatment was whiter in color and demonstrated an amorphous silica fingerprint based on FTIR analysis confirmed by XRD analysis. The onset of crystalline silica namely cristobalite supposed to be present on H₂SO₄ treated derived biosilica due to a small peak found at $2\theta \sim 22^\circ$. Biosilica produced from HCl treatment showed high purity corroborated by a quite high local Si concentration of ~24% based on SEM/ EDX analysis. The biosilica morphology appeared irregular and agglomerated. Process modifications were required in order to produce nanoparticles silica with higher yield and purity supported with additional characterisation techniques.

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