X-Ray Diffractometer (XRD) and Scanning Electron Microscopy (SEM) of Silica Extracted from Banana Stems via Acid Leaching Treatment

N. J. Yusaidi*, S. A. Abdullah and N. A. Zarib
Faculty of Mechanical Engineering, Universiti Teknologi MARA (UiTM), 40450 Shah Alam, Selangor, Malaysia

* jannahyusaidi38@gmail.com

Abstract. Silica is known as the natural resources where it has the second most abundant mineral in Earth’s crust. In order to help the expanding research more on agricultural waste for silica extraction, banana stems has been used for this experiment studies. The main aim of the study is to know the percentage of silica in banana stems via leaching process. Hydrochloric acid (HCl) and citric acid (C6H8O7) was used as a leaching reagent for removing the inorganic impurities from the banana stems and the effect of leaching time, reagent concentration and the effect of acid on the banana stems are investigated. Both acids results used same concentration of 0.1, 0.5 and 1.0 mol while leaching time were 30, 60 and 90 minute respectively. The results of hydrochloric acid (HCl) and citric acid (C6H8O7) leaching treatment test using the Energy Disperse X-Ray Spectroscopy (EDX) spots shows silica can be produced at 85.5% and 99%, respectively. Lastly, the silica obtained from the experiment was crystalline silica using the analysis of X-Ray Diffraction (XRD) and the morphological surface of the banana stems were obtained using the Scanning Electron Microscopy (SEM).

1. Background of study
Silica is defines as a solid, unreactive, colourless compound which occurs as the mineral quartz same as the principal constituent of sandstone and other rocks. Amorphous silica has many uses in its application such as additive in painting and coatings, concrete and filter media [1], [2]. Silica can be found in agricultural waste such as rice husks and palm ash where both of these source has the highest silica production which is more than 90% [3], [4]. In 2018, Malaysia has planted bananas more than 110,000 metric tonnes where 47% of banana production were original from Johor followed by Pahang and Sabah [5]. This can be seen as one of the opportunities to use banana stems as the agricultural waste for silica demand. The large number of farmers disposed the banana stems using an open burning method where it can cause major environmental issue [6], [7]. For silica extraction, leaching treatment is the best method to do since it is less expensive [8]. Previous researcher has used hydrochloric acid (HCl), sulphuric acid (H2SO4), nitric acid (HNO3) for strong acid and citric acid (C6H8O7C6H8O7) for weak acid [8], [9]. According to [10], they discovered silica content in banana stem ash with the percentage of 16.5%. Therefore the focus of this project is to determine the potential of banana stems as a source of silica.

2. Methodology
In this study, the materials used was banana stems collected from Shah Alam, Selangor. Hydrochloric acid (analytical reagent, fuming 37) and citric acid (Anhydrous, chemically pure) where both product of
R&M Chemicals with the total volume of 2.5litre and 500grams respectively. Distilled water are used for leaching and filtration process.

Banana stems were cut into small pieces and washed using the tap water to remove dirt and impurities. The sample was placed in the Drying Oven TYPE T3D-YT at 40°C for 24 hours to remove excessive moisture. Then the sample grind using Planetary Mono Mill PULVERISETTE 6 classic line for 300rpm and sieve using Vibratory Sieve Shaker Analysette 3 Spartan for 250μm (60 mesh) to become fine powder. There are 3 different acid concentration that was prepared for the leaching process. 15g of banana stems was mixed together into total volume of 500ml of hydrochloric acid and distilled water into the beaker. For 0.1mol of hydrochloric acid, the sample were stirred for 30minutes, 60minutes and 90minutes. The beakers was placed onto the hot plate magnetic stirrer (Protechno DS-30) using the magnetic stirrer bar. After the leaching was completed, the sample was rinsed using distilled water at room temperature to remove the hydrochloric acid content from the banana stems. The steps are then repeated using 0.1mol, 0.5mol, 1.0 mol for citric acid and hydrochloric acid for 0.5mol and 1.0mol. After the leaching process, the materials were dried at 85°C for 1 hour.

After the sample has been prepared, it was placed in the Scanning Electron Microscopy (SEM) with the model of Type Leo DSM 982 Gemini which was supplied from Hi-Tech Instruments (Malaysia). It is for examine the morphological surface at 15kV and the electron beam was directed at the sample with a range diameter of 5-10 nM. Before the sample were placed in the machine, it must be coating using the coating spatter where the material used for coating is platinum. The magnification range used for the banana stem samples was 20x to 1000x.

Lastly, the X-Ray Diffractometer (XRD) machines model used are Rigaku Ultima Model IV where the supplier are from Rigaku Corporation and its Global Subsidiaries. For sample preparation, the sample are placed onto the specific holder specimen (2cm x 2cm) by compacting it before placing it in the diffractometer. The initial angle was taken at 10° and end at 70° with reading rate of 0.5°/min.

3. Results and Discussion

Figure 1a) shows the SEM images of untreated banana stems powder at magnification of 1000x. It shows that the particles has smooth surface and irregular shape. This is because the sample was not treated with any acid leaching. Energy Dispersive X-ray spectroscopy (EDX) test are used to determine the measureable chemical analysis at certain spots on the sample image. The EDX results shows the SiO₂ has percentage of 85.8%. The SEM images of Figure 1b) shows there is presence of rough surface condition. This is because of the citric acid plays role on removing the metallic impurities during leaching treatment. The wt% of treated banana stems (1.0M of citric acid for 30 minute) using spot EDX is 99.9% which has increased from the untreated banana stems. Lastly, Figure 1c) shows the SEM images of treated banana stems (0.5M of hydrochloric acid for 90 minute). It can be seen the particle has rougher surface due to the acid leaching. The weight percentage of silica for 0.5M banana stems via HCL was 85.5% which are slightly lower than the untreated banana samples and 1.0M of citric acid. During the EDX test, there are might have higher silica content presence on other spot area. Noratiqiah et al. reported in their study that they have obtained smooth surface and rough surface on the rice husk particle [11].

![Figure 1](image-url)
Figure 2a) shows graph Intensity (a.u) versus Diffraction, $2\theta$/° for non-leaching sample. The banana stems sample was not added with any acids in order to know the element existed in the sample. The graph indicates the highest peak of silica was 22.04°, 35.135°, 37.81°, 43.33°, 52.49° and 57.44° of two theta. Figure 2b) shows the sample was added with 1.0 mol of citric acid for 30minutes of stirring time. The graph shows the silica are in crystalline form. It can be seen from the graph that the highest peak was range between 14.89° to 77.03° of two theta (degree). Lastly, the XRD pattern on figure 2c) shows the sample has undergo the leaching process. The sample was added with 0.5mol of hydrochloric acid for 90minutes of stirring time. Based on the graph below, the highest peak of silica in HCL was 22.34°, 25.56°, 35.14°, 43.29°, 52.61°, 57.45°, 66.44°, and 68.2° of two theta. It can be concluded that the silica existed in banana stems was crystalline silica. Y. Shinohara and N. Kohyama mentioned in their finding where they have also found the silica existed in crystalline form on the rice husk ash.

![Figure 2](attachment:image.png)

**Figure 2:** XRD Pattern of (a) untreated banana stems (b) treated banana stems of 1.0M citric acid (30 minute) (c) treated banana stems of 0.5M hydrochloric acid (90 minute)
4. Conclusion
In this study, the process for silica extraction has been conducted using acid concentration and leaching time for the parameters. Based on the SEM micrograph, as the acid concentration has increased, the surface of the sample material will become flakier and has a rougher surface. Meanwhile for XRD graph, all of the sample shows the silicon dioxide was presented in the crystalline form and the higher peaks indicates it was silicon dioxide. The findings also shows the highest silica contained was in 0.5mol hydrochloric acid with 90 minute leaching time and 1.0mol citric acid with 30 minute leaching time which is 85.5% and 99%, respectively. The results of the current investigation is important to know the silica percentage in the natural fibre due to the need of expanding research in agricultural waste. Therefore, by expanding the research in agricultural waste via leaching treatment is important to reduce the unwanted waste to reach sustainable growth.

Acknowledgement
The authors would like to thank Universiti Teknologi MARA (UiTM) for financial support [Project Grant No: 600-IRMI/PERDANA 5/3 BESTARI (051/2018)].

Reference
[1] L. Hao et al., “Controllable fabrication and characterization of biocompatible core-shell particles and hollow capsules as drug carrier,” Appl. Surf. Sci., vol. 252, no. 24, pp. 8724–8733, 2006.
[2] B. Mistry, “Properties and Industrial Applications of Rice husk,” Int. J. Emerg. Technol. Adv. Eng., vol. 6, no. 10, pp. 2677–2679, 2016.
[3] K. G. Patel, R. R. Shettigar, and N. M. Misra, “Recent Advance in Silica Production Technologies from Agricultural Waste Stream – Review,” J. Adv. Agric. Technol., vol. 4, no. 3, pp. 274 279, 2017.
[4] C. P. Faizul, C. Abdullah, and B. Fazlul, “Extraction of silica from palm ash via citric acid leaching treatment,” Adv. Environ. Biol., vol. 7, no. 12, pp. 3690–3695, 2013.
[5] M. of Agriculture, “Statistik Tanaman (Sub-Sektor Tanaman Makanan),” in Buku Statistik Tanaman (Sub-Sektor Tanaman Makanan), pp. 44–58.
[6] A. Mohiuddin, M. K. Saha, M. S. Hossian, and A. Ferdoushi, “Usefulness of Banana (Musa paradisiaca) Wastes in Manufacturing of Bio-products: A Review,” Agric., vol. 12, no. 1, pp. 148–158, Aug. 2014.
[7] N. Abdullah, F. Sulaiman, M. Azman Miskam, and R. Mohd Taib, “Characterization of Banana (Musa spp.) Pseudo-Stem and Fruit-Bunch-Stem as a Potential Renewable Energy Resource,” Int. J. Energy Power Eng., vol. 8, no. 8, pp. 815–819, 2014.
[8] N. Permatasari, T. N. Sucaya, and A. B. D. Nandiyanto, “Indonesian Journal of Science & Technology Review : Agricultural Wastes as a Source of Silica Material,” Indones. J. Sci. Technol., vol. 1, no. 1, pp. 82–106, 2016.
[9] K. V. Selvakumar, A. Umesh, P. Ezhilkumar, S. Gayatri, P. Vinith, and V. Vignesh, “Extraction of silica from burnt paddy husk,” Int. J. ChemTech Res., vol. 6, no. 9, pp. 4455–4459, 2014.
[10] M. Marlinda, R. Ramli, and M. Irwan, “A Comparative study Of Catalytic Activity Of Heterogeneous Base Of Banana Stem Ash And Fly Ash On Production Of Biodiesel Byultrasonic Silica,” Int. J. Sci. Technol. Res., vol. 4, no. 08, pp. 169–172, 2015.
[11] N. Syahirah and S. Abdullah, “Effect of C 6 H 8 O7 Concentration on Silica Extraction of Rice Husk , Rice Husk Ash and Mixture of Rice Husk With Rice Husk Ash Via Acid Leaching Process,” Int. J. Eng. Technol., vol. 7, no. 4.18, pp. 190–195, 2018.