# Structural Analysis of Silica Extract from Banana Stems via Acid Leaching under Different Reaction Time

**Nurul Jannah Yusaidi, Shahrul Azam Abdullah, Noratiqah Syahirah Mohd Zarib**

***Abstract*: Production of silica from agricultural waste such as banana stems are mainly synthesized from acid leaching treatment. Citric acid ($C_6H_8O_7$) was used as substitute for conventional hydrochloric acid (HCL) and sulphuric acid ($H_2SO_4$) because it is safer and less harmful to the human health. The main parameters in leaching treatment are leaching time and acid concentration. The acid concentration used was 1.0 mol/L of citric acid ($C_6H_8O_7$) and leaching time of 30, 60 and 90 minutes, respectively. The leaching treatment produced 47.8% to 99.9% of extracted silica. The samples of extracted silica were characterized using X-Ray Diffraction (XRD) and Scanning Electron Microscopy (SEM).***

***Keywords: Banana stems, citric acid, scanning electron microscopy (SEM), X-ray Diffraction (XRD)***

## I. INTRODUCTION

Silica was discovered in the early 1800s from Berzelius where it is commonly found in quartz type [1]. Back then, the people used the silica sand to increase the concrete strength. When the silica interacts with oxygen, it will form into silicon dioxide ($SiO_2$) where it falls under group IV in the periodic table. Silica has many usages in the industrial application such as in paintings and coatings to improve the appearance and durability of the architectural and industrial paint and coatings. For Malaysia's country, the miner used mining method to extract silica from the Earth's crust where this method has many side effects to the workers especially health and time factor [2]. Furthermore, this conventional method also requires extraordinary high pressure and temperature to obtain silica. Therefore, the researcher have done research on the natural fiber to determine the silica content in it.

In Malaysia, banana fruit is the second most planted fruit with a total production of 530, 000 metric tonne per year where most of the bananas were planted in Johor and followed by Pahang and Sabah [3], [4]. Banana stems are considered as waste since the fruit was harvested, the banana tree will be cut down to let new banana tree grows from it [5]. Most of the farmers often faced difficulties of disposing the banana pseudo stems therefore, these wastes are getting accumulated in the banana plantation area which can cause hazards and imbalance ecosystem [6].

Research shows that silica can be found in agriculture waste such as the rice husk ash and bamboo leaves ash respectively [7], [8]. The methods used to extract silica were via the acid leaching treatment such as the citric acid ($C_6H_8O_7$), hydrochloric acid (HCL) and sulphuric acid ($H_2SO_4$). Citric acid is categorized under the weak acid meanwhile hydrochloric acid and sulphuric acid were strong acid. Citric acid ($C_6H_8O_7$) was proven to be much effective in extracting the silica from the rice husks compared using the sulphuric acid ($H_2SO_4$) as the leaching [9].

Previous researchers such as K. G. Patel et al. mentioned there are two ways of extracting silica by using thermal and chemical methods. The result showed using chemical method is more efficient and has extremely good quality [7]. Ghorbani et al., have successfully extracted 95.55% of high purity silica from the rice husk ash using 1 N of hydrochloric acid (HCI) followed by heat treatment of 600 °C [10]. W. Xu et al. has proven using 1 N of hydrochloric acid (HCL) followed by controlled calcination at 600 °C for 2 hours has achieved up to 96% of amorphous silica for rice husk [11]. J. J. F. Sacceda and R. L. de Leon has successfully achieved nearly 100% amorphous silica in rice husk and 97.56% of crystalline silica in rice husk ash using acid and heat treatment. The chemicals they used in the experiment were hydrochloric acid (HCL), sodium aluminate ($Na_2SiO_2$), sodium silicate ($Na_2SiO_3$) and a seed gel [12]. Lastly, according to Marlinda et al., they have obtained 16.5% of silica from the banana stems ash using methanol ($CH_3OH$) and potassium hydroxide (KOH) [13]. Therefore, the focus of this project is to determine the potential of banana stems as a source of silica via citric acid treatment.

## II. METHODOLOGY

### A. Materials

In this study, banana stems collected from Selangor were used. Citric acid from R&M Chemicals for 500 grams (Fig. 1) was used during leaching process and distilled water are used in leaching and filtration process.

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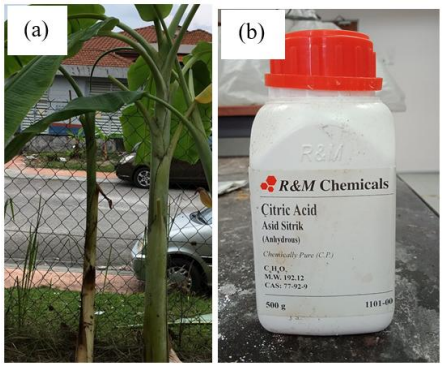


**Fig. 1: (a) Banana stems (b) citric acid ($C_6H_8O_7$)**

## B. Sample preparation

Firstly, the banana stems were cut into smaller pieces (Fig. 2a) and washed using tap water to remove dirt and impurities. The sample were then placed into the drying oven Type T3D-YT at temperature of 40°C for 24 hours to remove excessive moisture via Fig 2(b). After the sample has completely dried, the banana stems sample were grind using Planetary Mono Mill Pulverisette 6 classic line for 300rpm and sieve using Vibratory Sieve Shaker Analysette 3 Spartan for 250$\mu$m (60 mesh) to become fine powder via Fig 3(a) and Fig. 3(b). 15g of banana stems were mixed together into total volume of 500ml of citric acid and distilled water into the beaker. For 1.0 mol/L of citric acid, the sample were stirred for 30, 60 and 90 minutes, respectively.

The beaker was placed onto the hot plate magnetic stirrer (Protechno DS-30) using the magnetic stirrer bar for stirring process (Fig. 4). After the leaching was completed, the sample was rinsed using distilled water at room temperature to remove the citric acid content from the banana stems. The filtration process was completed after the water had turned to colorless via Fig. 5(b). After the leaching process, the materials were dried at 85°C for 1 hour.

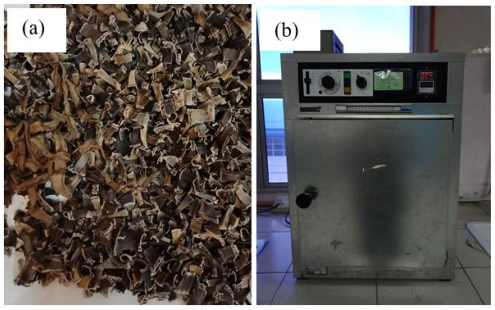


**Fig. 2: (a) Banana stems were cut into small pieces (b) Drying Oven**


**Fig. 3: (a) Planetary Mono Mill PULVERISETTE 6 classic line (b) Vibratory Sieve Shaker Analysette 3 Spartan**

**Fig. 4: Leaching process**

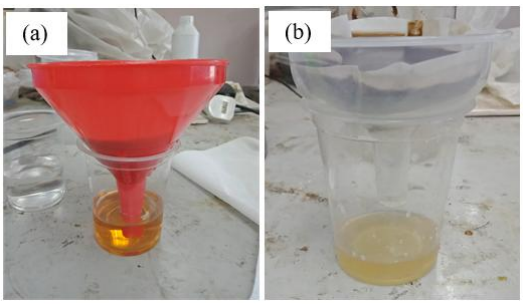


**Fig. 5: Sample undergo filtration process (a) before distilled water were added (b) after distilled water were added**

## C. Characterization

The banana stems sample was placed in the Scanning Electron Microscopy (SEM) machine with the model Type Leo DSM 982 Gemini shown in Fig. 6. This purpose of the machine is for examining the morphological surface at 15kV and the electron beam was directed at the sample with a range diameter of 5-10 NM. The samples used magnification range of 20x t0 1000x. Lastly, the X-Ray Diffractometer (XRD) machines model used is Rigaku Ultima Model IV as shown in Fig. 7 where the supplier is from Rigaku Corporation and its Global Subsidiaries. The sample is placed onto the specific holder specimen (2cm x 2cm) by compacting it for sample preparation process. After it was compacted, the sample was placed into the diffractometer. The initial angle was taken at 10° and end at 70° with reading rate of 0.5°/min.

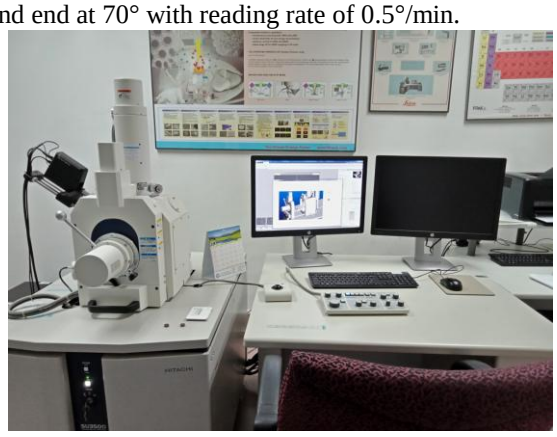

**Fig. 6: Scanning electron microscopy (SEM)**

**Fig. 7: X-Ray Diffractometer (XRD)**

## III. RESULTS AND DISCUSSION

### A. Scanning electron microscopy (SEM)

After the banana stems were leached using citric acid for 30 minutes for 1.0 mol/L, it can be seen from the SEM image for Fig. 8 that the particle has rough surface and irregular shape. This is because of the chemical reaction react with the banana stem sample. Based on the EDX result, the element existed in the sample is gallium (Ga), oxygen ($O_2$) and silica (Si). It was detected that $SiO_2$ contained of 99.9% which is the most concentrated in weight percentage. Other researchers also reported they obtained irregular and crushed shape after the palm ash was treated with citric acid which has come to the same agreement with the banana stems [14]. I.J. Fernandes *et al.* also has obtained 99.6 % of silica purity rice husk ash which has achieved the same consent with banana stems sample [15].

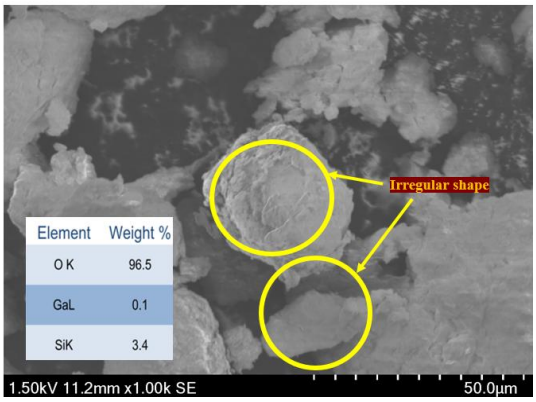


**Fig. 8: SEM image of treated banana stems via citric acid of 1.0 mol/L for 30 minute for 1000x magnification**

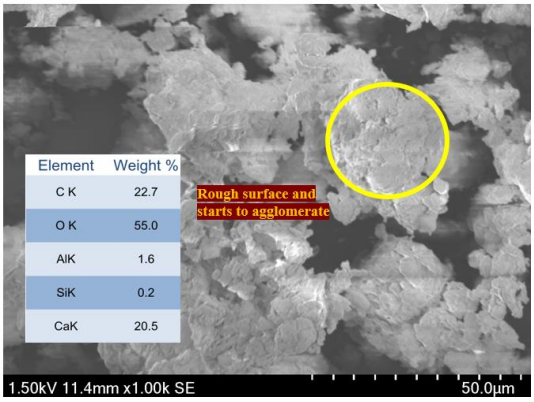


**Fig. 9: SEM image of treated banana stems via citric acid of 1.0 mol/L for 60 minute for 1000x magnification**

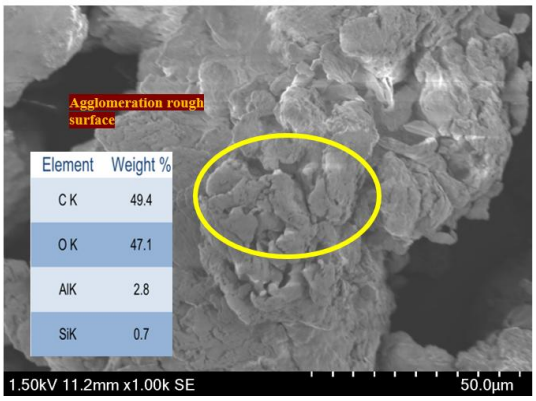


**Fig. 10: SEM image of treated banana stems via citric acid of 1.0 mol/L for 90 minute for 1000x magnification**

The SEM images based on Fig. 9 above shows the treated banana stems for stirring time of 60 minute for 1.0 mol/L. In the SEM image, the particle indicates that it has rougher surface and the surface starts to agglomerates due to reaction of citric acid for leaching time of 60 minute. The EDX results showed the element existed in the sample were carbon (C), oxygen ($O_2$), aluminum (AI), calcium (Ca) and silica (Si). It showed that $SiO_2$ has higher concentrated in weight percentage about 55.2 wt % where the other element has weight percentage of 44.8 wt %.

Lastly, the banana stems were mixed together with 1.0 mol/L of citric acid for 90 minutes of leaching time (Fig. 10). The magnification used for the SEM image was 1000x. Referring to Fig. 10, the SEM image showed the particle has high agglomeration of the rough surface due to the effect of citric acid. According to EDX result, the existed elements contain of carbon (C), oxygen ($O_2$), aluminum (Al) and silica (Si). The $SiO_2$ has lower concentration in weight percentage about 47.8 % compared with other impurities. Based on Fig. 9 and Fig. 10, element carbon (C) and calcium (Ca) has higher wt % due to factor of leaching time. The purpose of using 1.0 mol/L compared to other value of acid concentration was to observe the capability of citric acid to extract silica from banana stems sample via leaching time. It can be seen that the longer the leaching time, the lower the weight percentage of $SiO_2$. It is preferable to select the sample of 30 minute leaching time because it has the highest $SiO_2$ percentage of 99.9 wt % due to short reaction time.

### B. X-Ray Diffractometer (XRD)

The XRD pattern of banana stems of 1.0 mol/L citric acid at 30 minute was shown in Fig. 11 (a). The major peaks of $SiO_2$ occur at Bragg's 2θ angles of 14.89°, 21.8°, 25.55°, 35.12°, 37.74°, 43.27°, 52.55°, 57.49°, 66.40°, 68.11° and 77.03° for 30 minute leaching time. The graph indicates the x-axis as the range of two-theta (degree) and y-axis as intensities of the peak. The major reflection of $SiO_2$ occurs at Bragg's 2θ angles for Fig. 11(b) were 14.91°, 21.9°, 25.56°, 30.04°, 35.095°, 37.74°, 43.317°, 52.56°, 57.48°, 66.49°, 68.21°. The major peaks of $SiO_2$ occur at Bragg's 2θ angles for Fig. 11(c) were at 22.31°, 25.60°, 35.19°, 37.79°, 43.38°, 52.64°, 57.58°, 66.57°, 68.17.

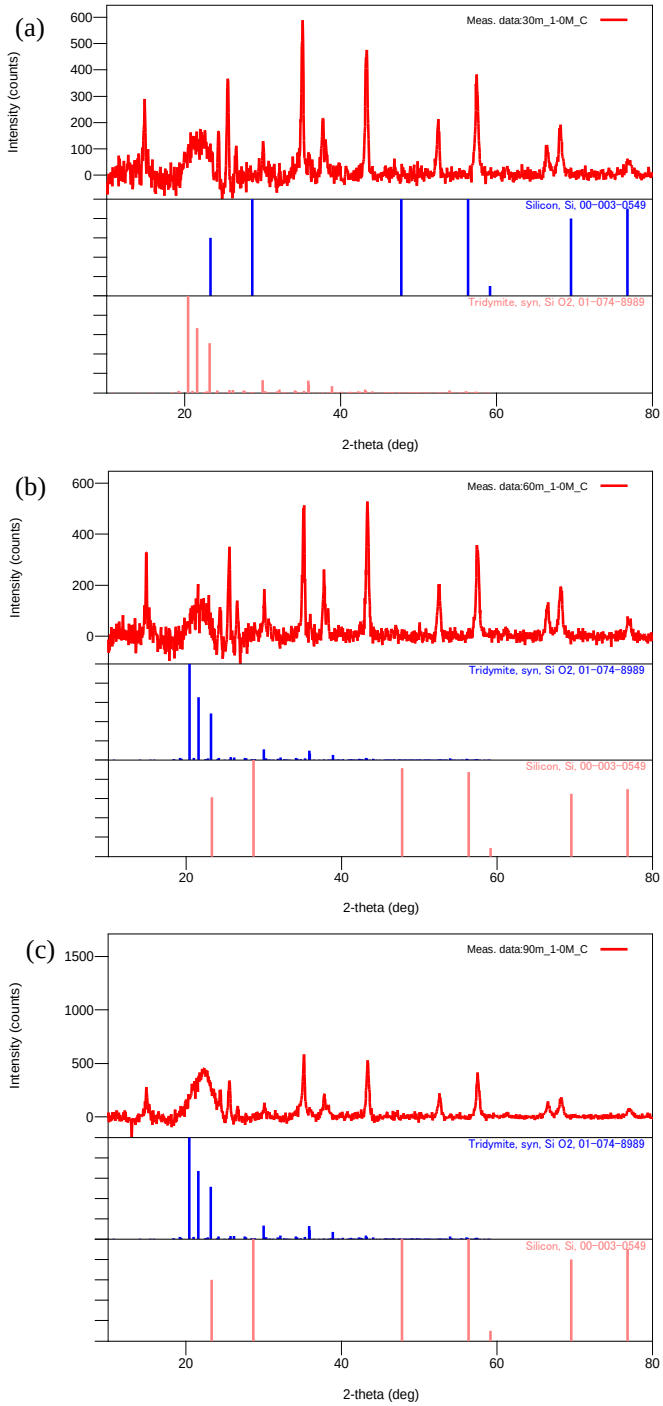


**Fig. 11: XRD pattern of 1.0 mol/L citric acid for (a) 30 minute (b) 60 minute (c) 90 minute**

Based on the XRD pattern, the silica was also presented in the tridymite phase. Tridymite are occurs when the heating temperature is at 867°C. However, the crystallization of tridymite are also observed in the room temperature. Y. Shinohara and N. Kohyama mentioned in order to prevent the crystallization of tridymite, a sufficient amount of alkali ion is required to act as a flux or mineralizers [16]. The graphs have shown that the silica is presented in the crystalline structure. Other researcher has also reported the silica they obtained were in the crystalline form where it has come to the same agreement for rice husk ash [12].

## IV. CONCLUSION

This work studied the effect of acid concentration and leaching time for the silica extraction process via leaching treatment. The result shows that the highest percentage of silica obtained using 1.0 mol/L citric acid was 99% at 30 minutes leaching time.

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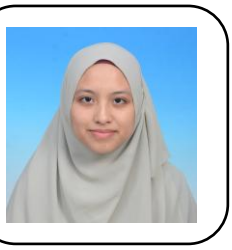
## AUTHORS PROFILE



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