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Preparation of Silica from Agricultural Leaves waste by Organic Acid Leaching Treatment

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Abstract: In this paper, two types of raw material, Toddy leaves and sugarcane leaves were used. Leaves are an inexpensive raw material that can be used to extract silica from agricultural wastes, due to silica as a useful raw material for industrial application. In this study, the environmentally benign and economically effective process to produce SiO_2 materials from toddy leaves and sugarcane leaves have been established by using citric acid leaching, not the conventional strong acids. The silica content of the product samples was characterized using X-Ray fluorescence (EDXRF), X-Ray powder diffraction (XRD), scanning electron microscopy (SEM), and Fourier transform infrared (FTIR) analysis. The highest silica content (83.9%) was produced by Toddy leaves.

Keywords: Toddy leaves, sugarcane leaves, acid leaching, calcination, Silica

1. INTRODUCTION

In Myanmar, they are largely grown in middle region of the country. Myanmar is an industrial based on agricultural country. The agricultural wastes have a huge content of silicate materials and the cheapest source for silica production. The reasons which are responsible for agricultural waste to be considered as good silica source and have potential for the large scale production are

(a) Low cost of the raw material
(b)Height silica content in agricultural waste
(c) Comparable silica quality [1], [2].

Silica (SiO2) is one of the valuable inorganic multipurpose chemical compounds .It can exit in gel, crystalline, amorphous forms and variety of other forms. Silica has played a continuous part in development of technology and been one of the basic raw materials supporting the industrial revolution. [3], [4].

Silica in elemental form is used as a component for building material. Silica in amorphous form used as absorbent, catalyst, refining agent etc. Silica in crystalline form is widely used in glass and ceramic industry. [5]

The objective of this study is to produce silica from Toddy palm leaves and Sugarcane leaves and to study the silica content from different raw types using thermo chemical leaching method. Citric acid is an organic acid and more environmentally friendly, economic and ecological compared to inorganic acids.

2. MATERIALS AND METHOD

A. Materials

Toddy palm leaves were collected from the Thanlyin Township, Yangon Division and sugarcane leaves were collected from the market, Yangon Division. The raw material was cut, cleaned, and dried before undergoing the treatment process. Citric Acid ($C_6H_8O_7$) was used for acid leaching treatment and was bought from market. Pure water was also used for acid washing.

B. Leaching Methods

Leaching generally refers to the removal of the substance from a solid via a liquid extraction media. The desired component diffuses into the solvent from its natural solid form. There are three important parameters in leaching which are temperature, contact time per unit area and solvent selection. The temperature can be adjusted to optimized solubility and mass transfer. Leaching can be divided into two categories which are percolation and dispered solid. For percolation , the solvent is contacted with solid in a continuous or batch method and widely used for extreme amount of solids. In dispersed solids, the solid are usually crushed into small pieces before being contacted with solvent. In simple words, percolation is for liquid added into solid while dispersed solid is for solid added into liquid [6], [7], [8].

C. Preparation of silica from leaves (Toddy & Sugarcane)

Leaves and citric acid were mixed by 2:2(m/m) in beaker. These mixture was heated at 80℃ and stirred by magnetic stirrer for 2hrs.After acid leaching, these leaves were washed with pure water to removes the leaching acid. And then, they were dried in oven about (50-60℃) for 24hrs.

After drying, these were subjected to sequential burning in a carburizing furnace. The burning program consisted of different temperatures and holding times, as follows: 30minute at 310℃, 60minutes at 450℃, 210minutes at 510℃ and, 30minutes at 600℃.After the calcination, the treated ash became white ash to get product silica [9].

3. RESULTS AND DISCUSSION

A.X-Ray Fluorescence Analysis

The chemical analysis result from XRF analysis is shown in Table 1. Silica is absent in raw leaves because leave contains a high amount of organic volatiles. The organic material consists of cellulose and lignin which can be turned to carbon dioxide (CO_2) and carbon monoxide (CO).

Table-1. Chemical composition of raw and product silica

Component	Raw (%)		Product Silica (%)	
	Toddy leave	Sugarcane leave	from Toddy leave	from Sugarcane leave
SiO_2	-	-	83.875%	67.806%
K_2O	97.199 %	88.041%	9.895%	22.401%
CaO	-	7.299%	3.547%	5.501%
Cl	0.404%	-	-	-
Fe_2O_3	1.853%	1.603%	2.288%	2.643%
MnO	0.229%	2.178%	0.3%	0.84%
Rb_2O	0.093%	0.346%	-	0.031%
SO_3	0.174%	0.250%	-	0.213%
LOI	0.048%	0.283%	0.095%	0.565%

After acid leaching and calcination processes, the products are high in silica content and significant reduction of the other metal oxides and chlorine, which are regarded as impurities [12].Chlorine was almost completely remove from both the samples while the other impurities were significantly reduced .The organic material in biomass are hydrolyzed by the citric acid[13].

B. Phase Identification by XRD

X-ray diffraction were recorded from products of raw toddy palm leaves and sugarcane leaves. The product from raw Toddy palm leaves exhibited sharp crystallinity peaks at 2ϴ values of 20.86º, 21.348º, 26.524º, 35.377º, 36.191º, 39.186º, and 40.12º. These peaks indicated d-values of 4.2548, 4.1587, 3.3577, 2.5352, 2.48, 2.279, and 2.2457 respectively.

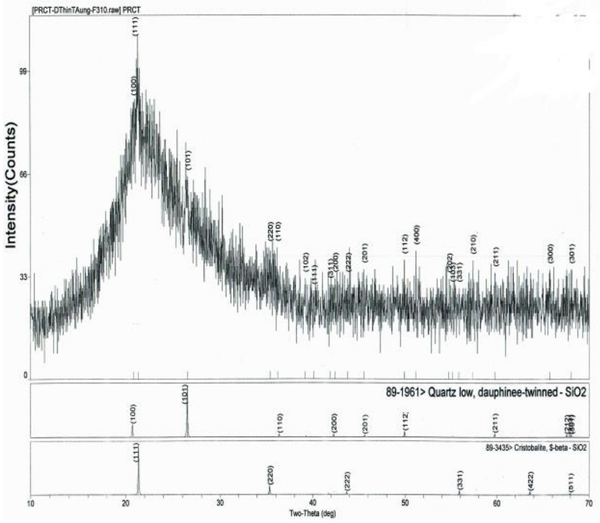

Figure -1 .XRD pattern of product silica from Toddy leaves

The sharpest crystallinity peaks at 2ϴ values of 20.687º, 26.380º, 38.582º, 42.539º, 45.631º, 49.639º, and 54.290º were obtained after calcination of raw sugarcane leaves. These peaks resulted in d-values of 4.2901, 3.3757, 2.3316, 2.1234, 1.9865, 1.8351, and 1.6883 respectively.

An XRD analysis was used to identify the two different phases of the product silica. The pattern of Toddy palm leaves confirmed that the quartz low silica formed and the pattern of Sugarcane Leaves confirmed that the quart silica formed [10].

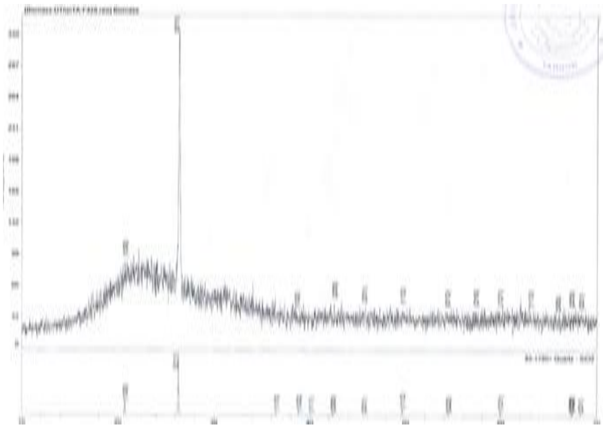

Figure-2 . XRD pattern of product silica from Sugarcane leaves

C. Surface Morphology of Products (SEM)

After sample has been leached with citric acid, the particle became spongy and porous structure of varied shape [14].

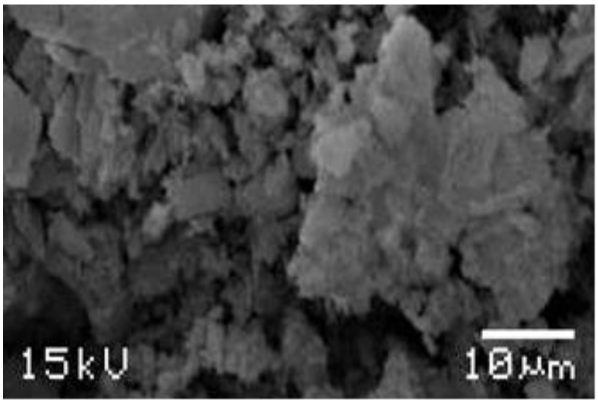

Figure-3. SEM image of product silica from Toddy leaves

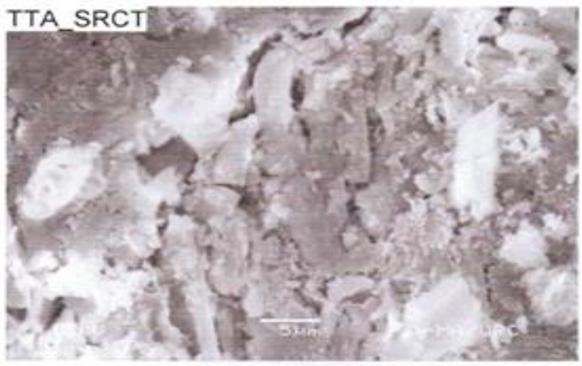

Figure -4.SEM image of product silica from Toddy leaves ash

As observed in Figs. 3 and 4, various sizes and geometry , tubular-shaped porous aggregates and spherical or and fibrous particles. The spherical particles contain mostly silica (Si) but also potassium (K), calcium (Ca), ferum (Fe) and manganese (Mn) [15].

D. Identification of results by FTIR

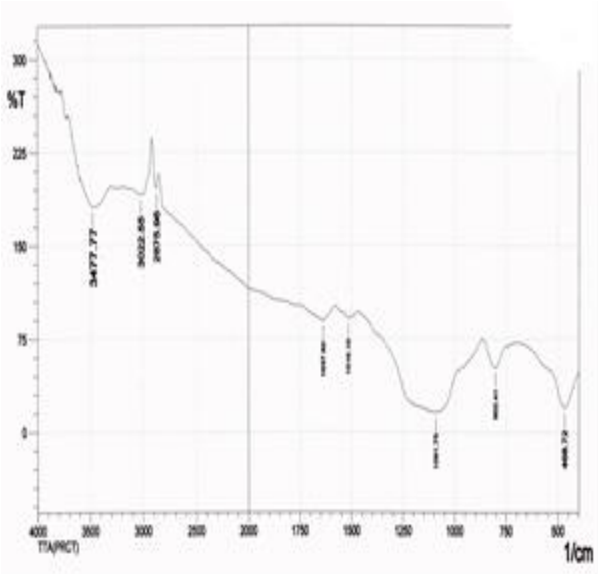

Figure-5. FTIR image of product silica from Toddy leaves

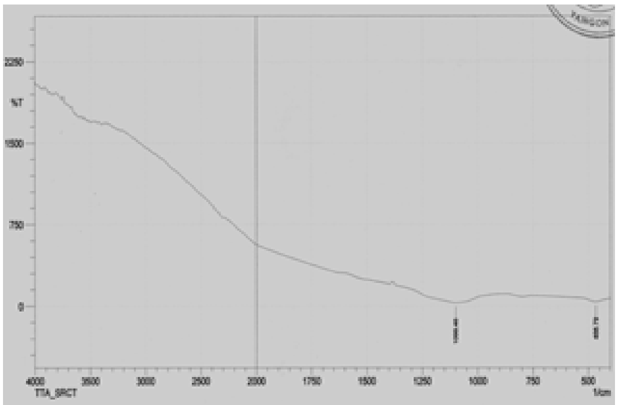

Figure- 6 . FTIR image of product silica from Toddy leaves

The silica powders obtained were confirmed by FTIR examination in Figure 3.The peak from 450 to 600 cm^{-1} corresponds to the O-Si-O bending vibration [16].

This extracted, product silica characterization and position of the peaks were identical with pure silica. Peak number of two samples is 468.72cm^{-1}.

The vibration modes of the gel network are appeared in spectrum between 700and 1200cm^{-1}[17].

The bonds from 1630 to1647cm^{-1} belongs to C-O group [16].The broad band between 2850 and 3700$cm^{-1}$is due to absorbed water OH and silanol OH groups[17].In all figures, IR images describe the major chemical groups present in leaves (Toddy& sugarcane) and extracted silica.

4. CONCLUSION

Citric acid is seen as a viable alternative to replace strong acid in the leaching process. This is because, as an organic acid, it has low level of hazardous effect comparing to the usage of a stronger acid. This study is in line with other research all over the world that is focusing on ways to establish environmentally benign process to produce silica from agricultural wastes.

This study had shown that it is possible to extract crystalline silica from toddy palm leaves and sugarcane leaves using organic acid leaching method. It consists of a citric acid leaching and calcination process. Citric acid leaching treatment is significantly useful and effective to remove the impurities and increased the purity of silica in toddy palm leaves.The silica content can be raised by studying the optimum extracting conditions (solution temperature, reaction time and concentration of citric acid).

5. APPENDIX

A. Preparation of 6%citric acid solution

500ml amount of 6% citric acid solution would be made from 30.8g of dry powder citric acid and 480.8ml of distill water.

B. Determination of moisture

$$\text{Percent of moisture Content} = \frac{(\text{initial mass}) - (\text{final mass}) \times 100}{(\text{initial mass})}$$

C. Determination of Ash content

$$\text{Percent of ash Content} = \frac{(\text{initial mass}) - (\text{final mass}) \times 100}{(\text{initial mass})}$$

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