Effect of Acid Treatment on Extraction of Silica from Cogon Grass by Using $C_6H_8O_7$ and HCL Acid

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Abstract. Cogon grass is a type of agricultural wastes that can easily be found around the world. A cogon grass contains with relatively low lignin (5.67%), ash (8.24%) and acceptable holo-cellulose and cellulose solubility of 64.9% and 37.1% respectively. The focus of this paper is to analyse the effect of acid treatment of silica extraction of cogon grass due to silica as a raw material that can be apply in industrial. Parameters such as concentration of acid and weight percentage used in cogon grass during organic and inorganic leaching method were also compared. In this paper, citric acid ($C_6H_8O_7$) and hydrochloric acid was used for production of silica from cogon grass. Samples were characterized using Scanning Electron microscopy (SEM), X-ray diffraction (XRD) and Thermal gravimetric Analysis (TGA). The result indicates that highest percentage silica of cogon grass after leaching was on 1.0M via HCL acid leaching and has an amorphous structure.

1. Background of study
Cogon grass known as Imperata cylindrica was popularized from Japan to the United States in 1912, then purposely introduced as a ravage crop in 1920 [1]. However, the plant was unsuccessful as a ravage crop due to high silica content on cogon grass itselfs. Cogon grass is a C₄, rhizome producing perennial plant that can reach heights of three meters, but typically grows to heights of 1.2 meters. Cogon grass spread lusty, through seeds, and not lusty, through rhizomes [2]. Many author mostly do not mentioned detailed in literature that silica content of cogon grass has not been recognized yet. However, other researcher stated that cogon grasses are existing a lot of silica impurities and can be used to produce high purity amorphous silica due to smooth blades were detected to be highly deposited at the boundaries of cogon grass [3]. In addition, high-purity silica may be obtained by removing minerals in the biomass through acid leaching treatment [4]. Cogon grass shows the lowest amount of holo-cellulose which is 64.9% than C.tataria (70.5%) and Palmyra palm fruit (68.5%). Commonly, holo-cellulose is the total content of hemicellulose and cellulose in dried materials and usually should accounts for 65 to 75% of the plant dry weight in order to be latent alternative fibre [5].

Chemical analyses were executed for each property under specific methods and normal values of cogon grass were summarized in Table 1. The measured data characterized by cogon grass with rather ash (8.24%), low lignin (5.67%) and acceptable holo-cellulose and cellulose solubility of 64.9% and 37.1% respectively. Chemically, holo-cellulose and cellulose are important parameters in measuring the suitability of material for paper production. Imperata cylindrical (64.9%) shows the lowest amount of holo-cellulose than Palmyra palm fruit (68.5%) and C.tataria (70.5%). Generally, holo-cellulose is the
total content of cellulose and hemicellulose in dried materials and usually should accounts for 65 to 75% of the plant dry weight in order to be potential alternative fibre [6]. Therefore, the amount of holo-cellulose obtained in cogon grass is acceptable to be applied in paper production.

### Table 1. Comparison of chemical composition of Cogon grass by published non-wood (Sridach, 2010)

| Property             | Species                  | L.cylindrica (Kassim,2015) | C,tataria (Tutus,2010) | Switch grass (Madakazde,2010) | Palmyra grass (Sridach,2010) |
|----------------------|--------------------------|-----------------------------|------------------------|-----------------------------|-----------------------------|
| Holo-cellulose       |                          | 64.9                        | 70.5                   | n.a                         | 68.5                        |
| Cellulose            |                          | 37.1                        | 40.1                   | 41.2                        | 37.0                        |
| Lignin               |                          | 5.67                        | 24.5                   | 23.9                        | 18.5                        |
| Ash                  |                          | 8.24                        | 7.83                   | 4.83                        | 0/64                        |

2. Materials and method

Cogon grass was provided by the Universiti Teknologi MARA (UiTM) laboratory. Silica is obtained from the cogon grass in this research work. Firstly, 20 grams of cogon grass was prescribed become powder through the process of milling in 30 minutes to produce fine powder. Next, cogon grass was ground via sieving machine for 10 minutes to turn into fine powder before the acid leaching process. Then, cogon grass powder with a mean particle size of 45 μm was put into a 500 ml of HCL acid solution into the beaker. The concentrations of 1.0M were used for this research at different acid used. A constant temperature of 60°C was set up for hot plate magnetic stirrer and the beaker was placed on it. The water rinsing treatment of the cogon grass was carried out with the distilled water at room temperature after the acid leaching process to eliminate the both acid content from husk. The samples were dried for 60 minutes at 110°C in the furnace. Silica powder gained from the process of extraction underwent characterization technique. The acid leaching process then replace with HCL to citric acid (C₆H₅O₇)

2.1 Morphology Surface Analysis

The Hitachi SU3500 instrument was used. A diameter of 15mm each samples coated with aluminium in an aluminium sputter coater for 60 seconds at 15mA current output. The aluminium coating was necessary to ensure a conducting surface was obtained for electron bombardment and characterization. An elimination of electron charging effects the SEM was operated at 3kV and a working distance of 15mm. The selected areas of interest were focused and micrographs were taken. Software of Smart Insight EDAX was used for EDX analysis.

2.2 Thermal gravimetric analysis (TGA)

Thermal analyses were performed using thermogravimetric differential thermal analysis TG=DSC equipment (Mettler Toledo/ TGA/DSC1) apparatus in alumina crucibles with ambient air supplement of 150 ml per minute and heating rate was 10°Cmin⁻¹. Nitrogen gas was used as ambient air supplement. The samples used had 20 mg under air flux and were heated from room temperature until 800°C.

3. Discussion

3.1 Morphology Surface Analysis

The morphology surface of the particle size and the agglomeration of nature at the ground samples were examined by Scanning Electron Microscope (SEM). In order to find out the effect of chemical treatment of silica and carbon. Energy dispersive x-ray spectroscopy (EDX) act as determination of the elements on images at different magnification from SEM. The silica and organic impurities distribution of untreated rice husk particle were analysed by using EDX. Figure 1 shows the image of (a) untreated cogon grass powder, (b) 1.0M of treated cogon grass via citric leaching treatment and (c) 1.0M of treated cogon grass via HCL leaching treatment respectively at magnification of 2000x.
Figure 1: SEM images and EDX analysis of (a) untreated Cogon grass (b) 1.0M of treated cogon grass via citric leaching treatment and (c) 1.0M of treated cogon grass via HCL leaching treatment respectively at magnification of 2000x.

Figure 1(a) illustrates the SEM image of untreated Cogon grass at magnification of 2000x respectively. It shows that the particles have uneven porous surface (irregular shape), uniform and unblemished with a smooth surface. This is due to the sample is not treated with any chemical or any treatment. Energy dispersive X-ray spectroscopy (EDX) test also handled to make quantitative chemical analysis of Cogon at specific spot. It was detected that SiO$_2$ is the most concentrated in weight percent which is 49.6 wt % than other impurities. The inorganic impurities, mainly carbon (C) and platinum was also still present in cogon and concentrated in the surface. Platinum was present in SEM image due to coating process before. Figure 1(b) observed that cogon grass particles has an uneven small porous surface that giving the particles a disorganized cylindrical shape. The small porous appear when citric acid takes an action to remove metallic impurities and silica extract during leaching treatment. The weight percentage of treated cogon for citric acid increase compared with untreated rice husk. Citric acid is an organic and weak acid that insufficient or incapable to extract more silica or remove impurities metallic that resulting the metallic elements such as carbon and platinum have high weight percent rather than silica. Figure 1(c) show SEM micrographs of the cellulose fibres exposed an agglomerated aspect of elongated fibres with thin extremities and persisted along the fibre surfaces, giving them a rough aspect HCL resulted in more degradation. At this concentration, the particle size of the sample was increased and rough surface consist of non-cellulosic materials was added. Some cracked fibre are also appear due to extraction process and it could also reduce the feature and strength properties of particles. Besides, weight percentage of silica for 1.0M cogon grass via HCL acid was 85.8% higher than untreated cogon grass and 1.0M of citric acid. It can be conclude that the morphology surface and weight percentages silica (SiO$_2$) of 1.0M cogon grass is higher by using HCL as acid leaching treatment. Jatinder, 2014, stated in his research that the surface of cogon grass is uneven with porous surface, similar to surface morphology of cogon grass in this research work [7].
3.2 Thermal gravimetric analysis

The thermogravimetric analysis of 1.0M cogon grass at 90 minutes samples of different acid used for leaching treatment in inert atmosphere at 10°C/min is observed in Figure 2. The initial descending slope from the start of the curve to about 190°C correlated to loss of hygroscopic water at phase I (drying or evaporation). Hygroscopic is a substance is able to absorb or adsorb water from its surroundings. At phase II, mass loss from temperature of 190°C to 390°C can be isolate into two parts for 1.0M cogon grass via citric acid leaching treatment. First peak of the mass loss in the range of 190°C to 338°C was due to the volatilization and thermal decomposition of the organic part in cogon grass, while the mass loss of from 338°C to 390°C was due to oxidation process. Oxidation is the loss of electrons during a reaction by an ion, molecules or atom. Hence, thermal decomposition of untreated cogon grass starts at about 284°C which is quite late compared to acid-treated cogon grass which at range of 270°C. Besides, the HCL acid treated cogon grass sustained a greater mass loss than citric acid due to HCL acid is an inorganic acid that can remove metallic constituent. TGA curve also showed that decomposition of samples occur in stage of lignin decomposition (phase III) where the peak stable from 390°C to 800°C for all untreated and treated cogon grass respectively. Moreover, an increase in heating rate caused earlier consideration of thermal degradation which basically resulted in an earlier completion of mass loss phenomenon.

![TGA curve of Untreated Cogon grass and 1.0M Treated cogon grass at 90 minutes of different acid leaching treatment](image)

**Figure 2:** TGA curve of Untreated Cogon grass and 1.0M Treated cogon grass at 90 minutes of different acid leaching treatment

4. Conclusion

The properties of silica dioxide (SiO$_2$) formed via acid leaching treatment process has been deliberated. The samples were prepared and blend with 1.0M of C$_6$H$_8$O$_7$ for 60 minutes. 3 samples were evaluated using SEM and EDX through characterization techniques and it is recognized that highest silica content of 1.0M of cogon grass increased from 49.6% to 53.6% by citric acid solution while for HCL acid leaching treatment increase immensely from 49.6% to 85.8%. The silica extraction and its morphological research are done by using SEM. Therefore, untreated cogon grass showed that SiO$_2$ enclose of smooth surface while for 1.0 M cogon grass of citric acid showed the small porous surface structure and for 1.0 M cogon grass of HCL acid show the particle is an agglomeration and rough surface. As a conclusion for TGA analysis can be summarize that between these two different acid used as a leaching treatments which is citric acid and HCL acid, the most effective acid used to this acid leaching treatment was HCL acid due to HCL is a strong acid compared with citric acid that can extract more silica content and remove others impurities. Hence, by using acid as a leaching treatment and increase the concentration, the degradation of thermal decomposition cogon grass is faster. Furthermore, through reuse techniques from waste materials; it is possible in improvise the environment quality and lower cost in the production of silica by using the waste materials.
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