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Erianthus Plant: A Rich Silica Source for Extraction of Amorphous Silica

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Abstract. In this paper, *Erianthus* plant, a grass type of lignocellulosic biomass, is presented as an alternative source for the production of amorphous silica. Thermal treatment (combustion) of *Erianthus* plant under a controlled temperature of 600–900°C produces *Erianthus* Ash (EA). Then, silica powder was extracted from EA by the chemical extraction method. In this work, the effect of treatment temperature on the preparation of EA and extracted silica is studied. The EA samples and extracted silica are noted as EA_{600} – EA_{900} and Si_{600} – Si_{900} respectively with respect to the treatment temperature. To evaluate the effect of the concentration of NaOH solution on the purity of silica, NaOH solution (2–3 N) is verified in this work. The results revealed that the pure amorphous silica can be extracted using a 2.5 N NaOH solution from EA_{800}. The percentage of amorphous silica with a purity of about 99% was confirmed by X-Ray Fluorescence (XRF).

Keywords: Ash, *Erianthus*, Extraction, Silica, Sodium Silicate.

INTRODUCTION

Erianthus plant, a grass type of lignocellulosic biomass, belongs to the tribe *Andropogoneae* under the grass family *Gramineae* (Amalraj et al. 2008). It belongs to the wild relative of sugarcane (Aitken et al. 2007) and can be used in the sugarcane breeding program due to its excellent biological traits and genetic relatedness to the sugarcane (Wang et al. 2019). As a warm-season species, it is better to grow under unfavorable environmental conditions such as submerged conditions and acidic soil, as well as under soil drought during the dry season. In Southeastern Asia, including Myanmar, *Erianthus* plant can be grown in subtropical and tropical regions (Hu et al. 2017). In Myanmar, *Erianthus* plant is well known as giant grass as well as bushes or useless plant.

Erianthus plant is composed of cellulose, hemicellulose, and lignin (Yamamura et al. 2013) and thus, it is receiving much attention as an energy crop, a lignocellulosic biomass resource, for the production of biofuel (Hattori and Morita 2010, Ra et al. 2012, Dao et al. 2013, Hu et al. 2017). On the other hand, Yamamura *et al*. reported that *Erianthus* plant is rich in silica sources and they found that the percent of silica contained even in the leaf ash is more than 60% (Yamamura et al. 2013). However, to the best of our knowledge, no study has investigated the production of silica powder from *Erianthus* plant. Thus, this paper is attempted to produce amorphous silica from the *Erianthus* plant.

In Myanmar, the utilization of *Erianthus*

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plant is limited and it has not been successfully utilized in industries. Therefore, it results in tremendous waste generation and environmental pollution. Therefore, the plant will gain an additional value whether it can be used as a raw material for the production of a silica-based compound. Furthermore, it can be presented a novel utilization area for the efficient evaluation of this type of waste.

To investigate the experimental conditions for the production of amorphous silica from the *Erianthus* plant, thermal treatment prior to the chemical extraction method is referred from previous researches which investigated the extraction of silica from Rice Husk Ash (RHA) (Yuvakkumar et al. 2014, Rangaraj and Venkatachalam 2017). In this study, the thermal treatment temperature is considered at 600–900°C for 2 hr. In the chemical extraction method, the alkali extraction process is conducted followed by the acid precipitation method to produce amorphous silica. The effect of NaOH solution on the purity of produced silica is also investigated. The structural features of the extracted silica were thoroughly characterized by various techniques such as X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Fourier Transforms Infrared (FT-IR), Scanning Electron Microscopy (SEM), and Energy Dispersive Spectroscopy (EDS) analysis.

MATERIALS and METHODS

Raw Materials Preparation

Raw *Erianthus* plant was collected from Thanlyin Township in Yangon Region, Myanmar in the winter season. The plants were washed with water to remove dirt and other contaminants. Then, the plants were sun-dried for one day and cut into small pieces. The pieces were dried again in an oven to remove moisture. The dried pieces were characterized by XRF to observe the composition.

Thermal Treatment Process

Erianthus Ash (EA) was produced by thermal treatment (combustion) of raw *Erianthus* plant under certain controlled temperature and time. Thermal treatment was performed in a muffle furnace at 600–900ºC for 2 hr with a heating rate of 20ºC/min. The samples which are noted as EA_{600} – EA_{900} were stored in a desiccator after the thermal treatment process. And then, the EA samples were characterized by XRF to determine the amount of silica content in percent.

Extraction of Silica from EA

The extraction experiment was referred to by the previous work (Yuvakkumar et al. 2014, Rangaraj and Venkatachalam 2017). The EA collected after combustion was leached with 100 ml of 6 N hydrochloric acid solution under stirring at 70ºC for 1.5 hr. Then, the acid-leached EA was filtered and washed with distilled water until the filtrate became neutral. After washing, EA was dried in an oven at 105°C for 24 hr to remove water. In the extraction process, EA was refluxed with sodium hydroxide solution (2–3 N) under stirring for 2 hr at 80ºC to obtain sodium silicate solution. Then, this solution was filtered to extract the pure silica and remove impurities. Sodium silicate solution was precipitated by acidification process using 5 N sulfuric acid solutions. At this time, sodium silicate solution was accomplished under constant stirring at the controlled condition which the temperature is around 80±5ºC for 1 hr. Finally, silica gel was formed by dropping sulfuric acid solution until pH level reaches 8±0.5. The precipitated silica

(silica gel) was washed repeatedly with distilled water until the filtrate become completely free alkali. The clean silica gel was dried in an oven at 100±5°C for 12 hr to remove water. Finally, the pure silica powder was obtained after calcination in a muffle furnace at 450°C for 2 hr. The extracted silica samples were noted as Si_{600} – Si_{900} with respect to combustion temperature.

RESULTS AND DISCUSSION

Characterization of Raw *Erianthus* Plant

Table 1. Chemical Composition of Raw *Erianthus* Plant

Component	Percent (wt %)
SiO_2	43.29
K_2O	27.44
SO_3	13.58
CaO	8.30
P_2O_5	5.35
Rb_2O	0.02
Fe_2O_3	1.16
MnO	0.41
ZnO	0.08
SrO	0.01
CuO	0.15
Cr_2O_3	0.11
PbO	0.06
Ag_2O	0.04

To examine the content of silica in the raw *Erianthus* plant, its chemical composition was characterized by XRF as shown in Table 1. It was found that the plant is mainly composed of silica, SiO_2, which has an amount of 43%. Other metallic impurities are also present in *Erianthus* plant as minor elements. Potassium oxide (K_2O) is noted as the major impurities.

Table 2. Chemical Composition of EA at Various Temperature

Component (wt %)	Raw *Erianthus* Plant	EA_{600}	EA_{700}	EA_{800}	EA_{900}
SiO_2	43.29	53.6	58.1	68.0	64.82
MgO	NA	NA	NA	NA	9.97
K_2O	27.44	20.95	18.31	12.37	8.52
SO_3	13.58	9.84	9.80	5.96	4.98
P_2O_5	5.35	5.48	4.88	6.51	5.78
CaO	8.30	5.72	5.42	6.28	5.12
Cl	NA	3.85	2.89	NA	NA
CuO	0.15	0.02	0.02	0.02	0.01
TiO_2	NA	0.02	0.02	0.03	0.02
Fe_2O_3	1.16	0.23	0.28	0.48	0.37
MnO	0.41	0.19	0.21	0.28	0.33
ZnO	0.08	0.03	0.02	0.02	0.03
SrO	0.01	0.02	0.02	0.03	0.03
Rb_2O	0.02	0.02	0.02	0.01	0.01
Cr_2O_3	0.11	0.01	NA	0.01	0.01
Br	NA	0.01	0.01	NA	NA
Ac	NA	0.01	NA	NA	NA
PbO	0.06	NA	NA	NA	NA
Ag_2O	0.04	NA	NA	NA	NA

NA - Not Available

Effect of Temperature on Thermal Treatment Process

The chemical composition of EA_{600} – EA_{900} is shown in Table 2. It was obvious that SiO_2 percent in ash samples after thermal treatment were increased when compared to that in the raw *Erianthus* plant.

It can be seen that percent SiO_2 in EA samples is slightly increased up to 68% at 800°C while it was decreased to 64% at 900°C. This might be due to the dissociation of potassium at this combustion temperature of 900°C. At high combustion temperature, the surface of ash particles begins to melt and potassium can block the transportation of carbon dioxide and oxygen which can cause the increasing amount of unburnt carbon

(Ugheoke and Mamat 2012). The present study discussed that the thermal treatment process affected the percent composition of SiO_2 in EA samples. Therefore, the suitable burning temperature for the production of EA is 800°C for this study.

Effect of Thermal Treatment Temperature on Extracted Silica

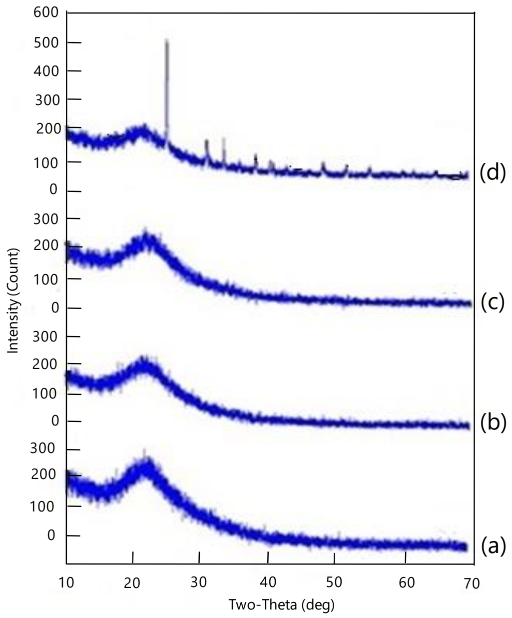

Fig. 1: XRD patterns of extracted silica (a) Si_{600} (b) Si_{700} (c) Si_{800} (d) Si_{900}

The phase formation (amorphous or crystalline) of extracted silica was determined by XRD as shown in Figure 1. The broad diffused peaks with maximum intensity at 2θ = 22° are observed in Si_{600} – Si_{800}, indicating the amorphous nature of silica. However, the presence of a sharp peak at 2θ = 25° was observed in Si_{900}. This indicates that the phase of extracted silica transformed to crystallization state at 900°C. The results of the present study agreed with the previous researches which used RHA as raw materials for the extraction of silica (Deshmukh et al. 2012, Bakar, Yahya, & Gan 2016). The researchers reported that the phase of RHA-derived silica is sensitive to the combustion temperature. Similarly, this study also confirmed that thermal treatment temperature affected the phase of the extracted silica. In contrast, thermal treatment temperature, over 800°C, is not suitable to produce amorphous silica from the *Erianthus* plant.

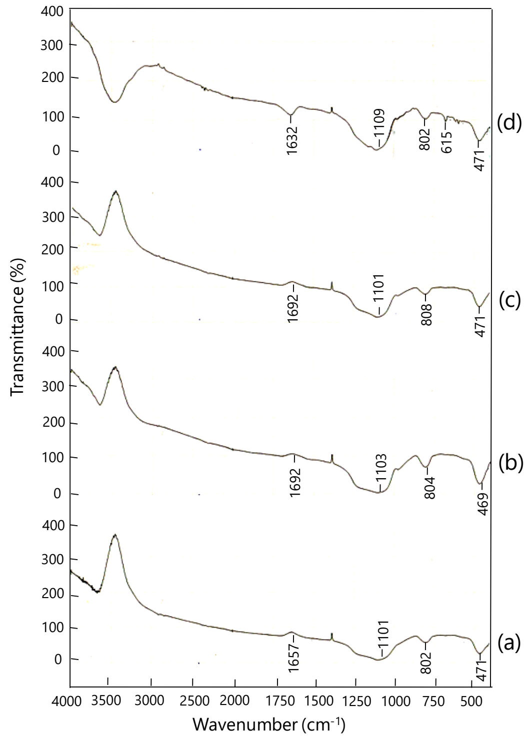

Fig. 2: FT-IR spectra of extracted silica (a) Si_{600} (b) Si_{700} (c) Si_{800} (d) Si_{900}

FT-IR spectra of extracted silica, Si_{600} – Si_{900}, are shown in Figure 2. The IR band at around 471 cm^{-1} belongs to bending vibrations of O-Si-O groups. The strong band around 808 cm^{-1} and 1109 cm^{-1} reveal the existence of Si-O-Si symmetric and asymmetric stretching vibrations. The regions around 1692 cm^{-1} assigned to the absorption band for H-O-H bending vibration in water (Amutha, Ravibaskar, and Sivakumar 2010, Music, Filipović-Vinceković, and Sekovanić 2011).

(a)
(b)
(c)
(d)

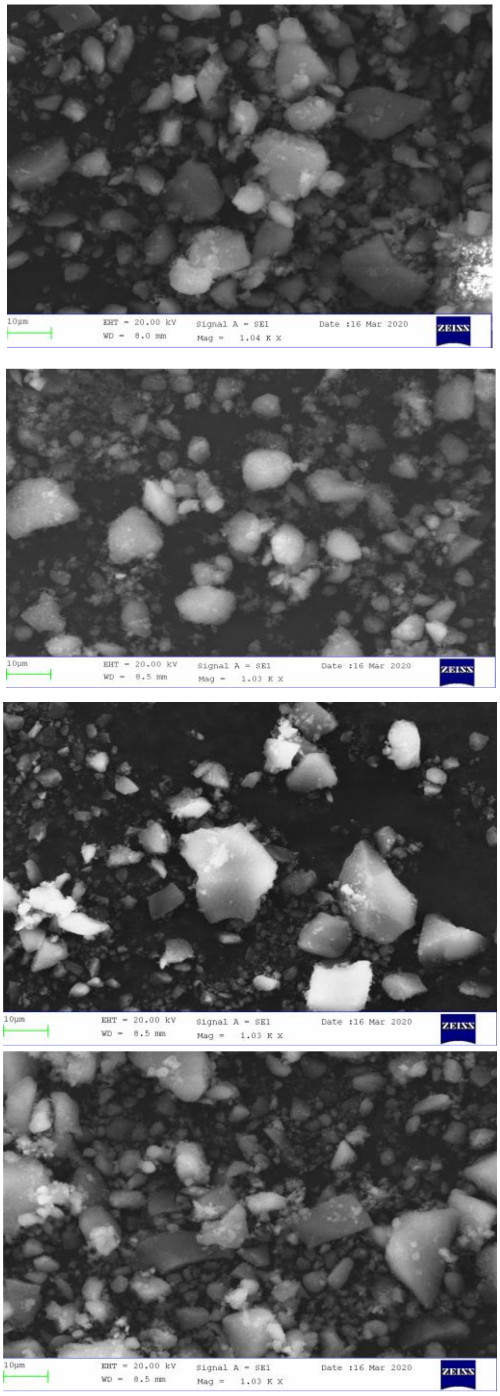

Fig. 3: SEM analysis of extracted silica (a) Si_{600} (b) Si_{700} (c) Si_{800} (d) Si_{900}

Thus, FT-IR spectra of all extracted silica, Si_{600} – Si_{900}, indicate the major functional groups of silica. Notably, the band at around 615 cm^{-1} might correspond to cristobalite SiO_2 in Si_{900} (An et al. 2011, Azmi et al. 2016). This fact correlates with XRD analysis which indicates that the phase of silica transformed to cristobalite form at thermal treatment temperature, 900°C. This implies that thermal treatment temperature is affected by the phase of the extracted silica.

SEM images of extracted silica are shown in Figure 3. It was observed that the shape of silica particles of all samples, Si_{600} – Si_{900}, was irregular morphology with agglomeration. The size of particles was displayed approximately about 10 µm. According to the SEM results, it can be suggested that thermal treatment temperature does not affect the morphology of the extracted silica in this study.

Effect of NaOH Solution Concentration on Purity of Silica

The chemical composition of the extracted silica was determined by XRF and the results are shown in Table 3.

Table 3. Composition of Silica Extracted from EA_{800}

Component (wt %)	2 N	2.5 N	3 N
SiO_2	97.27	99.00	98.34
K_2O	0.04	NA	0.30
SO_3	0.63	0.60	0.04
CuO	0.50	0.03	0.01
TiO_2	0.03	0.02	0.31
Fe_2O_3	0.70	0.17	0.20
MnO	0.01	0.04	0.10
ZnO	0.17	0.03	0.20
ZrO_2	0.02	NA	0.10
CaO	0.60	0.10	0.10
Rb_2O	0.01	NA	0.10
Cr_2O_3	0.02	0.01	0.20

NA - Not Available

It was observed that the purity of extracted silica with 2.5 N NaOH solution has confirmed the highest silica content, 99%. Notably, K_2O which is the main metallic impurities could be removed at this condition. Thus, the results reveal that NaOH solution concentration has the effectiveness of the purity of silica. This result bears a resemblance to the investigation on production of silica from RHA reported by Yuvakkumar *et al.* They investigated that NaOH affects the production of high purity nano-silica powder from RHA (Yuvakkumar et al. 2014).

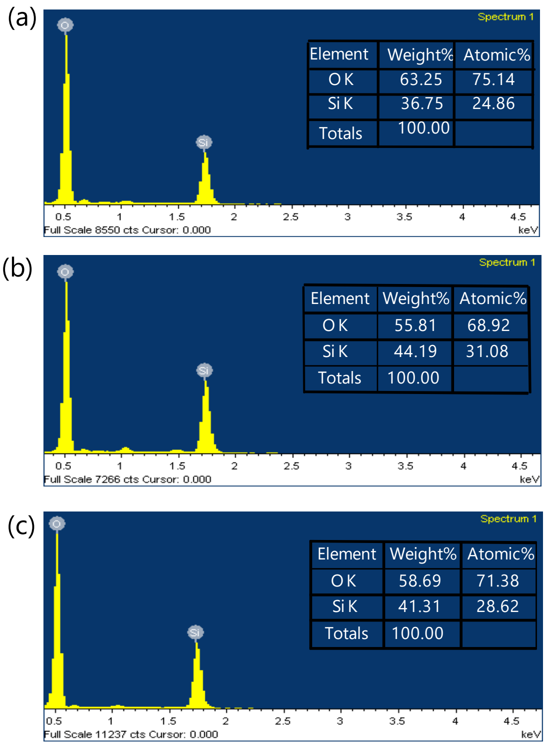

Fig. 4: EDS pattern of extracted silica powder with various NaOH solution (a) 2 N NaOH (b) 2.5 N NaOH and (c) 3 N NaOH

The EDS patterns described in Figure 4 also confirmed that extracted silica samples predominantly contain elements such as Si and O as the results indicate the absence of other elements such as K and C. Hence, the results confirm the formation of pure silica structures. Notably, the silica sample extracted with 2.5 NaOH solution contains 44.19 wt% silicon and 55.81 wt% oxygen while the actual composition of silicon in silica is 46 wt% (Yuvakkumar et al. 2014). From the EDS results, it can be confirmed that the silica sample extracted with 2.5 N NaOH solution contains higher purity of silica, 97% than the two others. Since the other samples extracted with 2 N and 3 N NaOH solution contain 80% and 90% purity of silica respectively. Thus, the EDS results also confirm the effectiveness of the concentration of NaOH solution, and 2.5 N is noted as an optimum condition to extract the high purity of silica.

The yield of silica extracted from ash at various temperatures with 2.5 N NaOH solution is shown in Table 4.

Table 4. Yield of Silica at Various Temperature with 2.5 N NaOH Solution

Sample	Yield (%)
Si_{600}	74
Si_{700}	75
Si_{800}	77
Si_{900}	70

CONCLUSIONS

This work is contributed to the optimization of experimental conditions for the production of amorphous silica from *Erianthus* plant. In this study, *Erianthus* plant was thermally treated at 600 – 900°C to produce EA and then chemical extraction of EA was conducted to produce amorphous silica. It appears that the production of amorphous silica from *Erianthus* plant can be effective. The combustion temperature for the production of EA is also studied and it was found that the EA produced at the temperature of 800°C for 2 hr duration time is the optimum condition in terms of amorphous silica. The effect of NaOH solution on the extraction of silica is demonstrated. It was observed that 2.5 N NaOH solution can be produced high purity amorphous silica, 99% from EA_{800} in this study.

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