PREPARATION AND CHARACTERIZATION OF SELECTED BIOCHAR FROM BIOLOGICAL WASTES

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Abstract

Biochar is a solid material that is produced by heat decomposition of biological wastes biomass. The experiments were carried out to study the characterization of the Coconut Husk and Areca nut Husk biochar. The Coconut Husk and Areca nut Husk were heated at 300˚C for 1h in muffle furnace and ground into the uniform powder. The most important properties that provide information about the fuel were proximate analysis such as moisture, ash content, volatile matter and fixed carbon contents. The fixed carbon content of Coconut Husk and Areca nut Husk were measured to be 10 mf wt%. The structural characterizations of Coconut Husk and Areca nut Husk biochar were identified by X-ray diffraction (XRD) technique. The Fourier transform infrared spectroscopy (FTIR) analysis showed the presence of a variety of functional groups of Coconut Husk and Areca nut Husk biochar. Scanning Electron Microscope images of the biochar were obtained for morphological features analysis.

Keywords: Biochar, Coconut Husk, Areca nut Husk, Proximate analysis, XRD, FTIR, SEM

Introduction

Biomass is biological material derived from living, or recently living organisms. In the context of biomass for energy this is often used to mean plant based material, but biomass can equally apply to both animal and vegetable derived material. [Mohammad Hariz A.R, etal. 2015] Biomass is non-fossilized and biodegradable organic material originating from plants, animals and microorganisms. This includes the products, byproducts, residues and wastes from agriculture, forestry, industrial and municipal wastes. [Qian, K, etal 2015] Pyrolysis conversion process is one of the prominent methods through which biomass is converted into three significant by-products namely solid char, bio-oil and gases. [Sukiran, M. A. B. 2008)] [Abnisa, F.,etal. 2013] Biochar is a solid carbon-rich product produced when biomass such as stalks, straw, wood, fruits, seeds and leaves is heated in a closed container without or limited presence of air. [Qian, K.; etal 2015] Biochar is produced during the pyrolysis of biomass materials. [Sulaiman, F., etal 2011] [Safaba aminu Aliyu., etal 2018] Biochar is a low-cost, carbon rich material derived from the thermochemical composition of lignocellulosic biomass in limited or absence of oxygen. Compared to other types of carbonaceous materials, biochar emerges as a new cost-effective and environmentally friendly carbon material with great potential of application in many different fields. [Ahmed Y. Elnour. etal 2019] The main objective of the present study is to investigate the preparation and characterization of biochar from coconut husk and areca nut husk at 300^{o}C. In order to utilize this biochar for further application, it is desirable to understand the most important pyrolysis indicator such as proximate analysis, i.e., information of moisture, ash content, volatile matter and fixed carbon content. Fourier transform infrared

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spectroscopy (FTIR), X-ray diffraction (XRD) and scanning electron microscopy (SEM) were also investigated.

Materials and Methods

Starting Materials

Two different feed stocks of coconut husk and areca nut husk were selected for the preparation of biochar. Raw feed stocks were collected from local environment and dried properly under natural conditions. After drying, the materials were cut into desirable size pieces and then dried in air oven in batches at 105°C for the removal of moisture. The dried coconut husk and areca nut husk pieces are then sent to ball mill for grinding. The product is then ground to smaller size and the finer product is separated using a sieve. Thus final particle size of ground coconut husk and areca nut husk are less than 1 mm. And then the pyrolysis temperature used in these study at 300 ºC. The conversion of coconut husk and areca nut husk biomass into biochar was shown in Figure 1 and 2. The flow diagram of the sample preparation of two biochar was shown in Figure 3.

Figure 1 Conversion of Coconut Husk biomass into Biochar

Figure 2 Conversion of Areca nut Husk biomass into Biochar

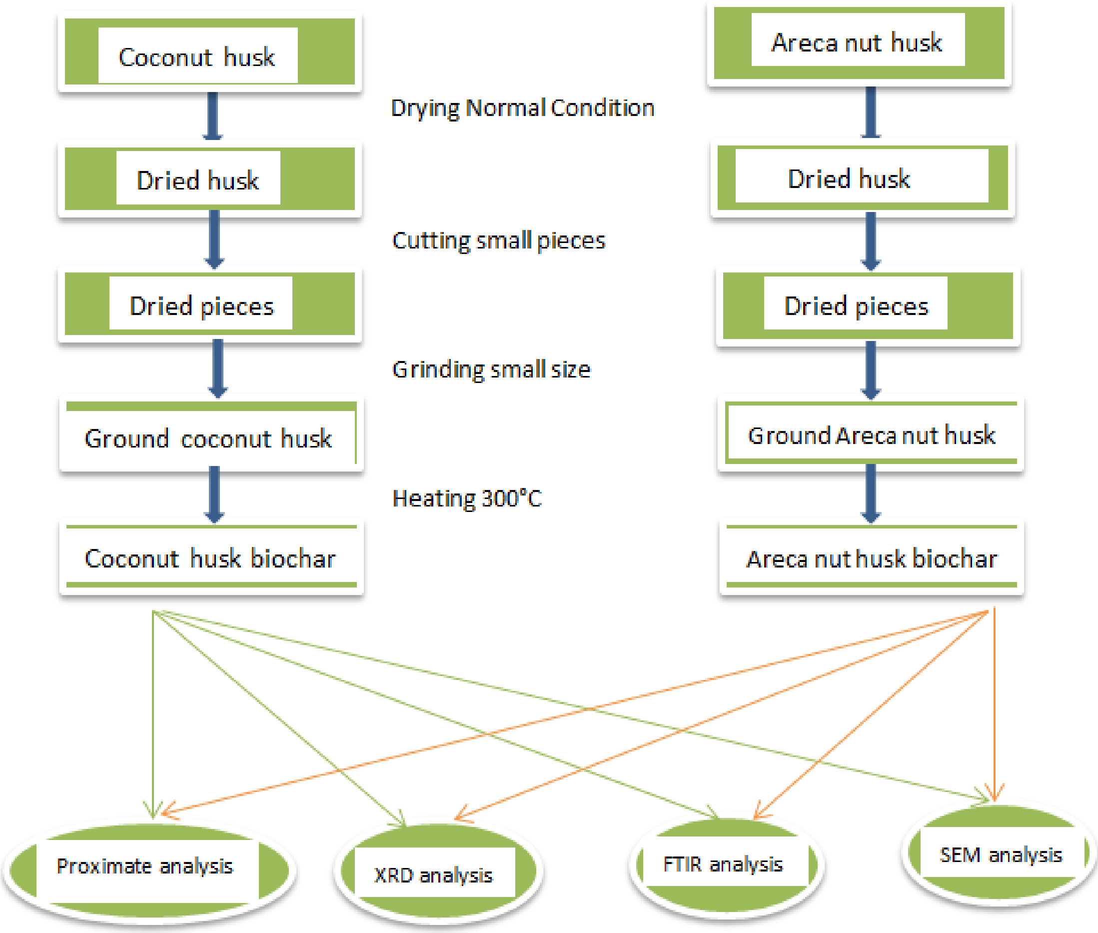

Figure 3 The flow diagram of the sample preparation of two biochar

Characterization of coconut husk and areca nut husk biochar

The biochar of coconut husk and areca nut husk were characterized to determine its fundamental properties by proximate analysis of moisture content, ash content, volatile matter content and fixed carbon content. Fixed carbon other than ash does not vaporize when heated in the absence of air. Fixed carbon is usually determined by subtracting the sum of the first three values that is moisture, ash, and volatile matter (weight percent from 100 percent).So, it is very important for economic reasons to know the moisture and ash contents of the material. The phase composition and crystal structure of the obtained carbon were analyzed on an X-ray diffractometer (Smart Lab) with (Cu/K-alpha1) radiation (λ = 1.54056°A) under a current of 50mA and a voltage of 40kV. Fourier Transforms Infrared Spectroscopic analysis of biochar was recorded on (Thermo-Scientific Nicolet iS5) spectrometer to determine its functional groups. The FTIR spectra were collected with a spectrometer using potassium bromide pellets and the wave number ranging from 400 to 4000 cm^{-1}. Surface morphology of biochar was identified by Scanning Electron Microscopy were obtained using an acceleration voltage of 15kV and a magnification x300 and x2000. (JEOL- BENCHTOP).

Results and Discussion

Proximate Analysis of coconut and areca nut husk biochar

In order to determine the volatile matter, fixed carbon and ash content in the biochar product of two different biomasses, coconut husk and areca nut husk, the proximate analysis was performed. The comparative results of the proximate analysis of the biochar are shown in Table 1. The results disclose that the proximate analysis of the coconut husk and areca nut husk were almost similar. However, areca nut husk contained a little higher moisture content than and lower than volatile matter content and ash content than coconut husk. The fixed carbon content of areca nut husk higher than coconut husk was observed.

Table 1 Proximate analysis for Coconut and Areca nut Husk

Sample	Moisture content (%)	Volatile matter content (%)	Ash content (%)	Fixed Carbon Content (%)
Coconut Husk	9.200	75.996	0.047	14.757
Areca nut Husk	9.307	68.380	0.028	22.282

XRD Analysis of Coconut Husk and Areca nut Husk Biochar

The XRD diffractogram of the coconut husk and areca nut husk biochar are represented in Figure 3 and 4 has shown several peaks, although the overall characteristic of the biochar is amorphous. There are a little sharp diffracted peaks were observed. The peaks at d spacing 3.1477°A and 2.2250°A (2Ɵ values 28.330° and 40.510°) are assigned to the presence of potassium chloride and it is consistent with coconut husk and areca nut husk biochar. These are indicated the presence of components, which were related to the crystalline forms of potassium chloride and other components of carbon and graphite peaks. The present data suggests that most of the amorphous carbon rich regions. It could be said that the coconut husk and areca nut husk biochar were amorphous material with little crystalline. The crystalline percent and amorphous percent of coconut husk and areca nut husk biochar were shown in Table 2.

Table 2 Crystallinity results of Coconut and Areca nut Husk biochar

Biochar	Crystalline (%)	Amorphous (%)
Coconut husk	3.66	96.34
Areca nut husk	5.39	94.61

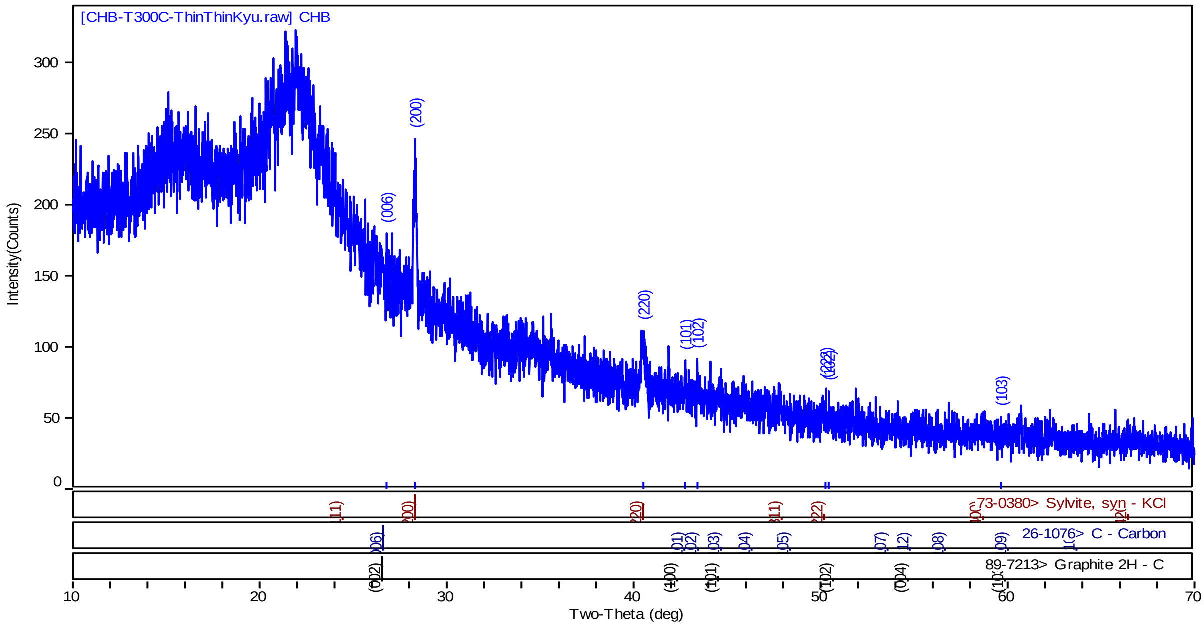

Figure 3 XRD analysis of Coconut Husk Biochar

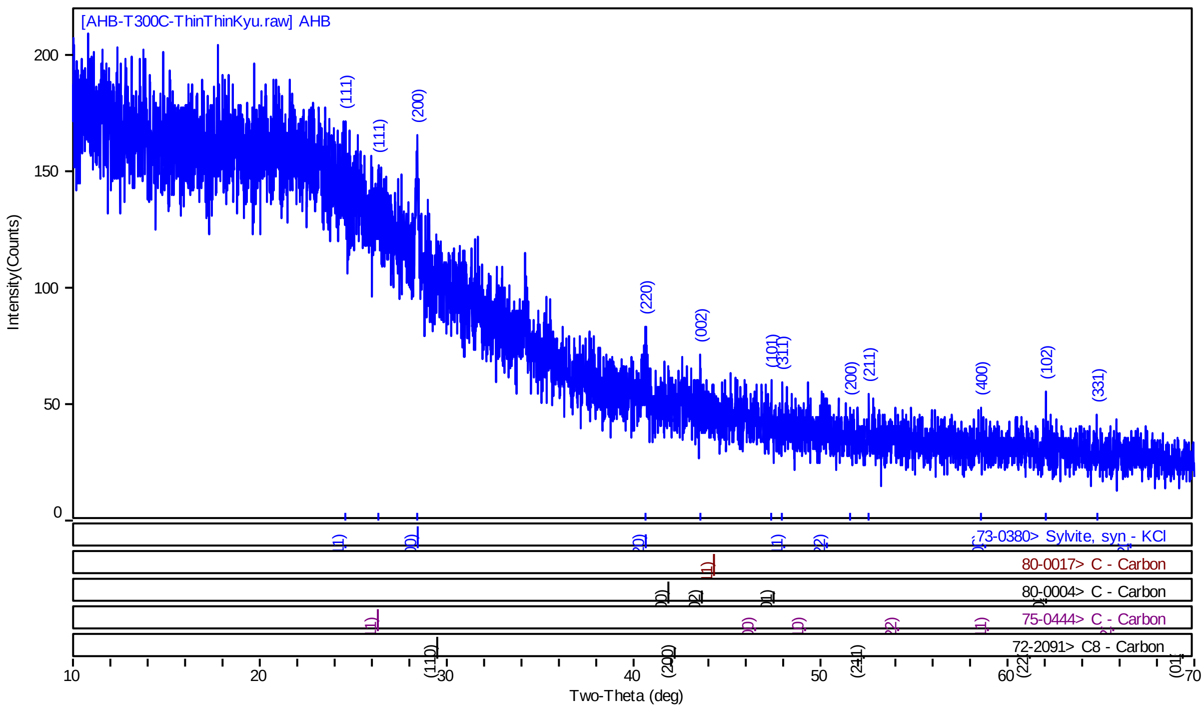

Figure 4 XRD analysis of Areca nut Husk Biochar

Functional group of Coconut Husk and Areca nut Husk Biochar

Fourier Transform Infrared spectroscopy (FTIR) is an important analysis technique which detects various characteristic functional groups present in biochar**.** The FTIR spectrum of biochar obtained from pyrolysis of coconut husk and areca nut husk at the pyrolysis temperature at 300 °C in one hour are shown in Figure 5 and 6. The O-H stretching vibrations at 3337.55cm^{-1}

and 3342.09cm^{-1} indicate the presence of phenols and alcohols and the C-H stretching vibrations at 2927.68cm^{-1} and 2930.02cm^{-1} indicate the presence of aromatic ring. A strong stretching vibration at 1700.99cm^{-1} shows the presence of C=O of carboxylic acid. A stretching vibration at 1594.29 and 1596.92 cm^{-1} shows the presence of C=C of alkenes. The C-O stretching vibration at 1212.63, 1104.36, 1029.34 and 1032.65cm^{-1} indicates the presence of alkanes. These functional groups are responsible for the sharp stretching vibration at 1029.34 cm^{-1} shows the presence of C-O stretching vibration and 1596.92 cm^{-1} show the presence of C=C strong stretching vibration. The different assignments of the FTIR spectra of coconut husk and areca nut husk biochar were summarized in Table 1 and 2.

Table 2 Peaks and functional groups from FTIR spectrum of Coconut Husk Biochar

Wave Number (cm^{-1})	**Type of vibration**	**Functional group**
3327.30	H–bonded O–H stretch (Broad strong band)	Alcohols, Phenols
2927.68	C–H stretch (strong)	Aromatic Rings stretching
1594.29	C=C stretch (medium)	Alkene
1508.09	C=C stretch(weak)	Aromatic Rings
1420.40	C–H (variable)	scissoring and bending
1212.63	C–O stretch	Aromatic
1029.34	C–O stretch (strong)	Ether

Table 3 Peaks and functional groups from FTIR spectrum of Areca nut Husk Biochar

Wave Number (cm^{-1})	**Type of vibration**	**Functional group**
3342.09	H–bonded O–H stretch (Broad strong band)	Alcohols, Phenols
2930.02	C–H stretch (strong)	Aromatic Rings stretching
1700.99	C=O stretch (strong)	Ketone , Carboxylic acid
1596.92	C=C stretch (medium)	Alkene
1508.30	C=C stretch(weak)	Aromatic Rings
1437.66	C–H (variable)	scissoring and bending
1204.75	C–O stretch	Aromatic
1104.36	C–O stretch (strong)	Alcohols, Ethers, Carboxylic acids
1032.65	C–O stretch (strong)	Ether

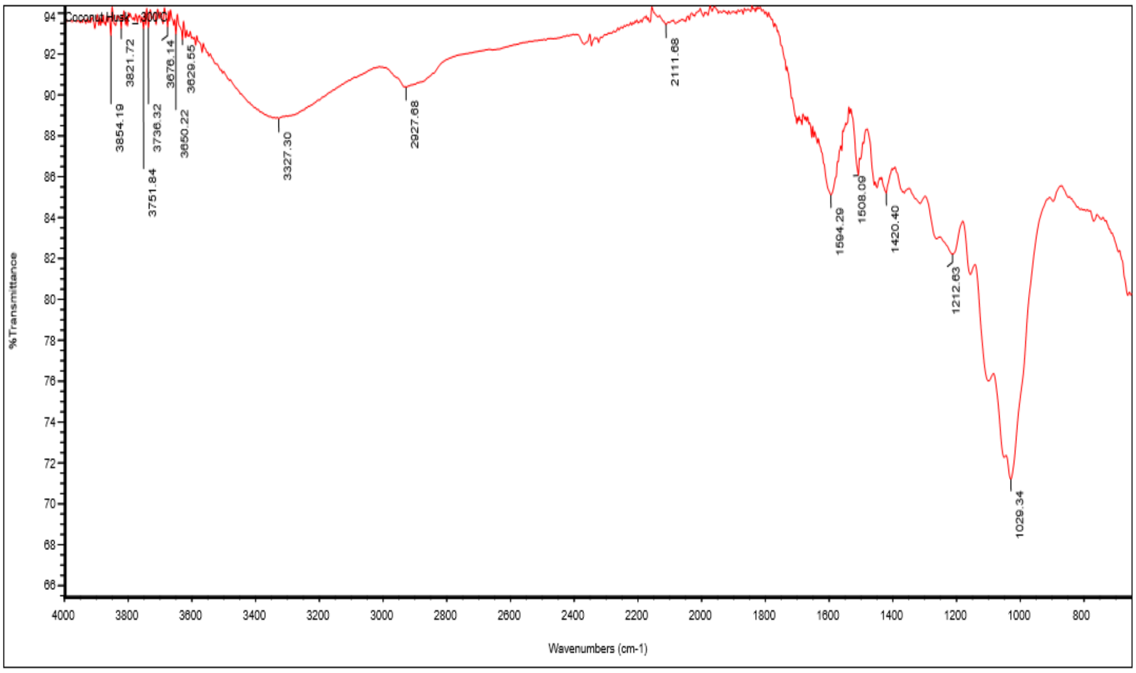

Figure 5 FTIR spectra of Coconut Husk Biochar

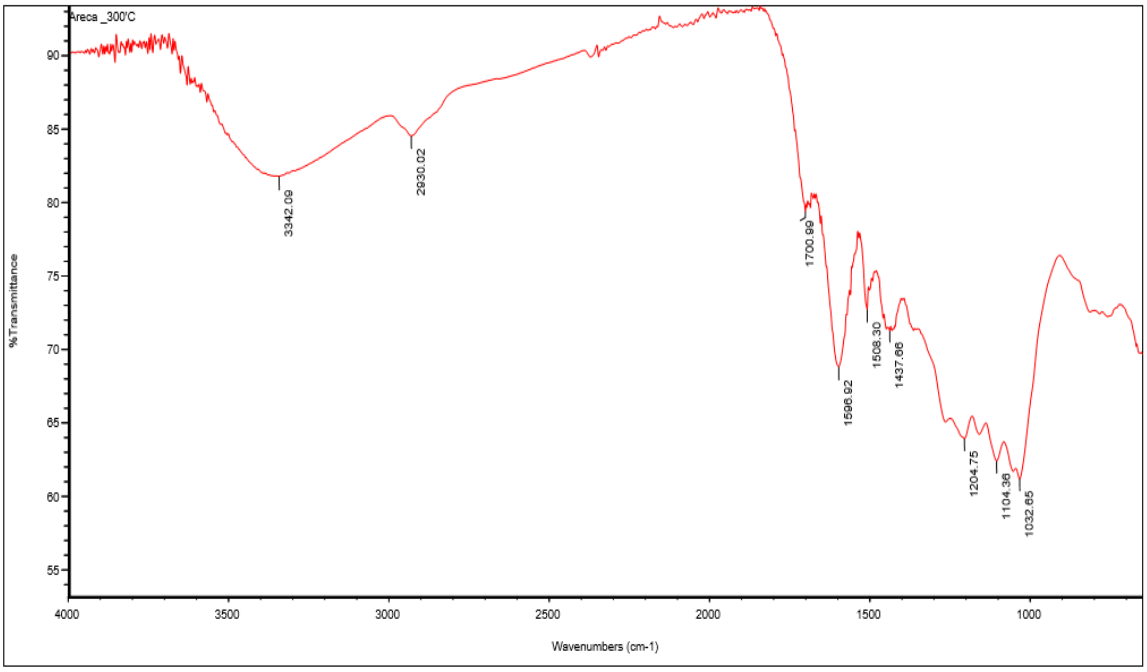

Figure 6 FTIR spectra of Areca nut Husk Biochar

Surface morphology structure of Coconut Husk and Areca nut Husk Biochar

The scanning electron microscopy (SEM) images of the prepared biochar samples of coconut husk and areca nut husk biochar are shown in Figure 7 and 8. The SEM images showed the variations in the porous structure of morphology. SEM image of coconut husk and areca husk biochar taken at 300 X and 2000X magnification. These two biochar image showed heterogeneous distribution of pores and rough texture. Presence of micropores and mesopores were detected. The size of the pores indicated that the volatile compounds had evaporated creating macro pores on the surface. The SEM images of areca nut husk biochar possess well-defined pores structure and the smooth wall dense surfaces. The size of the pores on a surface with varying diameters as follows the average diameter of coconut husk and areca nut husk was 2.20µm and 2.62µm respectively. As a result of these developed pores, that two biochar will have a high surface area.

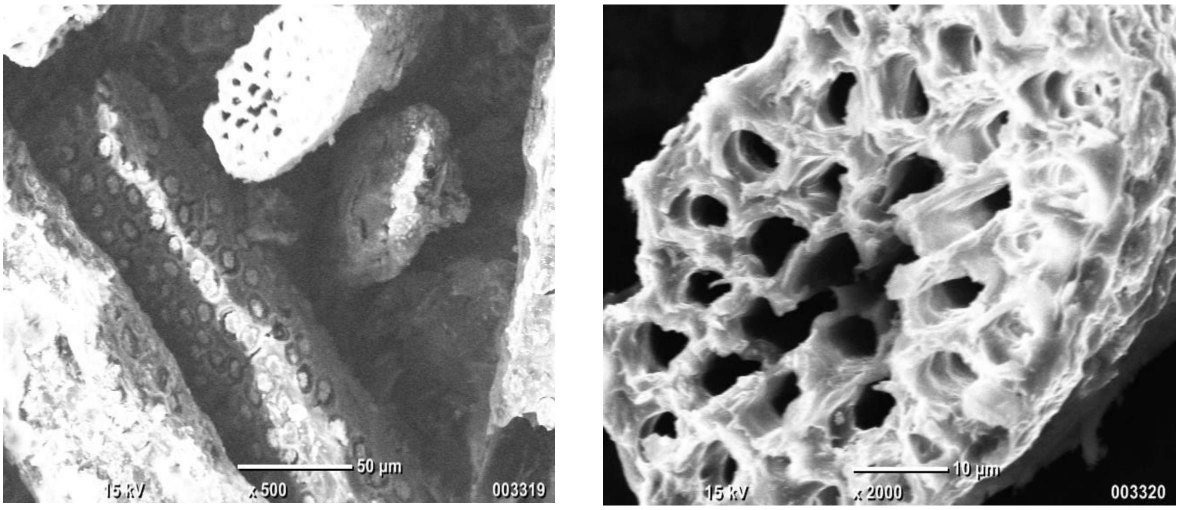

Figure 7 SEM image of Coconut Husk Biochar at (a) 500X and (b) 2000X magnification

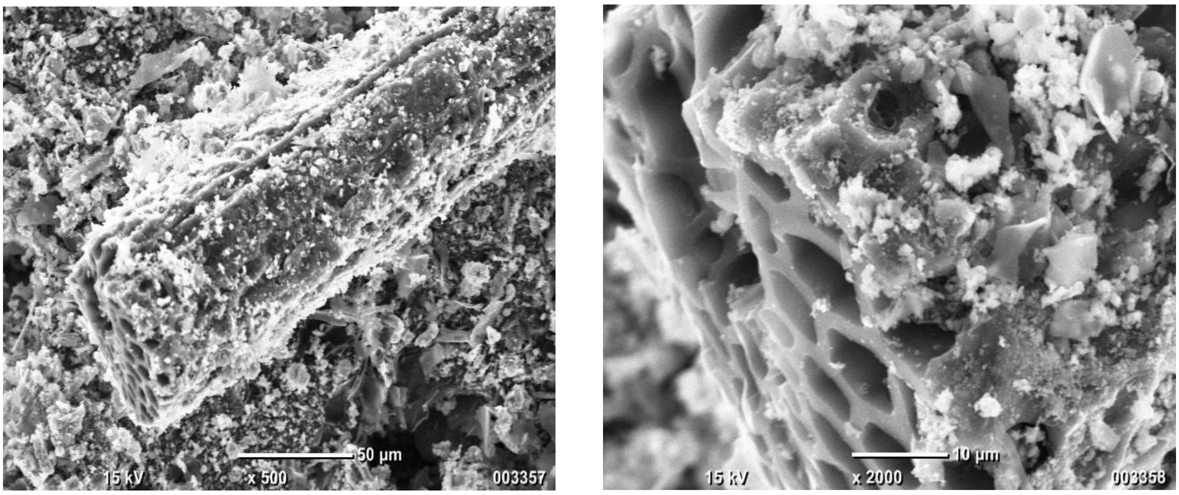

Figure 8 SEM image of Areca Husk Biochar at (a) 500 X and (b) 2000X magnification

Conclusion

Characterization of biochar derived from pyrolysis of coconut husk and areca nut husk were investigated. The results disclose that the proximate analysis of the materials were almost similar. A mineralogical study of the coconut husk and areca nut husk biochar by XRD revealed of amorphous mainly carbon, graphite were found. XRD spectra of two biochar peak in figure and there are two diffracted peaks were observed. It could be said that the two biochar were amorphous material with little crystalline. The most important observation was the difference in the number of oxygenated functional groups, on the surface of the two biochar as shown by FTIR analysis of two biochar peak The FTIR spectra of two biochar display the bands containing O-H, C-H, C=O and C-O stretch. Coconut husk biochar and areca nut husk biochar differed in their surface morphology in terms of pore size. These coconut husk biochar image showed heterogeneous distribution of pores and rough texture. Presence of micro-pores and mesoporous detected. The size of the pores indicated that the volatile compounds had evaporated creating macro pores on the surface. This study was carried out to provide basic information as to the possibility of reuse of biochar derived from different biomass of biological waste. According to the characterization of biochar obtained by coconut husk and areca nut husk biomass, we can conclude that such biochar can be suitable for different specific application and may be an effective way for recycling the waste resources.

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