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Characterization of Rubber Sludge-Derived Biochar at Different Pyrolysis Temperatures as A Supplementary Media in Anaerobic Digestion of Food Waste Process

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Anaerobic digestion (AD) is recognized as one of the most sustainable, cost-effective and efficient means to cater food waste (FW) problems. However, the inconsistency of the organic content in the FW increases the instability of the system and results in a significant rate of failure. Thus, a biochar is introduced to the system to aid the process. This study aims to conduct the characterization of biochar derived from rubber sludge to develop a more stable AD system. The raw rubber sludge undergoes a pyrolysis process at temperatures ranging from 350 to 750 °C for 2 hours. The properties of the produced biochar were then thoroughly analyzed using elemental analyzer, SEM-EDX, FTIR, and BET methods. The analysis indicates that the biochar possesses a high specific surface area and high roughness with micropores formed, making it an effective supplementary media. The essential nutrients required for AD system are also present in the biochar. Hence, the incorporation of biochar in AD shows great potential in enhancing the stability and efficiency of the AD system.

Keywords: Biochar, rubber sludge, food waste, anaerobic digestion, biogas

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The increasing production of rubber wastewater is very concerning, especially given Malaysia's growing number of rubber industries each year. Rubber sludge produced from rubber wastewater treatment plants was once thought of as waste, but today it can be a source of a wide range of materials, including organic matter, nutrients, and metals that can be recovered and used again. Massive sewage sludge production over the world necessitates careful management. Therefore, it is very important that the sustainable sewage sludge management shifts to introduce the implementation of a resource recovery approach, rather than only final disposal of sludge [1]. In general, the rubber particles in the sludge are typically fine, sticky, and have a high viscosity and its consistency varies depending on the composition and moisture content of the sludge [2].

The utilization of rubber sludge into resources can be a future trend by turning it into a biochar [3]. Biochar, an amorphous and carbonaceous solid material derived from a thermochemical degradation process in an oxygen-constrained condition called pyrolysis, is gaining global attention due to its favorable characteristics and properties [4-6]. Moreover, the relevance of biochar applications is due to their low-cost and eco-friendly process. Biochar has been extensively explored by researchers as a supplementary media or additive for the anaerobic digestion (AD) process. The role of biochar can be extended to buffer capacity and alkalinity, adsorption of inhibitors as well as a medium for microbial populations[7]. Furthermore, this role is aligned with the characteristics and properties of the feedstocks for the biochar.

Food waste is one of the largest components of municipal solid waste in Asian countries, anticipated to reach 4.3 billion tonnes by 2025 [8]. Generally, food waste has a high organic matter content and biodegradability, making it a good substrate for methane derivation during AD. However, anaerobic digestion of food waste (ADFW) deals with major limitation which is instability of the system due to the existent of inhibitory that led to the system failure [9-11]. To counter this problem, the addition

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of biochar containing carbon-rich material, high porosity, organic functional groups and surface area can enhance the system stability and substrate degradation, reducing or eliminating inhibition during AD [12-15]. Therefore, this study is proposed to elucidate the characteristics of rubber sludge-derived biochar for addition in ADFW process.

EXPERIMENTAL

Fabrication of Biochar

Raw rubber sludge was collected from the effluent treatment tank of Kilang Getah Kg Awah - FGV Holdings Berhad, Chenor, Pahang. Initially, the sludge was sieved to remove contaminants before being dried in a drying oven (Memmert, USA) at 70 ℃ for 2 h. Then, the dried rubber sludge was converted into biochar through pyrolysis in a muffle furnace (Carbolite, USA) at temperature range from 350-750°C with heating rate of 10°C/min and maintained for 2 h. The biochar derived from rubber sludge was cooled to room temperature. Consequently, it was finely ground using pestle and mortar and stored in a desiccator. The fabricated biochar was denoted as BC350, BC450, BC550, BC650 and BC750, respectively.

Characterization of Biochar

The characterization of biochar was conducted through several analysis and testing. Elemental composition analysis, surface morphologies analysis by scanning electron microscope (SEM) with EDX analysis, functional group analysis using fourier transform infrared spectroscopy (FTIR) and surface area using Brunauer-Emmett-Teller (BET) method were performed on the powder-formed biochar to investigate the physical and chemical properties of the sample. Through this thorough analysis, a deeper knowledge about rubber sludge-derived biochar can be explored and nature can be comprehended to better aid AD process.

The elemental composition analysis for the total Carbon (C), hydrogen (H) and nitrogen (N) contents in biochar was measured using elemental analyzer (Elementar, German). The surface morphologies of the biochar were observed using scanning electron microscope, SEM (TM3030 Plus, Hitachi, Japan with Energy Dispersive X-Ray Spectroscopy, EDX analysis (Oxford Instruments, UK) analysis at 20 kV. Functional group changes on prepared biochar were analyzed by Fourier Transform Infrared (FTIR) spectroscopy. The samples were mixed homogenously with dry potassium bromide (KBr) (0.01% w/w) in an agate mortar and a hydraulic press was used to produce composite pellets for analysis. The FTIR spectra was collected in the 400 to 4000 cm^{-1} range. The surface area and pore distribution were analyzed using a BET analyzer (Micrometics, USA) via BET methods.

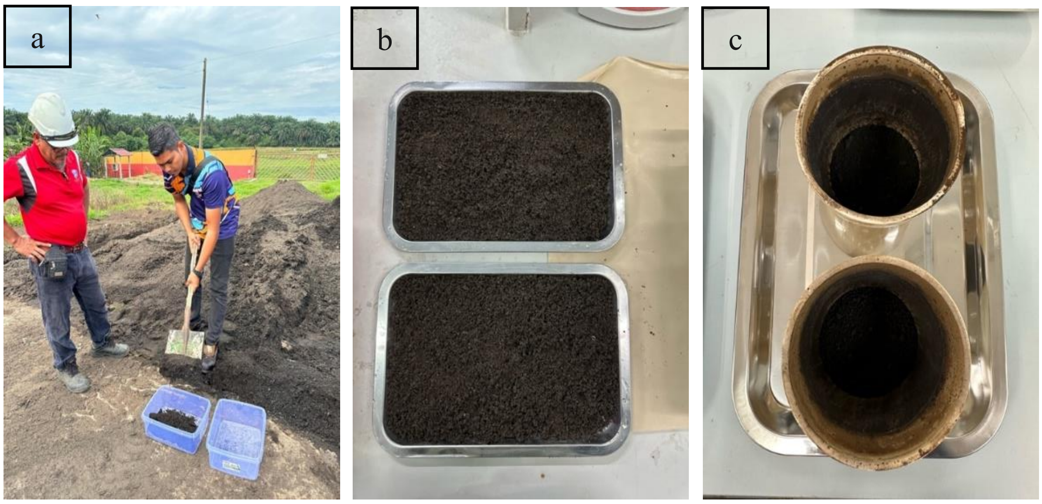


Figure 1. (a) Rubber sludge collection. (b) The sieved rubber sludge. (c) Biochar after pyrolysis.

RESULTS AND DISCUSSION

Elemental Compositions Analysis

Elemental composition analysis was carried out on biochar to quantify the carbon, hydrogen, nitrogen and sulfur content in the biochar. Table 1 provides data obtained from the elemental analysis. There is a decrease in hydrogen and nitrogen content. The observed reduction of non-carbon content may result from the volatility of the organic material. The volatile content such as hydrogen and nitrogen are decomposed and released as gases. In comparison, the carbon enrichment in the biochar can be seen with the increase of pyrolysis temperature. With volatile content released, the carbon retained in the remaining solid increases. In this study, sulfur is considered stable for all pyrolysis temperatures investigated.

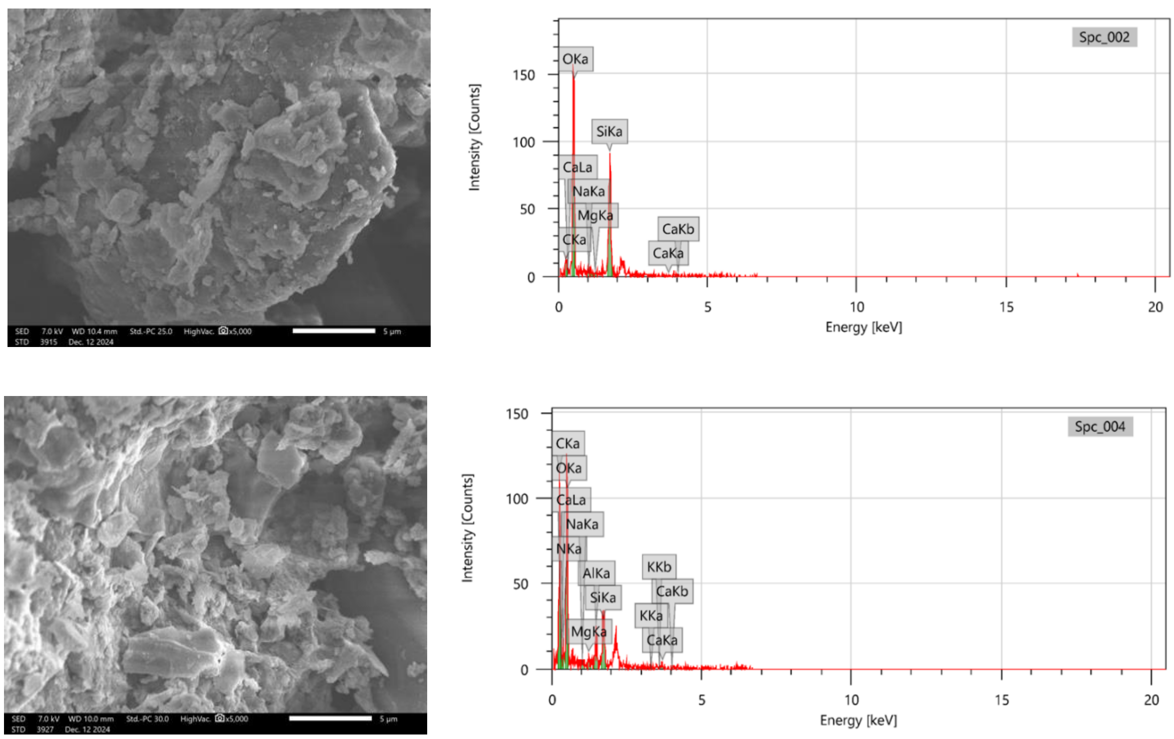
Morphologies Analysis

From the SEM images below (Figure 2), the first three samples (Figure 2a, 2b and 2c) show a low roughness structure with the absence of well-developed pore networks. Lower temperature provides insufficient energy to fully remove the volatile matter resulting in a denser and less porous structure. The mentioned surface characteristics can be seen in Figure 2b and 2c. Due to the incomplete thermal degradation, the uncarbonized organic matter retained in the biochar, thus less voids are formed.

In contrast, higher temperatures will contribute to a highly aromatic, which is attributed to the carbonation degree of organic matter and formation of micropores due to an extensive thermal degradation [16]. High temperature leads to more volatile matters released leaving behind a highly porous structure. The voids and the irregularity of the surface are the favorable characteristics in biochar as it provides better attachment and microhabitat for the microorganism's activities to take place. As seen in Figure 2d, the sample exhibits a visible porosity structure and rough surface in which likely due to the decomposition of hemicellulose and cellulose. The samples in Figure 2e and 2f show higher surface roughness.

Table 1. Elemental compositions of biochar.

Element	Raw rubber sludge	350°C	450°C	550°C	650°C	750°C
Carbon	34.9	31.2	36.4	37.7	27.2	40.1
Hydrogen	4.4	2.4	2.2	1.9	1.2	1.4
Nitrogen	0.5	0.6	0.6	0.5	0.3	0.3
Sulfur	1.1	1.1	1.2	1.2	1.2	1.3



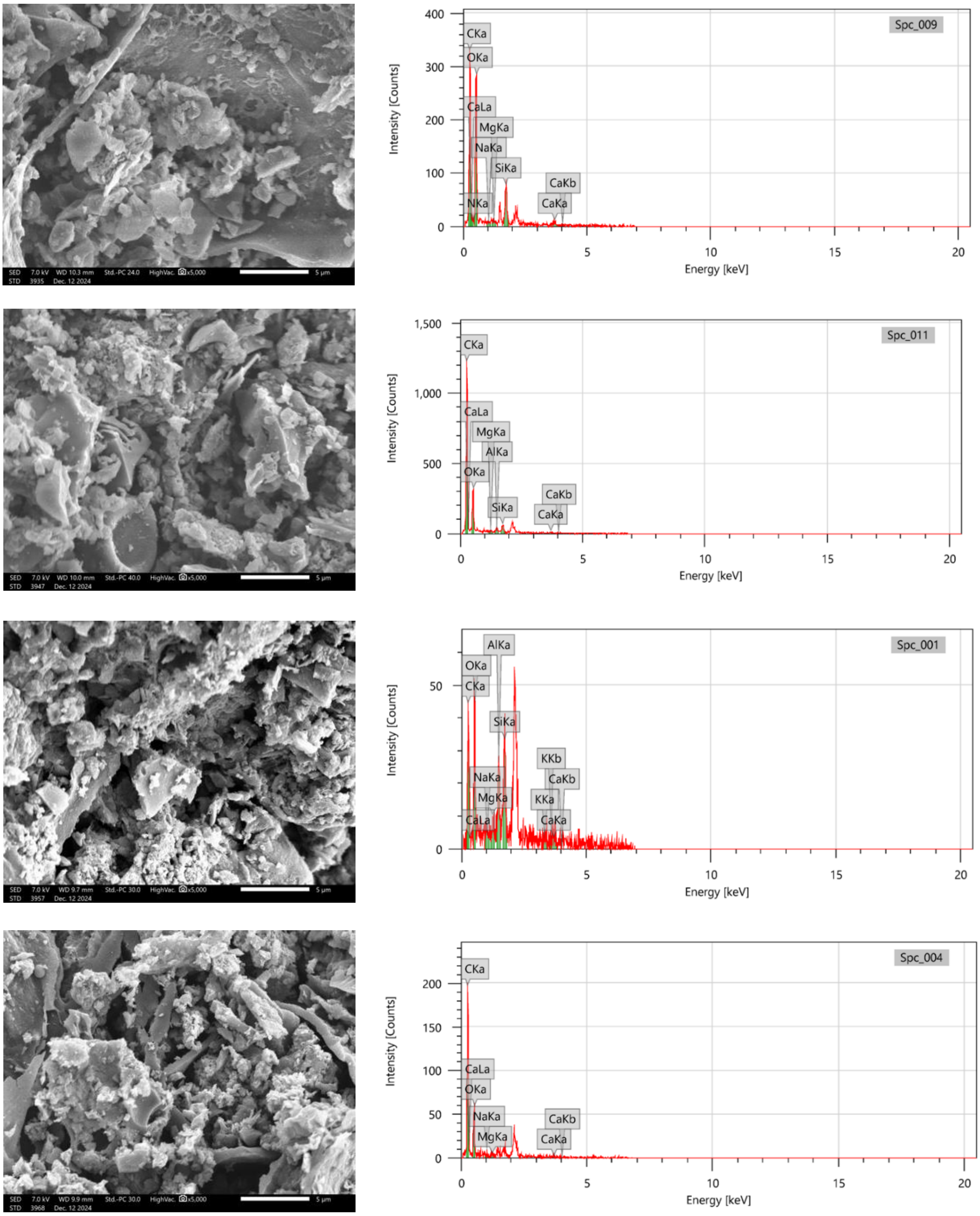


Figure 2. SEM image and EDX spectrum of (a) raw rubber sludge (b) BC350 (c) BC450 (d) BC550 (e) BC650 (f) BC750 at 5000x magnification.

Functional Group Analysis

The functional group of biochar were analyzed by FTIR spectroscopy, and the results are presented in Figure 3. The pyrolysis of feedstock at higher temperature produced more amounts of volatile substances and carbonaceous gases which resulted in lower total acidic functional groups. The absorption peaks at 3293cm^{-1}, 3443cm^{-1}, 3213cm^{-1}, 3385cm^{-1}, 3434cm^{-1}, and 3434 cm^{-1} correspond to the characteristic peaks of O–H for raw rubber sludge, BC350, BC450, BC550, BC650 and BC750 respectively. The band intensity of the O-H stretching is decreased as the temperature increases reflecting in the decrement of moisture content in the samples. Aliphatic C-H stretching was found in raw rubber sludge, BC450 and BC 550. The enhancement of C=C stretching at 1651 cm^{-1}, 1635 cm^{-1}, 1578cm^{-1} and 1561cm^{-1} represents the aromatization process that takes place during the pyrolysis process.

Surface Area Analysis

The surface area and size of the pores of biochar are mainly related to the pyrolysis temperature. These physicochemical properties are crucial in evaluating the porosity and the adsorption capacity of the biochar which directly affect the performance of ADFW. High surface area and larger pores are favourable, allowing for microbial colonization and activities. However, excessively large pores may cause material brittleness and poor strength [17]. The formation of micropores is important as it provides more active site and larger specific surface area [18]. However, the drawbacks of having too small size of pores is that it can hinder the entry of larger microorganisms and reduce the diffusion rate of small molecules. Slightly larger pores can ease the microorganisms and inhibitors penetrating pore channels thus reducing the inhibitions in ADFW [19].

The pores width of all samples displayed in Table 4.1 are considered as mesopores as the width lies within 2-50 nm. Raw rubber sludge, BC350 and BC450 have a relatively low surface area of 2.910, 4.431 and 17.524 m^2/g respectively. As the temperature reaches 550°C, the surface area for BC550, BC650 and BC750 is significantly improved to 137.778, 123.428,234.223 m^2/g similarly to the pore volume from 0.013 to 0.331 cm^3/g when the temperature increased from 350°C to 750°C. The surface area and porosity data collected shows similar trend to the literature which suggests that the findings are caused by the release of vaporous organic material and increase of carbonization temperature [20]Click or tap here to enter text.. Handiso et al., [21] suggests that the huge pores formation is due to the rupture of hemicellulose and many organic matters. Therefore, the surface area of biochar increases with the pyrolysis temperature due to enhanced pore formation and carbonization, peaking typically between 500 and 700 °C [22].

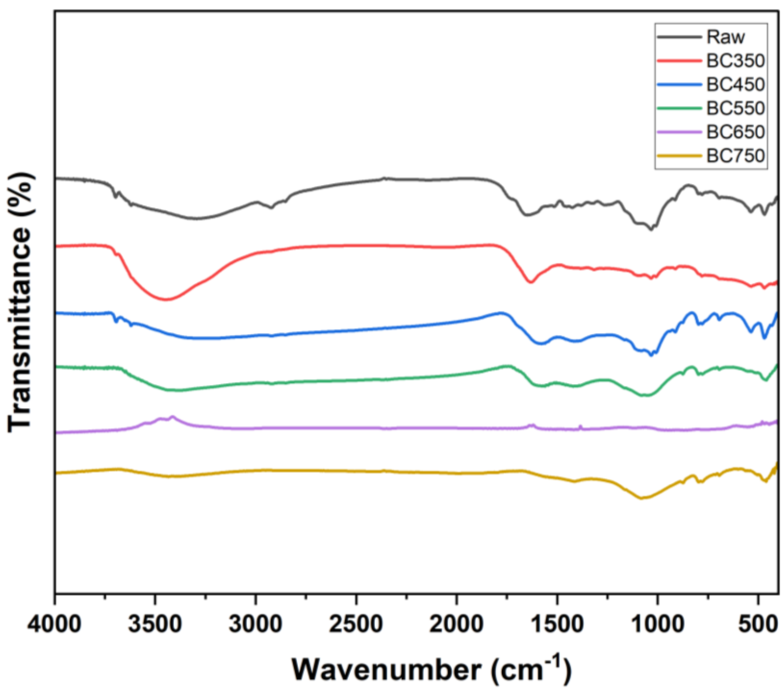


Figure 3. FTIR Spectra for raw rubber sludge and rubber sludge-derived biochar at various pyrolysis temperatures.

Table 2. Surface area, pore volume and Average pore width of the samples.

Sample	BET Surface Area (m^2/g)	Pore Volume (cm^3/g)	Average Pore Width (Å)
Raw	2.910	0.007	76.068
BC350	4.431	0.013	84.821
BC450	17.524	0.028	61.147
BC550	137.778	0.037	44.624
BC650	123.428	0.028	49.617
BC750	234.223	0.331	39.942

CONCLUSION

This study investigated the influence of pyrolysis temperature on the properties of biochar derived from rubber sludge. Biochar yield decreased, while carbon content increased with higher temperatures due to enhanced carbonization. SEM and FTIR analyses showed significant structural and chemical transformations above 500°C, including increased pore development and reduced O–H functional groups. BET results further confirmed higher surface area and pore volume, alongside reduced pore width, at elevated temperatures. Overall, biochar produced at 550°C exhibited the most favourable physicochemical characteristics for enhancing and stabilizing the ADFW process. These findings demonstrate the potential of temperature-tailored biochar to improve anaerobic digestion performance, support sustainable waste management, and contribute to SDG 7 (Affordable and Clean Energy) and SDG 12 (Responsible Consumption and Production).

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