Synthesis and Characterization of Micro-nano Carbon Filler from Jatropha Seeds

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Biochar was synthesized from biomass (jatropha seeds) through a low microwave pyrolysis temperature of 180 °C with microwave power of 2kW. A ball milling process reduced the jatropha seed biochar size and converted it into micro-nano carbon biofiller. After ball milling, the biochar size was reduced from 1 to 3 mm to the 10 µm to 600 nm range, which is around a 90% reduction in size. Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), and Brunauer-Emmett-Teller (BET) analysis were used to determine the jatropha seed biofillers properties with respect to the ball milling processes. BET results revealed increasing surface area from 0.10 to 3.67 m^2/g, and EDS results revealed the elemental composition of the jatropha seed biofillers. The carbon mass percentage increased from 72.6 to 81.2%. Both results were after ball milling for 30 hours. The FTIR results revealed an increase in transmittance intensity and some reduction in peaks after ball milling. Production of micro-nano carbon fillers from microwave pyrolysis jatropha seeds biochar are applicable as reinforcement fillers for high strength composite material fabrications. Scanning electron microscopy, EDS, FTIR, and BET analysis results indicated size reduction of the biochar with increased carbon content from 72.6 to 81.2% as surface area increased from 0.10 to 3.67 m^2/g after 30 hours of ball milling.

Keywords: Format; Synthesis; Characterization; Micro; Nano; Carbon; Jatropha

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INTRODUCTION

Jatropha seeds have gained interest with researchers due to their potential as a source of bio-oil. *Jatropha curcas* L. belongs to the family Euphorbiaceae and is a deciduous shrub that can grow to a height of 3 m to 5 m, with a productive lifespan of 50 years (Ugbogu *et al*. 2014). Srivastava *et al*. (2013) reported numerous case studies showing that jatropha seeds have high oil content, with potential application as an alternative to diesel. Depending on the variety, the seeds may contain 40 to 60% of oil. In recent years, several studies have been conducted examining jatropha seeds for production of bio-oil, measuring their properties and characteristics (Ugbogu *et al*. 2014; Kadry 2015; Audu *et al*. 2018). After extraction of the bio-oil, an alternative method of disposal alongside other organic waste utilizes microwave-assisted pyrolysis to yield biochar, which may be beneficial for numerous applications. There are several studies supporting pyrolysis

of jatropha fruit, which includes the seeds and cakes, to obtain both biochar and syngas (Figueiredo *et al*. 2011; Kanaujia *et al*. 2016; Odetoye *et al*. 2019).

Biochar has many applications, including soil modification in the agricultural sector and adsorption of heavy metals or dyes in industrial sectors. Li *et al*. (2019) reported microwave-pyrolysis biochar of *Artemisia selengensis* and its adsorption capacity for methylene blue, for which biochar pores, surface area, and pyrolysis conditions influenced the adsorption capabilities (Li *et al*. 2019). In a similar study on adsorption capabilities, Bakly *et al*. (2019) reported nitrate removal with macadamia nutshell biochar from slow pyrolysis; more than 45% of nitrate was removed at the highest concentration and at the lowest flow rate. Bakly *et al*. (2019) also attributed the adsorption capabilities to the biochar size, porosity, and surface area; the biochar samples were ground and sieved with a size range of 1.18 mm to 2.30 mm before adsorption testing. Furthermore, agricultural runoff is a major cause of degradation to freshwater sources, so the removal of nitrate content is a concern, due to the abundance of nitrogen-based fertilizers in agriculture (Bakly *et al*. 2019).

In studies of microwave-assisted pyrolysis, some researchers altered the microwave power in their experiment, in comparison to the alteration of temperature. The differences in microwave power led to monitoring the maximum temperature of the biomass, and researchers associated the maximum temperature achieved with biochar and oil yields. Huang *et al*. (2016) stated that the product yields among some studies are relatively different, as some studies have differed in product yield by varying factors such as, biomass characteristics, particle size, sample weight, microwave power, reaction temperature, reaction time, product vapor residence time, reactor design, and microwave heating manner. Among the studies, the reaction temperature appeared to be the main attribute influencing the product yield. A study by Budarin *et al*. (2009) reported the microwave-assisted pyrolysis of wheat straw at a maximum-minimum temperature of 180 °C, achieving product yields with mass percentages of 29%, 20.6%, 36.4%, and 14% for biochar, organics, water, and gas, respectively. With the addition of HCl, H_2SO_4, and NH_3, the researchers achieved greater biochar and gas yields in comparison to the neat wheat straw microwave-assisted pyrolysis sample at the same temperature (Budarin *et al*. 2009).

Some other studies have reported similar percentage product yields under low-temperature microwave-assisted pyrolysis, ranging from 180 °C to 600 °C, varying among the studies (Zhou *et al*. 2013; Zhang *et al*. 2015a, 2015b). According to Zhang *et al*. (2019), the pyrolysis preparation method may influence biochar characteristics such as biochar yield, pore size, surface area, and hydrophobicity; and ball milling is a good, low-cost modification method to increase biochar sorption capabilities and influence the hydrophobicity, by modification into micro-nano biochar powders.

According to a review of the literature, there has been a lack of studies focusing on producing micro-nano carbon fillers from any pyrolysis product. Most researchers only concentrate on the study of oil or biochar from the pyrolysis of biomass, as well as the types of pyrolysis process. Hence affirming the research gap, the aim for this research is to produce micro-nano carbon filler from microwave pyrolysis jatropha seeds biochar, utilizing a ball milling process. The production of micro-nano carbon filler may be implemented as reinforcement filler in applications for high strength composite material fabrications.

In this study, it could be highlighted that jatropha seed undergoes microwave pyrolysis at low pyrolysis temperature of 180 °C with a microwave power of 2 kW. The resulting biochar was ball-milled to decrease size and increase surface area, achieving a

micro-nano size range carbon biofiller. Variations of ball milling durations was investigated to understand when a micro-nano range has been achieved. Scanning electron microscopy (SEM), Energy dispersive X-ray spectroscopy (EDS), Fourier-transform infrared spectroscopy (FTIR), and Brunauer-Emmett-Teller (BET) analysis were used to investigate the characteristics of the ball-milled jatropha seed carbon biofiller, which includes changes in size, surface area, and composition.

EXPERIMENTAL

Materials

The jatropha seeds were supplied by Universiti Malaysia Sarawak (UNIMAS) (Kota Samarahan, Sarawak, Malaysia) and were air-dried prior to storage for subsequent use. A Kholler CT114A Pyrolyser 5010 (Kuala Lumpur, Malaysia) was used for the microwave pyrolysis process. A Retsch PM400 Planetary Ball Mill (Retsch GmbH, Haan, Germany) was used for all the ball milling processes. A Panasonic MX-AC400 Mixer Grinder (Panasonic Corporation, Kadoma, Japan) was used before the ball milling process as prerequisite to assist the ball milling process. A Fourier-transform infrared spectrophotometer (IRAffinity-1, Shimadzu Corporation, Kyoto, Japan) was used for the FTIR analysis of the dried jatropha seeds, biochar, and biofillers for comparison. A Hitachi TM4000Plus tabletop microscope with a Quantax75 TM series energy dispersive X-ray spectrometer (Hitachi, Ltd., Tokyo, Japan) was used for the SEM and EDS analyses of the jatropha seed biochar and biofillers to investigate the composition, surface structure, and size. A Quantachrome Autosorb iQ (Quantachrome Instruments, Boynton Beach, FL, USA) was used for the BET surface area analysis of the Jatropha seed biochar and biofillers. All experiments were conducted in the analytical chemistry lab of the Department of Chemical Engineering, UNIMAS.

Methods

Microwave pyrolysis of jatropha seeds

First, 100 g of slightly ground jatropha seeds was placed into a borosilicate beaker and into the microwave reactor chamber. The seeds were ground slightly to accommodate the beaker size. Prior to the microwave pyrolysis process, the microwave reactor chamber was purged using a vacuum pump to create an oxygen-free environment. The pyrolysis process was performed for 10 min for 3 rounds to 4 rounds under the following operating conditions: a microwave power of 2 kW, pyrolysis temperature set at 180 °C, a nitrogen flow rate of 0.5 L/min, and a chamber vacuum pressure of 0.8 kPa. After each round, the Jatropha seeds were stirred manually for even heat distribution, as the microwave reactor did not have a motorized turntable. This eliminated the variable hotspots during the microwave pyrolysis process. After completing microwave pyrolysis, the biochar and the condensed residual oil were collected.

Ball milling of biochar into biofiller

Before ball milling, the biochar was ground for a total of 5 min at the highest speed (approximately 18,000 to 20,000 RPM) with the mixer grinder. This was performed to ensure an even biochar powder to assist the ball milling process, according to the planetary ball mill specifications. Two cylindrical 25-mL vessels, each with 20 stainless steel balls weighing 0.5 g, was used during the ball milling process, in which biochar was added up

to 60% of the volume of each vessel. Two vessels were utilized to efficiently process more biochar into micro-nano biofiller. The biochar was ball milled for a total of 30 h, while samples were taken for testing at 4 h, 8 h, 12 h, 16 h, 20 h, 25 h, and 30 h.

Characterization and Testing

SEM and EDS analyses of biochar and biofiller

Scanning electron microscopy for the samples was conducted according ASTM E2015-04 (2014), in regard to electron microscopy testing procedure. Magnifications of 1000× were utilized to observe numerous jatropha seed biochar and biofiller samples. In addition, EDS was conducted according to ASTM E1508-12a (2019); three points were selected at random areas of the samples. The software automated the analysis of the elemental composition percentages. The EDS was repeated numerous times for each sample, and the most representative results were selected.

FTIR of biochar and biofiller

Fourier-transform infrared spectroscopy was conducted according to the ASTM E168-16 (2016) and ASTM E1252-98 (2013) standards for qualitative and quantitative analysis. The spectrum scanning was conducted in the wavenumber range of 4000 to 400 cm^{-1} for each sample. Fourier-transform infrared spectroscopy utilized the infrared spectrum transmittance and absorption of the samples to develop a unique molecular fingerprint spectrum. The test was repeated numerous times for each sample, and the most representative results were selected.

BET surface area of biochar and biofiller

The BET surface area analysis was conducted according to the ASTM D6556-14 (2014) standard. The degassing temperature was set at 130 °C for 3 h. Lab technicians at UNIMAS assisted in conducting the experiment. The test was repeated numerous times for each sample, and the most representative results were selected.

RESULTS AND DISCUSSION

FTIR

The FTIR results for the dried jatropha seeds, jatropha seed biochar before ball milling (BBM), and jatropha seed biochar after 30 h of ball milling (ABM30hours) are shown in Fig. 1.

As shown in Fig. 1, BBM and ABM30hours showed increased transmittance intensities in comparison to dried jatropha seeds. This result could be due to the development of pores and internal structure of the biochar after microwave pyrolysis and ball milling (Cheng and Li 2018). In the region from 3800 to 3100 cm^{-1}, the peak bands could be characterized as O–H stretching and H-bonds consisting of alcohols, phenols, and water. The peaks detected for dried jatropha seeds and BBM were broader and deeper in this region compared to ABM30hours, indicating a reduction of alcohols, phenols, and water (Jouiad *et al*. 2014). Ball milling process generates heat due to high impact energy and friction, hence allowing the release of moisture. In which explains the reduction of alcohols, phenols, and water for ABM30hours.

Double peak bands in the region from 3000 cm^{-1} to 2800 cm^{-1} could be characterized as C–H stretching, consisting of alkanes. Peaks detected for ABM30hours

were slightly broader and shorten in this region compared to BBM, which could be associated with the size reduction and increased surface area after ball milling processes, affecting the presence of the biochar surface functional groups (Zhang *et al*. 2019).

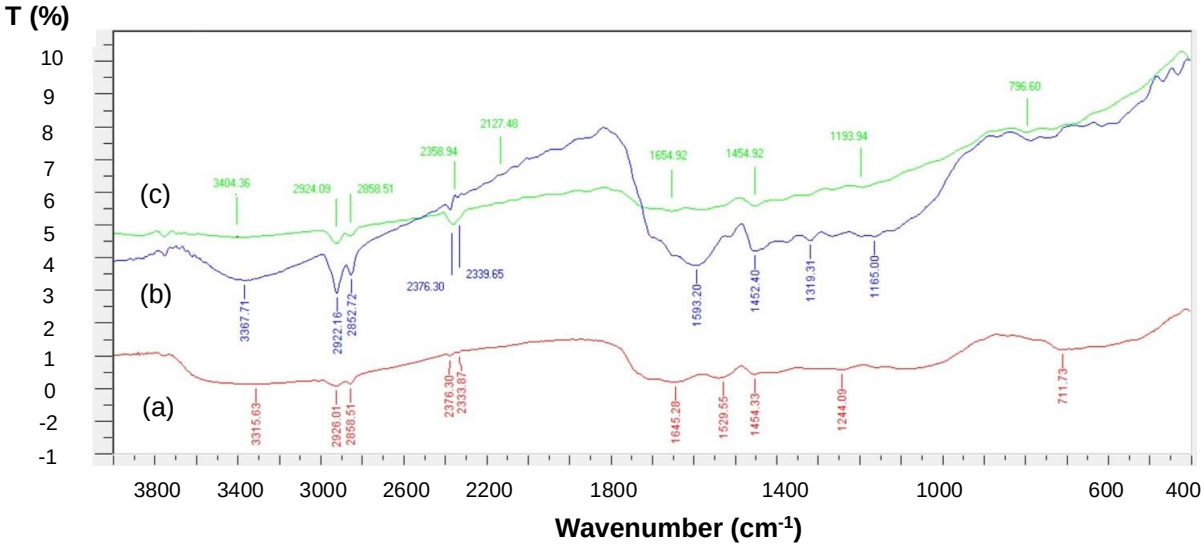

Fig. 1. FTIR results for (a) dried jatropha seeds, (b) BBM, and (c) ABM30hours

In the region from 2400 cm^{-1} to 2200 cm^{-1}, peaks for both BBM and ABM30hours could be characterized as O=C=O stretching (carbon dioxide, CO_2). As shown in the spectrum, there was a slight shift in the peaks and transmittance intensity for ABM30hours compared to BMM, indicating a lower presence of CO_2. The ball milling process decrease size & increase surface area of the biochar may cause the slight shift in peaks indicating a lower presence in CO_2. Similar effects could be seen in one study which chemically pretreats biochar and diminishing peaks of CO_2 caused by pyrolysis process (Meri *et al*. 2018). Hence, ball milling may have similar effects as some chemical pretreatments on biochars.

In the region from 1600 cm^{-1} to 1500 cm^{-1}, peak bands detected for ABM30hours and BBM could be characterized as alkenes' –C=C– stretches. As shown in the spectrum, ABM30hours had stronger peaks compared to BBM, with a slight decrease in transmittance intensity. This result indicates some degree of biochar activation due to increased surface area, as was the case for the region from 3000 cm^{-1} to 2800 cm^{-1} (Tongpoothorn *et al*. 2011).

EDS of Biochar

Table 1 shows the EDS element contents for BBM. Figure 2 shows the spectrum graph in correlation with the data in Table 1. Elements present in the sample, according to Table 1, were carbon (C), oxygen (O), potassium (K), magnesium (Mg), phosphorus (P), calcium (Ca), aluminum (Al), and platinum (Pt).

Table 2 shows the EDS element content for ABM30hours. Figure 3 shows the spectrum graph in correlation with the data in Table 2. Elements present in the sample, according to Table 2, were carbon (C), oxygen (O), potassium (K), magnesium (Mg), phosphorus (P), calcium (Ca), and iodine (I), which could be negligible, as the mass percentage detected was zero.

Table 1. EDS Element Composition for BBM

Element	Atomic No.	Mass (%)	Atom (%)	Absolute Error (%) (1 sigma)	Relative Error (%) (1 sigma)
C	6	72.57	80.39	7.97	10.98
O	8	19.93	16.58	2.54	12.72
K	19	2.36	0.80	0.10	4.23
Mg	12	1.49	0.82	0.11	7.10
P	15	1.44	0.62	0.08	5.66
Ca	20	1.32	0.44	0.07	5.13
Al	13	0.70	0.35	0.06	8.47
Pt	78	0.18	0.01	0.03	19.21

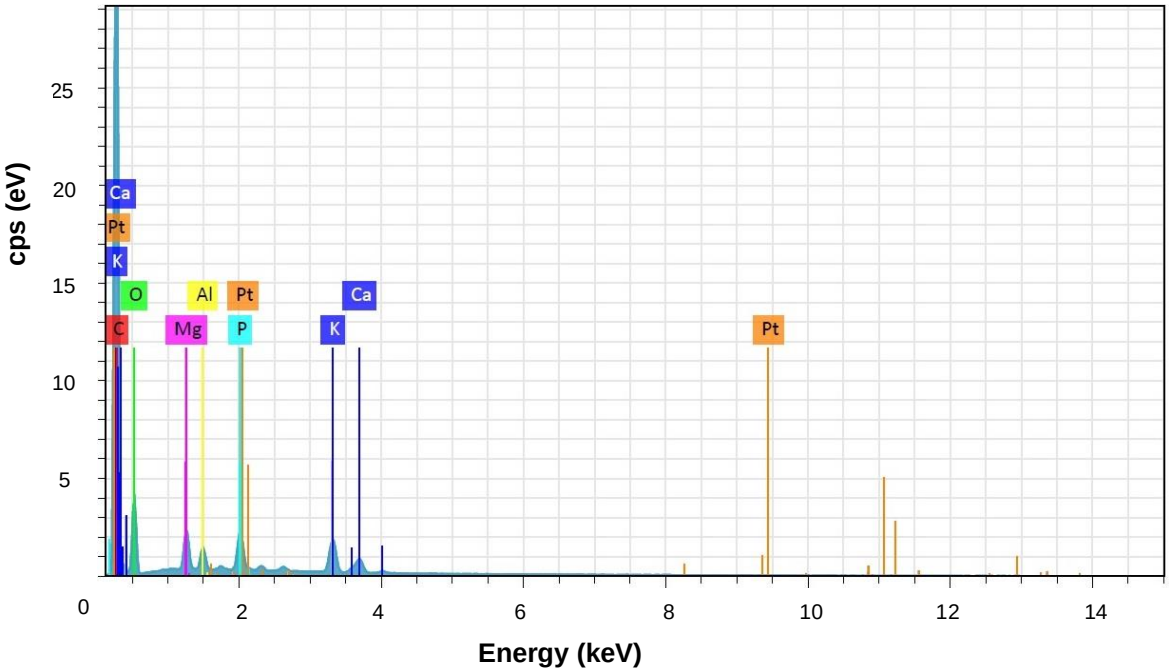

Fig. 2. EDS spectrum graph for Jatropha seed biochar before ball milling

Table 2. EDS Element Composition for ABM30hours

Element	Atomic No.	Mass (%)	Atom (%)	Absolute Error (%) (1 sigma)	Relative Error (%) (1 sigma)
C	6	81.17	86.19	9.29	11.45
O	8	15.97	12.73	2.47	15.49
K	19	1.33	0.43	0.07	5.52
Mg	12	0.53	0.28	0.06	11.11
Ca	20	0.53	0.17	0.05	9.25
P	15	0.47	0.19	0.05	10.25
I	53	0	0	0	0.90

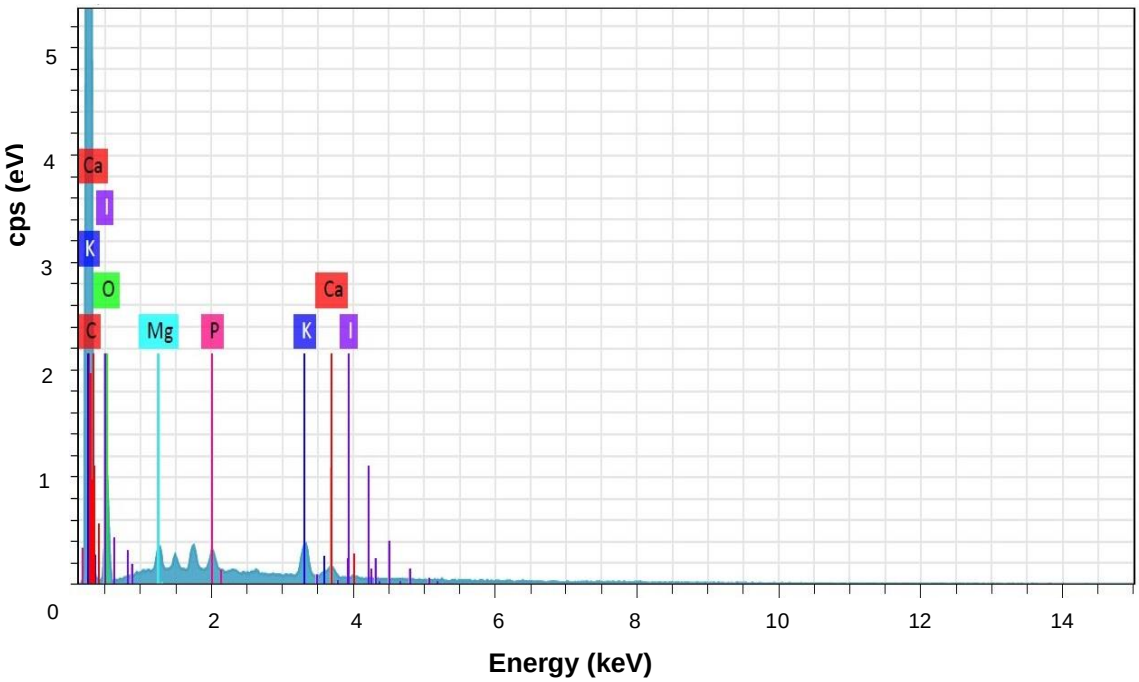

Fig. 3. EDS spectrum graph for ABM30hours

In total, there were eight elements potentially detected in the jatropha seed biochar samples according to the EDS results. The eight elements detected were carbon (C), oxygen (O), potassium (K), magnesium (Mg), phosphorus (P), calcium (Ca), aluminium (Al), and platinum (Pt).

Comparing Tables 1 and 2, the carbon mass percentage of the jatropha seed biochar increased, while the other remaining elements decreased in mass percentage. The changes in the mass percentages of the elemental components could be due to the effects of ball milling conditions, *i.e.*, milling time and ball-sample ratio. One study illustrated similar results, where the changes in mass percentage occurred with altering milling conditions (Supriyono *et al*. 2018). It was noted that carbon was also the most dominant element presence in their study, and the reduced particle size did increase carbon mass percentage indicated by their EDS experimental result. The results of work by Supriyono *et al.* (2018) showed the smallest biochar particle size to be 274 nm, with a carbon mass percentage of 93.0%.

Carbon, oxygen, potassium, magnesium, calcium, and phosphorous were the common elements present in the samples, with carbon having the greatest mass percentage. Moreover, the results listed in Tables 1 and 2 show that the carbon mass percentage increased from 72.6 to 81.2% for ABM30hours. One of the major correlates of carbon content in biochar is the method of production of the biochar, *i.e*. pyrolysis, carbonization, and processing time and temperature (Kim *et al*. 2012; Kloss *et al*. 2012; Al-Wabel *et al*. 2013). The varied elemental compositions could also be associated with the consistency of the EDS machine to detect and characterize elements present in the sample. Carbon content could possibly have been presentable during the EDS experiment due to the distribution of particles caused by size reduction and increased surface area, due to the ball milling processes (Zhang *et al*. 2019).

SEM of Biochar

Overall, the results obtained from SEM showed that the jatropha seed biochar progressively decreased in size after an extended duration of ball milling, totalling up to 30 h of ball milling.

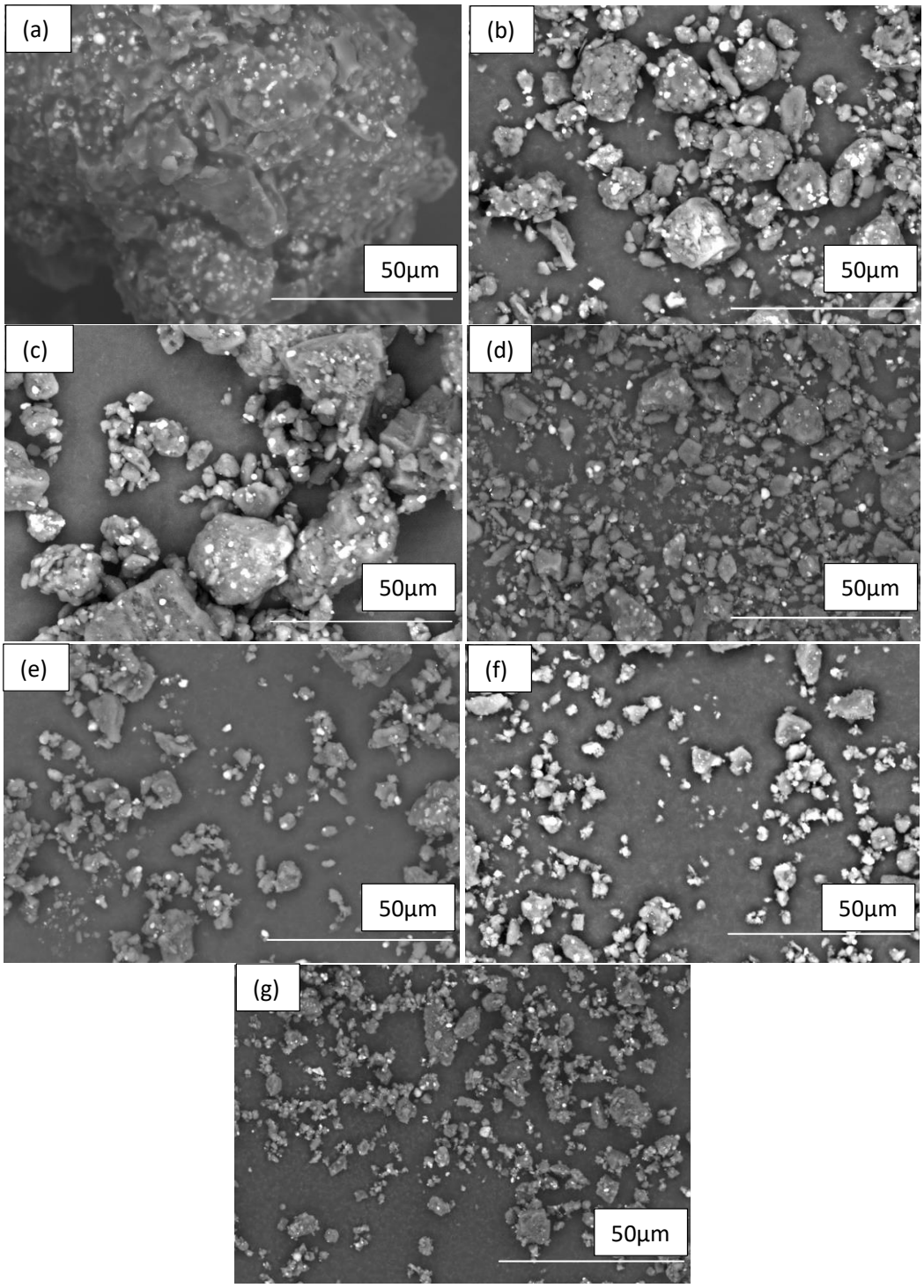

Fig. 4. SEM images at 1000× magnification for jatropha seed biochar: (a) BBM, (b) ABM4hours, (c) ABM8hours, (d) ABM12hours, (e) ABM16hours, (f) ABM25hours, and (g) ABM30hours

The jatropha seed biochar sample in Fig. 4(a) shows millimeter-size chunks (1-3 mm) with a rough surface structure, in which numerous macropores were present. The samples shown in Fig. 4(b) and 4(c) may look similar in size, but both exhibited notable size reductions after ball milling, compared to the sample in Fig. 4(a). As the duration of ball milling approached and exceeded 12 h, Fig. 4(d), 4(e), and 4(f) show that the jatropha seed biochar samples were further reduced in size and developed characteristic agglomerates comprising finer jatropha seed biochar particles attached onto the larger pieces (Tsai *et al*. 2019).

A great reduction in size overall is shown in Fig. 4(g) for ABM30hours, where agglomeration characteristics and a finer chip-like branches consistency of the biochar were more present (Wang *et al*. 2019; Zhang *et al*. 2019).

For ABM30hours, the jatropha seed biochar particles were observed to be in the micro-size range between 1 µm to 10 µm, and 400 to 600 nm noticeably branching together with the micro size particles. The agglomeration characteristics of the jatropha seed biochar may be attributed to the biochar not reaching the grinding limit. Overall, after ball milling, jatropha seed biochar size were reduced from 1 to 3 mm to the 10 µm to 600 nm range, which represents a reduction in size of around 90%. By altering ball milling conditions, *i.e*., increasing milling time and ball-sample ratio, agglomeration could be prevented, and the grinding limit can be reached. A grinding limit is the point where the duration/cycles of ball milling will no longer affect the particle size of the sample, and the sample is homogenous (Eckert and Börner 1997; Umemoto *et al*. 2001). Impact energy during ball milling may be affected, in correlation to altering ball the milling conditions (Supriyono *et al*. 2018).

BET Analysis of Biochar

Overall, the BET results indicated an increase in surface area after ball milling (Table 3). The smallest surface area was observed for BBM, at 0.10 m^2/g. The greatest surface area was observed for ABM30hours, at 3.67 m^2/g. This was a great increase in surface area, supporting the SEM results, which illustrated that the jatropha seed biochar particles had reduced to micro-nano size. The overall surface area was considerably smaller in comparison to other studies of jatropha seed biochar (Tongpoothorn *et al*. 2011; Garg and Das 2018; Zhang *et al*. 2019). Nevertheless, ball milling did increase the surface area by reducing the biochar from millimeter-size pieces into fine powders within the micro-nano size range. By contrast, the other studies were conducted within a macro size range. The type of pyrolysis process and the specific conditions (such as temperature, nitrogen gas flow rate, and time) may influence the initial surface area of the biochar before ball milling (Zhou *et al*. 2013; Zhang *et al*. 2015b; Zuo *et al.* 2018; Zhang *et al*. 2019). One study states that the pyrolysis temperature, among other conditions, has a large correlation to surface area and pore size due to the release of more volatiles from the biomass (Cheng and Li 2018). This explains the small surface area of the jatropha seed biochar samples, which may still contain some oils or volatiles from the microwave pyrolysis process. The increased surface areas of the biochar samples are established by the multi-point and isotherm BET plots presented in Figs. 5 to 7. According to Fig. 5, as the ball milling time increased, the volume at STP (cc/g) also increased, demonstrating the increased surface area of the jatropha seed biochar. Similarly, Figs. 6 and 7 show the increasing adsorption and desorption of the biochar samples with increasing duration of ball milling. The data presented also relates with the volume at STP (cc/g), further supporting the increased surface area after ball milling. A literature indicates there is a relation between the BET

isotherm plot with adsorption characteristic of Pb (II) using biochar. Isotherm plots could be used to detected adsorption capabilities of the biochar, and the literature best describe such adsorption behavior of Pb (II) using biochar to be a monolayer adsorption (Wu, Q *et.al* 2019). In relation to this study, BET isotherm plots showed increased adsorption capabilities of the biochar with increased ball milling duration (*i.e.* particle size reduction and increased surface area).

The biochar preparation methods could influence the biochar yield, carbon content, pore size, and initial surface area prior to the ball milling process. Future investigation on optimizing preparation variables could yield biochar with greater carbon content, porosity, and surface area. The preparation variables include; methods to produce the biochar (*i.e.* pyrolysis, carbonization), production conditions (*i.e.* temperature, time, pressure, and gas flow rate), and ball-ratio for ball milling processes.

Referring to Figs. 5, 6, and 7, P_0 is the gas saturation pressure, P/P_0 is the pressure of the adsorbate relative to its saturation pressure, known as the relative pressure, and P is the partial saturation pressure of the adsorptive gas in equilibrium with the surface.

Table 3. BET Results for All Jatropha Seed Biochar Samples (Surface Area)

Jatropha Seed Biochar Sample	Surface Area (m^2/g)
Before ball milling (0 h)	0.10
Ball milling (4 h)	1.14
Ball milling (8 h)	2.16
Ball milling (12 h)	2.51
Ball milling (16 h)	3.21
Ball milling (25 h)	3.51
Ball milling (30 h)	3.67

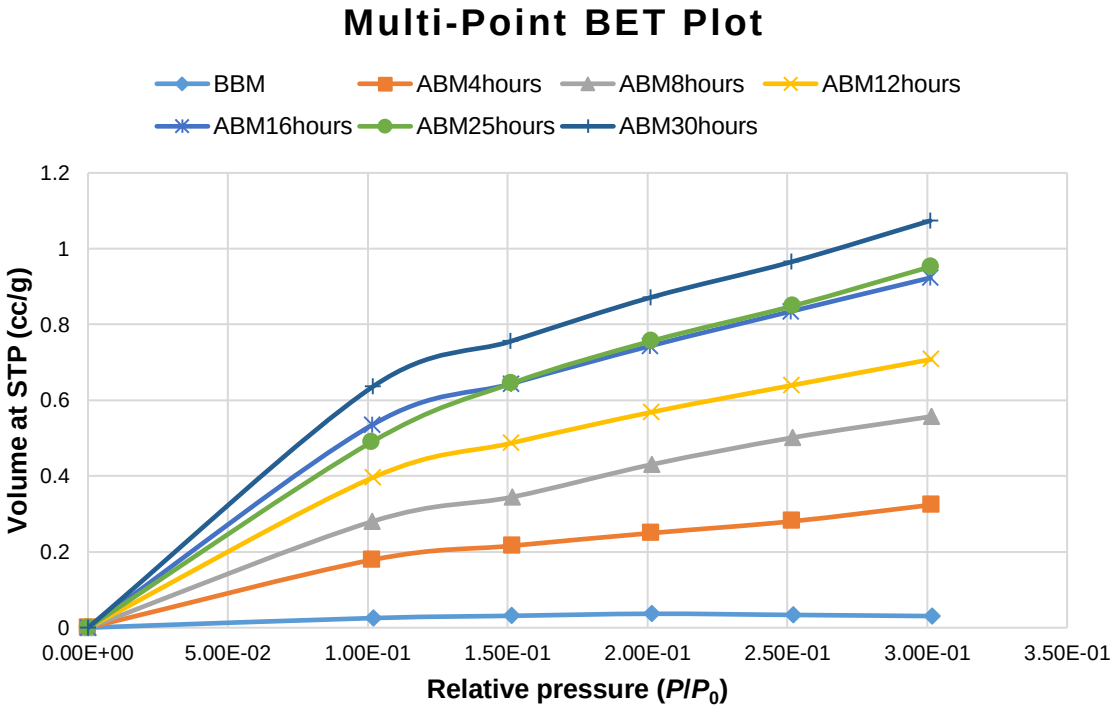

Fig. 5. Multi-point BET plot for all Jatropha seed biochar samples

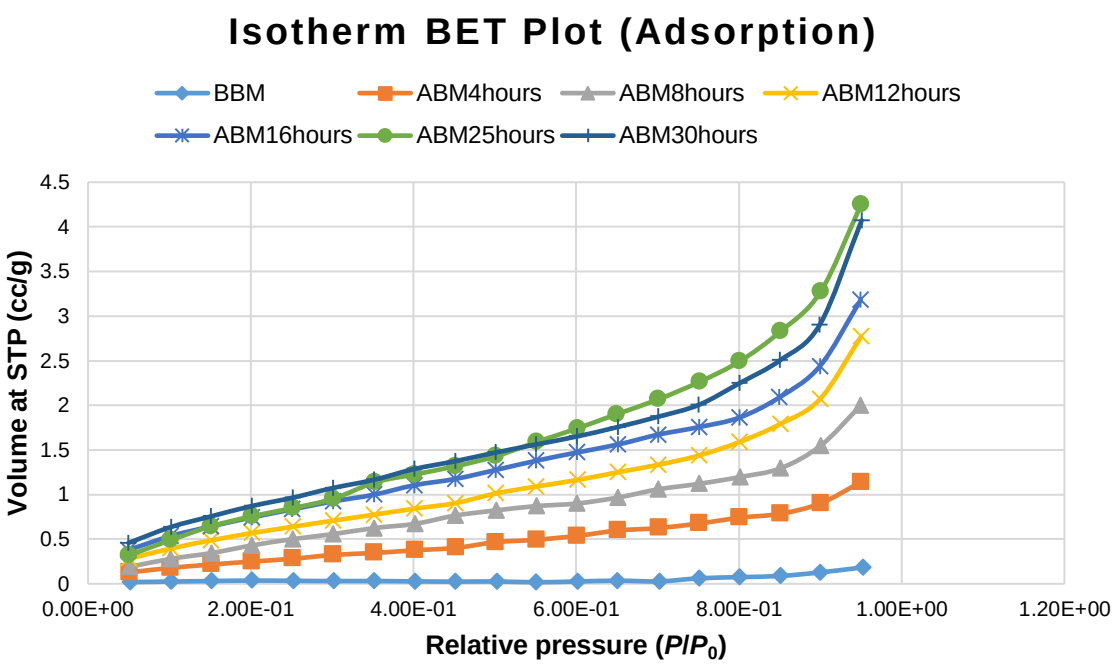

Fig. 6. Isotherm BET plot (adsorption) for all Jatropha seed biochar samples

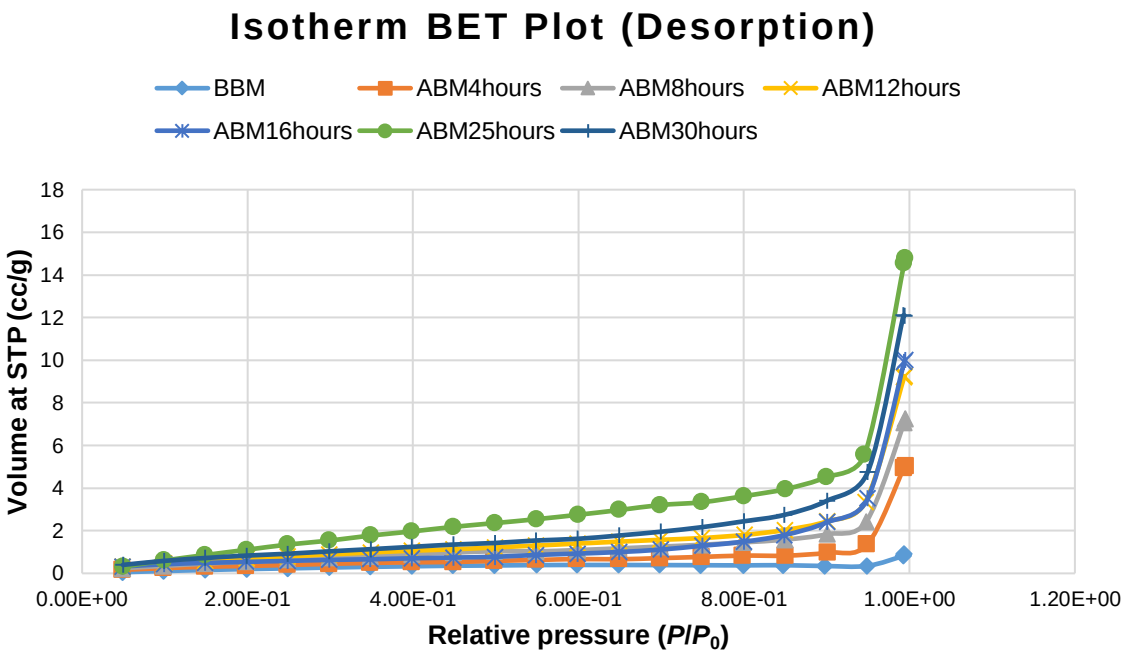

Fig. 7. Isotherm BET plot (desorption) for all Jatropha seed biochar samples

CONCLUSIONS

1. After ball milling for 30 h, SEM imagery indicated that the jatropha seed biochar samples were successfully reduced in size from 1 to 3 mm to the range 10 µm to 600 nm within micro-nano range, hence a 90% reduction in size. The samples have not reached the grinding limit, even after 30 hours of ball milling.
2. EDS and BET analysis results respectively indicate an increased carbon mass percentage from 72.6 to 81.2% with surface area increased from 0.10 to 3.67 m^2/g after 30 hours of ball milling.
3. FTIR shows increased transmittance after ball milling for 30 hours, reduction of the presence of moisture (alcohol, phenol, and water), with similar effects in surface functional groups peaks compared to chemical pretreated biochar by other literatures.
4. Resulting micro-nano carbon filler could be applicable as reinforcement filler in fabricating high strength composites due to the increased surface area, functional groups, and morphological structure. Characteristics and properties of the micro-nano carbon filler may modify mechanical, chemical, thermal, and electrical properties of the polymer matrix.

FUTURE WORK

Future recommendation includes; optimizing biochar yield with higher surface area by investigating various conditions of microwave pyrolysis, optimizing ball-ratio for ball milling processes may effectively achieve micro-nano size in less duration of time, and investigating the grinding limit of the microwave pyrolysis jatropha seed biochar.

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