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PRODUCTION AND PHYSICOCHEMICAL EVALUATION OF ULTRAFINE BAMBOO BIOCHAR PROCESSED USING FRICTION GRINDER SUPERMASS COLLOIDER

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Friction grinding technology has not been employed in refining biochar. In the current study, ultrafine biochar was produced from a native Philippine bamboo species, *Bambusa blumeana,* using a friction grinder supermass colloider. Pyrolysed bamboo was granulated, then refined with the friction grinder super mass colloider using various combinations of grinder clearance and number of passes. The properties such as pH, electrical conductivity and adsorption efficiency of the material were tested. The friction grinder effectively reduced the bamboo biochar to powder with less than 1 μm particle size and a size distribution of 0.48–3.07 μm after 7 passes and a negative 10 clearance setting. A basic pH ranging from 9.55 to 9.98 and electrical conductivity ranging from 11.58 to14.3 S m^{-1} were obtained. Both properties were unaffected by the conditions used for grinding. Adsorption of methylene blue at room temperature by the bamboo biochar was enhanced up to 79.8% with size reduction. Scanning electron microscopy imaging revealed that this was due to the increase in surface area and exposure of the pore structure of the refined bamboo biochar.

Keywords: Adsorption efficiency, *Bambusa blumeana,* biochar, electrical conductivity, friction grinder supermass colloider

INTRODUCTION

It is estimated that 2.5 billion people across the globe rely on bamboo for daily use and economic exchange (INBAR 1999). In Asian countries such as the Philippines, people engage in the cultivation, extraction, utilisation, and commercialisation of bamboo (Razal and Palijon 2009). Within the period 2008–2017, bamboo poles at 417,000 kg were exported from the Philippines, with an estimated value of US$ 272,000 (FMB 2017). Bamboo also contributes to the local market in terms of raw bamboo and bamboo products sold or exchanged, influenced by the rising demand in agriculture, construction, furniture and handicraft making industries (Razal and Palijon 2009). However, despite the emerging markets for bamboo and bamboo-based products, stakeholders, especially the local farmers and forest dwellers who rely on the material, remain poor and unable to cope with the increasing prices of goods.

Increasing use of bamboo-based products will boost the demand for this renewable resource. This will positively influence the value of bamboo, which could help address the economic challenges faced by local farmers. Bamboo biochar is beginning to be widely used in various applications such as absorbents, water purifier, humidity regulator, as supplier of negative charges, soil amendment and others (Zhao et al. 2008, Meng et al. 2018). Bamboo biochar has the added advantage of avoiding problems with contaminants and toxic anthropogenic nanomaterials during production and disposal (Tang et al. 2015, She et al. 2016, Oleszczuk et al. 2016). The bamboo feedstock is also abundant, renewable and can be outsourced from industrial production wastes. In addition, biochar can be used as fuel. It is eco-friendly since plant sources sequester carbon while the process of making biochar produces recoverable biofuels and syngas as by-products (Tang et al. 2015, Li et al. 2016).

In recent years, bamboo biochar utilisation focused on size refinement to develop products with enhanced properties for different applications e.g., absorbents, super magnets and conductors (Khan et al. 2015, Li et al.

2016, Naghdi et al. 2019). Bamboo biochar was shown to have improved its adsorption and surface energy quality when the particle size was reduced to the smallest scale possible (She et al. 2016, Naghdi et al. 2017, Qiu et al. 2017). The refinement of biochar relies mainly on mechanical processing, where the action of force (e.g., load or friction) under controllable conditions, reduces the biochar into ultrafine size. Ball milling is commonly used, and by far, the only method that was optimised by characterising the milled products under varying conditions (Naghdi et al. 2017, Lyu et al. 2018). The method was shown to be applicable in producing highly porous granular biochar particles (Peterson et al. 2012, Lyu et al. 2018). Although it has been previously reported, the ultrasonic vibrator is rarely used in producing refined biochar (Oleszczuk et al. 2016). These techniques require the refined biochar to undergo several pre- and post-treatment processes such as sieving and sonication before obtaining ground materials in the microsize range.

Refining with a friction grinder super mass colloider is a technology that has emerged to be useful for several applications. The mechanism of action involves a rotating ceramic grinder where the gap clearance narrows between the rotor and stator as the material undergoes refining. The rotational friction between the discs and the high impact grinding action disintegrates large particles and defibrillates fibrous materials (Osong et al. 2016). Using this technology for size reduction, post-processing treatment can be minimised or avoided. Pioneering studies with ultra-fine friction grinder produced microfibrillated materials from natural fibres (Taniguchi and Okamura 1998). Later, other studies used the ultra-fine friction grinder in a variety of applications such as pulp and soybean (Osong et al. 2016, Sidhu & Singh 2016). Until the present study, the friction grinder has not been employed in refining bamboo biochar. This research, therefore, evaluated the usefulness of friction grinding with supermass colloider in reducing the size of bamboo biochar and determined the changes that occurred in the properties of the material.

MATERIALS AND METHODS

Bamboo biochar preparation

Mature culms of *Bambusa blumeana* were cut into strips with dimensions of 150 cm (length) × 8 cm (width) and were sun dried until 10–15% moisture content (MC) was obtained. The bamboo culm strips were piled vertically inside a fabricated drum kiln pyrolyser at the Forest Products Research and Development Institute (FPRDI), College, Laguna Philippines. The walls of the drum kiln had bored holes along its height for air regulation. The operation proceeded as a batch-type slow pyrolysis reaction where the bamboo feedstock itself provided the heat energy. The process was initiated through partial ignition at the base of the kiln. Oxygen flow was slowly controlled by sequentially blocking the holes at the walls with metal pins, which also prevented heat loss. A thermocouple was attached to the system in order to monitor changes in temperature. Once the set maximum pyrolysis temperature reached 500 °C, it was maintained for 5 hours and then the reaction was stopped by plugging all the holes. The system was allowed to cool down and the bamboo biochar was recovered the following day, separating the solid char from the ash, the unreacted or incompletely charred bamboo, and other debris. A bamboo biochar yield of 32%, based on the initial mass of the bamboo feedstock, was obtained. Proximate analysis of the resulting biochar was carried out according to Singh et al. (2017), which was in accordance with ASTM D1762-84 to determine the proportion of carbon, volatile matter and ash. The same proximate analysis method was used on biochar from palm kernel shell, bamboo and rice husk (Ma et al. 2017, Zhang et al. 2017).

Friction grinding

Bamboo strip biochar was granulated using a rotary crusher, producing as small as 5 mm biochar granules. Then 5 L of deionised water (DIW) was added to 100 g of the granulated biochar for a 2% consistency. This mixture was subjected to grinding using a friction grinder super mass colloider. Preliminary trials showed that negative 10 clearance and 2000 rpm were the optimum grinder setting for refining bamboo biochar (BBC). Bringing the gap to a narrower clearance setting will lead to contamination of the BBC with debris from the grinder. The experiment was carried out by testing several combinations of number of passes with grinder clearance (np:gc) setting in the friction grinder. One complete pass pertains to one cycle of feeding the bamboo biochar feedstock into the friction grinder, the subsequent grinding action in the machine, and then recovery of the ground bamboo biochar. Grinder clearance is the machine setting that determines the distance between the upper and lower ceramic grinder stones that perform the grinding action, which in most cases eventually converts the feedstock into pastelike material. The different number of passes used were 3, 5 and 7. The maximum number of passes selected was 7, as exceeding this number was considered to consume too much time and energy. Each pass was combined with five grinder clearance settings of -2, -4, -6, -8 and -10 for a total of 15 different combinations of number of passes and grinder clearance settings. Thus, for the 3 passes, the combinations of number of passes and grinder clearance settings were designated as (3:-2), (3:-4), (3:-6), (3:-8) and (3:-10) and so on for the other number of passes. A more negative clearance setting would result in too much friction between the ceramic grinders. Differences between the treatments were statistically analysed in a factorial design experiment where average particle size was the dependent variable.

Morphological characterisation

Bamboo biochar samples for characterisation were prepared by placing a small portion in a silicon plate and drying in a vacuum oven for 4 hours. The physical structure, surface features, and size of bamboo biochar were elucidated using a 600i field emission scanning electron microscope (FESEM). Using SEM images (at 4000x magnification) of the ultrafine biochar produced from all grinding combinations of clearance setting and number of passes in the friction grinder supermass colloider, the size of the biochar granules wes determined, with each granule being measured on its longest side using the freeware IMAGE J processing program.

pH and conductivity

Five grams of air-dried, mechanically refined biochar samples were separately added to 50 ml deionised water and stirred with a magnetic stirrer for 1 hour. The suspension was allowed to stand for 30 minutes. The pH and conductivity were measured using a pH and conductivity meter.

Adsorption test

A 400 microliter methylene blue solution was added to 1 ml of water in a quartz cuvette and initial absorbance was measured using an ultraviolet-visible (uv-vis) spectrophotometer at 665 nanometer wavelength. Calibration was carried out using distilled water. A 0.01 gram of biochar was mixed with the methylene blue solution, and the mixture was agitated at room temperature for 3 hours, with collection of a portion at 30 min intervals, which was allowed to stand for 10 minutes before absorbance measurement in the uv-vis spectrophotometer. The concentration of methylene blue after adsorption (C_e) was computed using the Beer's law (Equation 1) below:

$$\%\ A = \epsilon\ L\ c \qquad (1)$$

where % A = absorbance, ϵ = molar absorptivity constant, L = length of path travelled by light, c = solution concentration (mol L^{-1}). The amount of methylene blue adsorbed was computed using Equation 2 below (Hameed et al. 2007, Ahiduzzaman & Sadrul Islam 2016, Lonappan et al. 2016):

$$q_e = \frac{(C_o - C_e)\ V}{m} \qquad (2)$$

where q_e = uptake of dye by biochar (mg g^{-1}), C_o = initial dye concentration (M L^{-1}), Ce = final dye concentration (M L^{-1}), V = volume of dye solution (L) and m = mass of carbon (g). The kinetics of adsorption of MB on the biochar surface is a commonly adapted technique to characterise the efficiency of the material to adsorb smaller particles such as dyes. This technique can demonstrate the potential use of adsorbents in wastewater treatment and water purification to remove pollutants such as phenols, dyes and heavy metals (Lonappan et al. 2016).

Data analysis

The results of the influence of number of passes and clearance on the average particle size was analysed using a two-factor ANOVA with unequal sample sizes, which was carried out using SPSS version 16.0 and validated with Origin Pro 2016.

RESULTS AND DISCUSSION

Bamboo biochar production

Preparation of *B. blumeana* biochar by pyrolysis at 500 °C for 5 hours in a drum kiln yielded 32% biochar by weight, from the total biomass used as initial feedstock. The proximate analysis of the bamboo biochar products revealed that *B. blumeana* biochar had 64% fixed carbon, 19% volatile matter and 17% ash. In comparison, *Dendrocalamus giganteus*, slow pyrolysed at 500 °C, provided a lower fixed carbon yield (Hernandez-Mena et al. 2014). Meanwhile, moso bamboo (*Phyllostachys edulis*), pyrolysed at 500 °C using a nitrogen-assisted tube furnace, provided fixed carbon that was 16% lower, with 23% volatile matter and 30% ash content (Zhang et al. 2017). The high ash yield of bamboo biochar can be attributed to the silica content common to the grass family (Singh et al. 2017).

Different bamboo species contain varying proportions of cellulose, hemicellulose and lignin (Mui et al. 2008). This can affect the composition and yield of the biochar products, with each chemical component having different reactivities and responses to temperature changes (Vassilev et al. 2010, Crombie et al. 2013). Cellulose, which is the major component of plant biomass, degrades at 250–500 °C, while other components, except lignin, degrade at lower temperatures (Oyedun et al. 2013). Lignin decomposes at a very slow rate at temperatures below 500 °C because of the strong linkages within its molecular structure (Mui et al. 2008).

Properties of bamboo biochar

Size and size distribution

The size reduction of bamboo biochar was carried out by crushing, followed by controlled friction grinding in a super mass colloider. Super mass colloider reduces the size of the biochar, controlled by grinding speed and clearance between the ceramic grinding stones. Regular biochar sieved through a 1 mm mesh has an average particle size of > 500 µm. Passing the biochar through a friction grinder mass colloider could further reduce the size to < 1µm. Figure 1 shows the size distribution of *B. blumeana*

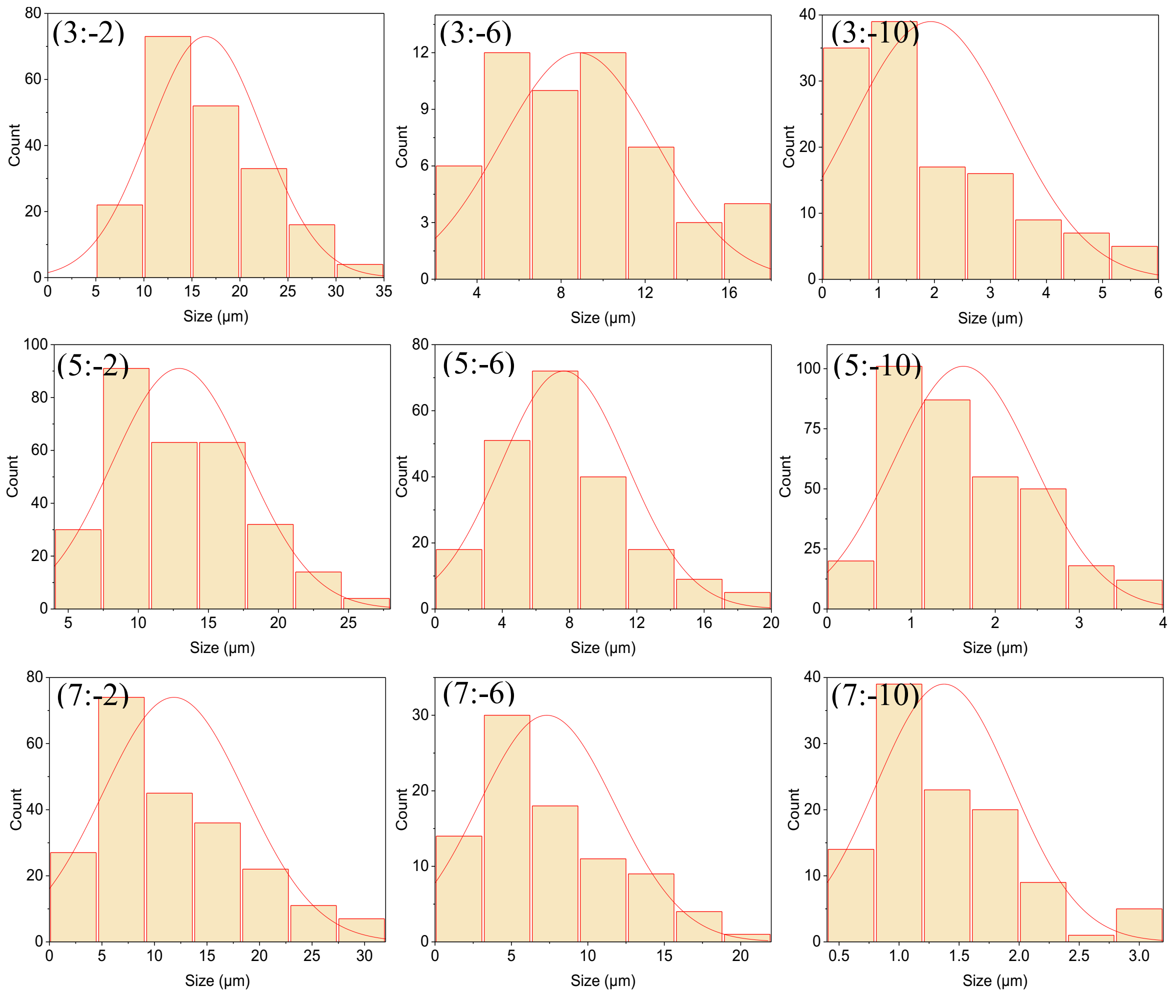

Figure 1 Size distribution of *Bambusa blumeana* biochar after controlled passes and clearance setting in the friction grinder supermass colloider; values at the upper left corner pertain to number of passes and grinder clearance (np:gc) setting

biochar from representative experiments combining different number of passes and grinder clearance settings. Figure 2 shows the corresponding scanning electron microscope images of the biochar particles with varying number of passes and grinder clearance settings. The combined effect of increasing number of passes and narrowing the clearance reduced the size and narrowed the size distribution of biochar particles. Fragments of biochar were irregularly shaped. The size was measured from the longest sides of the particles by taking numerous images using FESEM. The number of passes (3, 5 and 7) did not strongly influence the reduction of the biochar particle size, although the average particle size slightly reduced with increasing number of passes. On the other hand, the grinder clearance setting had a strong influence on the average size and distribution of particle sizes. As the gap in grinder clearance became smaller, there was substantial reduction in particle size. The interaction between the two factors was significant based on a two-way ANOVA, as shown in Table 1. Post hoc analysis (Figure 3) revealed that particle size reduced with increasing number of passes and narrower clearance. The strong influence of the number of passes was only seen in the 3 and 5 passes with larger clearance. The combination of both factors improved the uniformity and reduced the particle size. There were no significant differences found among the (3:-10), (5:-10) and (7:-10) combinations of number of passes and grinder clearance (np gc^{-1}) setting. However, it was found that as the number of passes changed, better particle uniformity was obtained, as the range in size distribution narrowed.

The limit of the grinder clearance setting was negative 10. In the preliminary experiments, setting to a narrower clearance than negative 10 introduced impurities from the grinder. The number of passes, on the other hand, was limited to 7 passes in consideration of energy consumption during production. Figure 4 shows

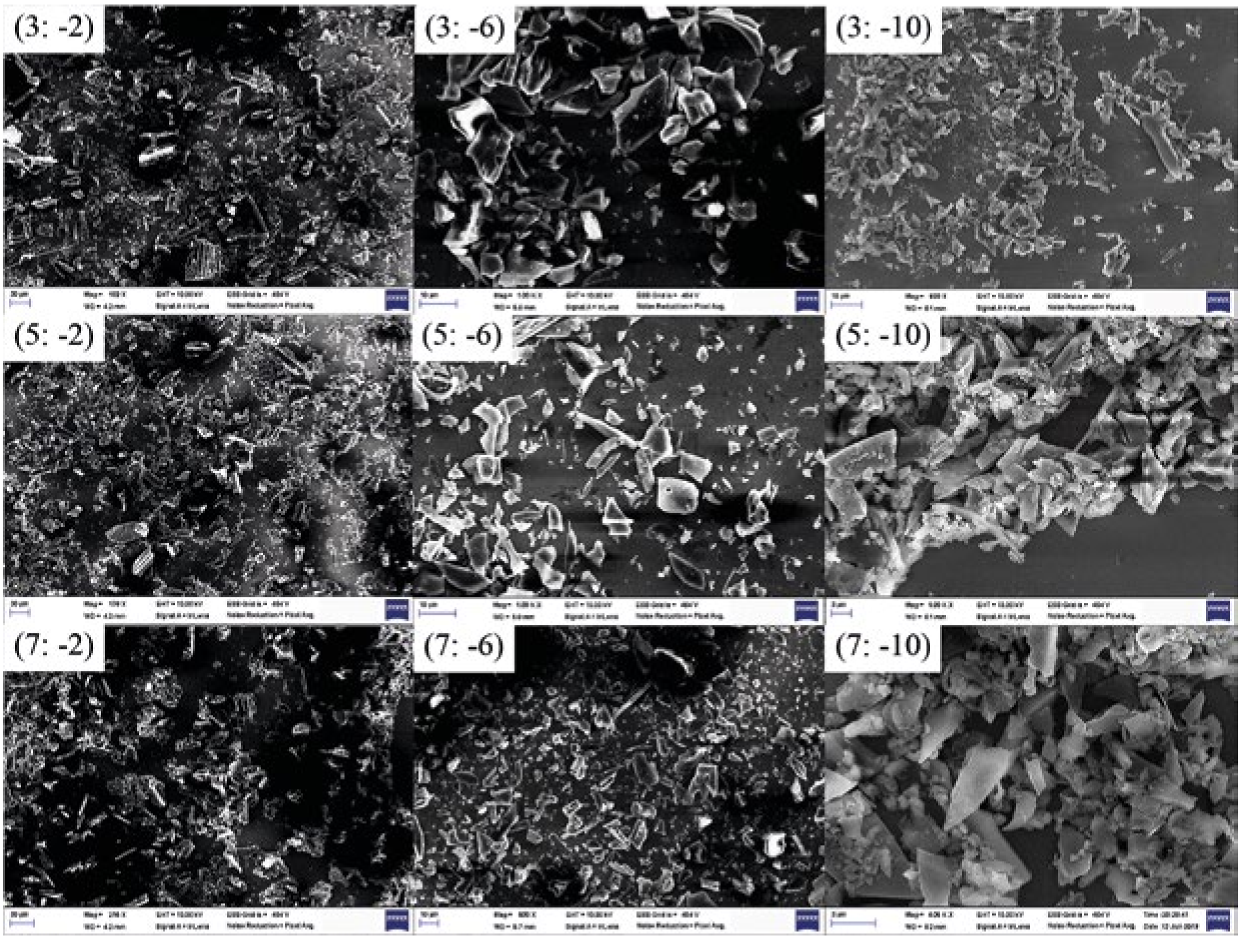

Figure 2 Field emission scanning electron microscope (FESEM) images of *Bambusa blumeana* biochar after controlled passes and clearance setting in the friction grinder supermass colloider; values in the upper left corner refers to number of passes and negative clearance (np:gc) setting

Table 1 Two-way analysis of variance (two-way ANOVA) of factors that influence the size reduction of bamboo biochar using friction grinder supermass colloider

	DF	Sum of squares	Mean square	F value	P value
No. of passes	2	1943.6	971.8	53.1	0.0
Clearance	4	53179.9	13295.0	726.9	0.0
Interaction	8	1299.1	162.4	8.9	0.0
Model	14	63544.9	4538.9	248.2	0.0
Error	2708	49528.4	18.3		
Corrected total	2722	113073.3			

At the ≤ 0.05 level, the population means of number of passes are significantly different, at the ≤ 0.05 level, the population means of clearance are significantly different, at the ≤ 0.05 level, the interaction between number of passes and clearance is significant

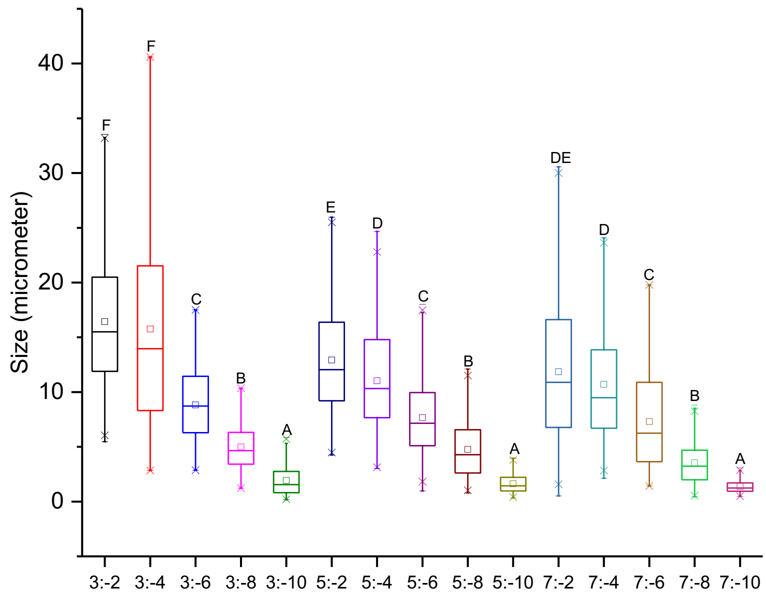

Figure 3 Boxplot of post hoc analysis of particle size processed in controlled passes and clearance in friction grinding; varying superscript letters means significant difference at p < 0.05 confidence interval

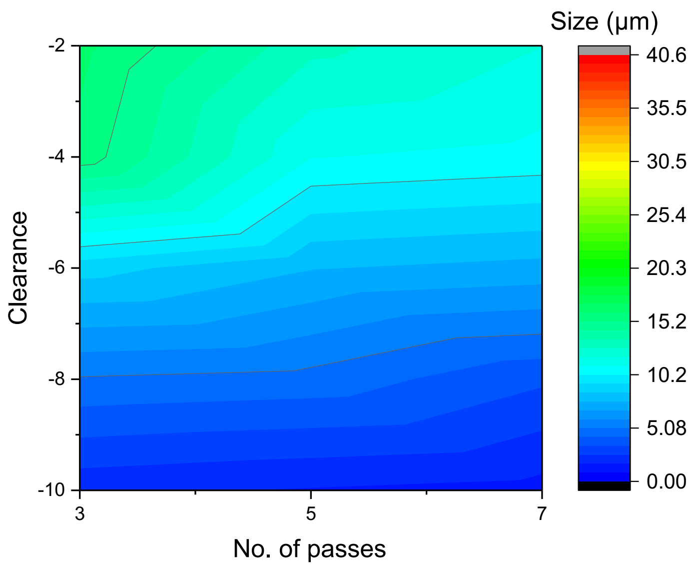

Figure 4 Contour plot of size reduction of *Bambusa blumeana* biochar after several passes and various clearance setting in the friction grinder super mass colloider

a contour plot depicting the effects of number of passes and grinder clearance on the size of bamboo biochar particles. It was found that friction grinding reduced the average particle size to ~ 1µm. The time required for the entire process was 30 minutes at most. When the friction grinder setting was at -2 clearance, a very low percentage of recovered particles gave sizes of ~ 40µm. This was overcome by increasing the number of passes, which reduced the size of the bamboo biochar to 10–15 µm. At -6 clearance, and with only 3 passes, particle size was down to 10 µm. When the number of passes was set at 7, the average particle size was 5 µm. The combination of -10 clearance and 7 passes provided the maximum reduction in size, averaging 1 µm. It is noteworthy that the 3, 5 and 7 passes could provide an average particle size of 1 micrometer; however the 7 passes yielded better uniformity of particle sizes.

The reduction in the particle size of bamboo biochar is relevant for its various applications. When added to composite materials, the small size of particles will improve the density of the composite and enhance the matrix by crosslinking the macromolecular chains (Zhu et al. 2016). Small particle size was also found to be effective when used as additive to packaging materials, to avoid growth of pathogenic fungi and contamination by adsorption of calcium ions (Yang et al. 2016). Smaller particle size biochar also has greater contaminant sorption capacity allowing the material to facilitate the removal of contaminants in liquid medium and in soil (Wang et al. 2012, Qiu et al. 2017).

pH, electrical conductivity and adsorption properties

The pH and electrical conductivity of bamboo biochar were measured following the procedure of Singh et al. (2017). Figure 5 shows the pH and electrical conductivity of raw *B. blumeana* biochar ground in a friction grinder super mass colloider. A basic pH ranging from 9.55 to 9.98 was obtained. No significant changes were observed in the pH of the bamboo biochar with reduced size, by friction grinding up to 1.4 µm size. The basic pH of the biochar can be attributed to the feedstock used. It is common to find the biochar from plant sources to have higher pH, due to the presence of base salt metals in ash (Mullen et al. 2010). However, the high pH could also be due to the pyrolysis temperature. A previous study found that biochar pH is highly affected by the production temperature, more than the kind of feedstock (Zhao et al. 2013). Higher pyrolysis temperatures tend to increase the pH of biochar (Bagreev et al. 2001). This will lead to a highly aromatic and hydrophobic surfaced biochar, making the material an excellent medium for organic and inorganic contaminant removal (Keiluweit el al. 2010, Oliveira et al. 2017). The high pH provides better ionic interactions with the exposure of both positive and negatively charged surfaces in the biochar (Oliveira et al. 2017). Aside from contaminants, biochar with high pH level is effective in adsorbing toxic gasses such as H_2S, with excellent removal efficiency (Shang et al. 2013).

Electrical conductivity test obtained 11.58 to 14.3 S m^{-1}, which did not show significant difference with size reduction. Similar to pH, the electrical conductivity is also highly dependent on pyrolysis temperature (Kloss et al. 2012, Naghdi et al. 2019). During the biochar production process, excessive heating of the feedstock cause structural changes, leading to loss in total mass (Cantrell et al. 2012). Since electrical conductivity is a measure of dissolved salts in a solution, a higher concentration of the biochar particles increased the conductivity of the material (Pansu and Gautheyrou 2007). However, in this study, it was observed that further reduction in particle size had no significant effect on the electrical conductivity of biochar. The values for electrical conductivity are comparable to those of bamboo charcoal, as reported by Li and co-workers (2017). Biochar is composed of imperfectly arranged aromatic rings of carbon atoms without oxygen and hydrogen (Lehmann and Joseph 2015). The condensed aromatic structure has electrons, which make the biochar a semi-conductive material (Li et al. 2017). The conductivity is governed by the amount and distribution of embedded sp^3 and sp^2 sites that allows electron hopping within the amorphous material (Dasgupta et al. 1991). The sp^2 in the biochar matrix serve as conductors while sp^3 as insulators (Gabhi et al. 2020). Bearing a conductive property that could be improved by pyrolysis conditions, low tortuosity porous structure, continuous carbon matrix and cost effectiveness in mass scaling, biochar is a possible choice for production of electrode materials for supercapacitors and capacitive deionisation (CDI) in wastewater treatment (Gabhi et al.

2020). This electrical property of the biochar can also serve as a basis for its use as an eco-friendly and economic source of carbon for shielding applications in films and structural materials (Quaranta et al. 2016).

The adsorption properties of processed and unprocessed biochar were measured by monitoring the change in the concentration of methylene blue (MB) using uv-vis spectrophotometer. The biochar was subjected to drying at 150 ˚C for 48 hours before the test was conducted. Figure 6 shows the adsorption of biochar to methylene blue. Values were computed based on the change in the concentration of MB at room temperature as a function of time. Prior to the actual tests, a prolonged duration of water immersion beyond 180 minutes, with constant agitation, resulted in unstable concentration

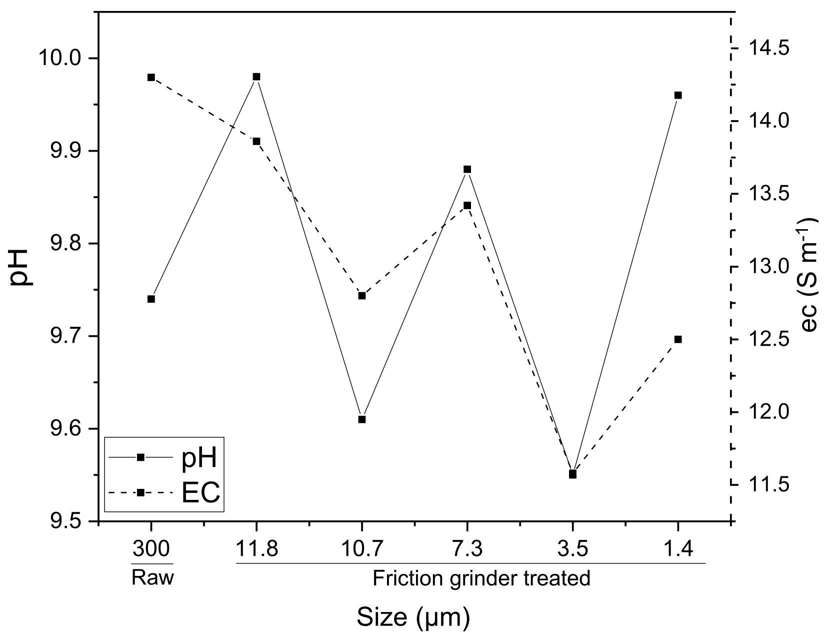

Figure 5 Contour plot of size reduction of *Bambusa blumeana* biochar after several passes and various clearance setting in the friction grinder super mass colloider

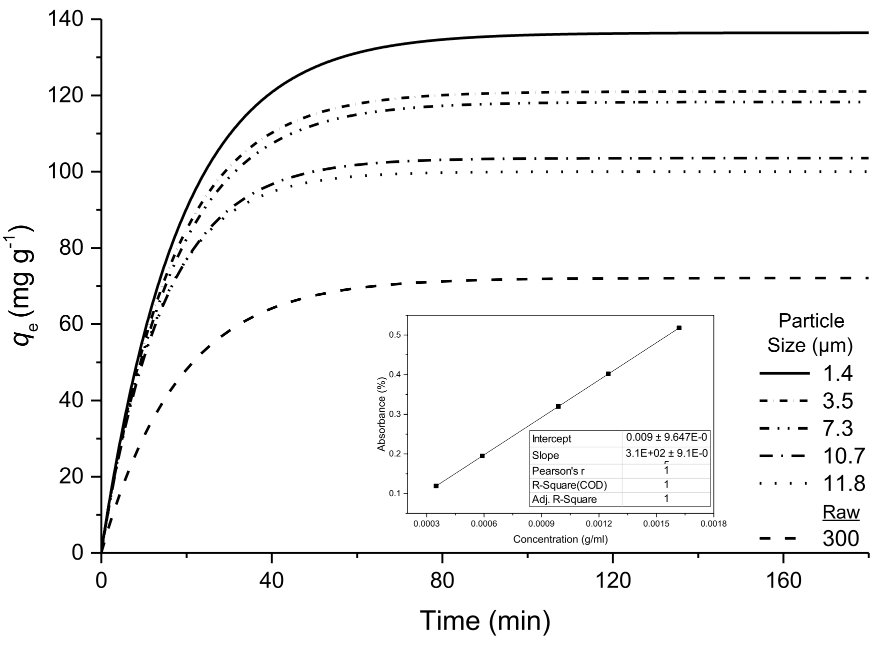

Figure 6 Methylene blue (MB) adsorption (qe mg g^{-1}) of raw and friction grinder processed biochar, inset is the concentration versus absobance graph to determine the coefficient of extinction

of MB, implying a desorption/adsorption in the mixture. Most of the MB were adsorbed within the 30-minute agitation and then slowed down until the optimum point was reached, in accordance to normal kinetics of adsorption (Kannan & Sundaram 2001). Biochar is a porous material having a very high surface area. As expected, reducing the particle size would improve the surface area and increase the avenues for contact with methylene blue molecules. Compared to unprocessed biochar, methylene blue adsorbed by process improved from 77.5 mg g^{-1} to 139.5 mg g^{-1}, a 79.8% increase. This was a significant improvement in the adsorption property, considering that the biochar was not activated. Biochar adsorption follows the Langmuir isotherm model, wherein MB molecules were attached to a monolayer with homogenous distribution of active sites on the surface (Sun et al. 2015, Lonappan et al. 2016). Increasing the exposed surfaces of the biochar, due to size reduction, and exposure of pores are relevant in the adsorption behavior of the material. Figure 7 shows the images of processed biochar with developed macro, meso and micro pore structures in the surface. Biochar have sub-micropores (≤ 2 nm), micropore and mesopores (2–50 nm) and macropores (> 50 nm), which are easily observed in the SEM images (Brewer et al. 2014). The mesopores are the most active in terms of chemical sorption which highly contributes to restraining desorption of MB attached to the biochar surface (Brewer et al. 2014).

CONCLUSION

As the first research to report on using friction grinder supermass colloider for reducing the particle size of biochar, the study has proven the capacity of ultra-fine friction grinding in reducing the particle size of bamboo biochar to less than 1 μm. This was achieved by narrowing the grinder clearance and increasing the number of passes in the colloider. No changes were observed in pH and conductivity of biochar, but there was huge improvement in the MB adsorption efficiency of the material, increasing up to 79.8%, compared to raw biochar. With low requirement of energy and time, the friction grinder can produce ultra-fine biochar which could be used in various applications such as nanosized activated carbon production, adsorbents in contaminated soil and water treatment, composite fillers and shielding material.

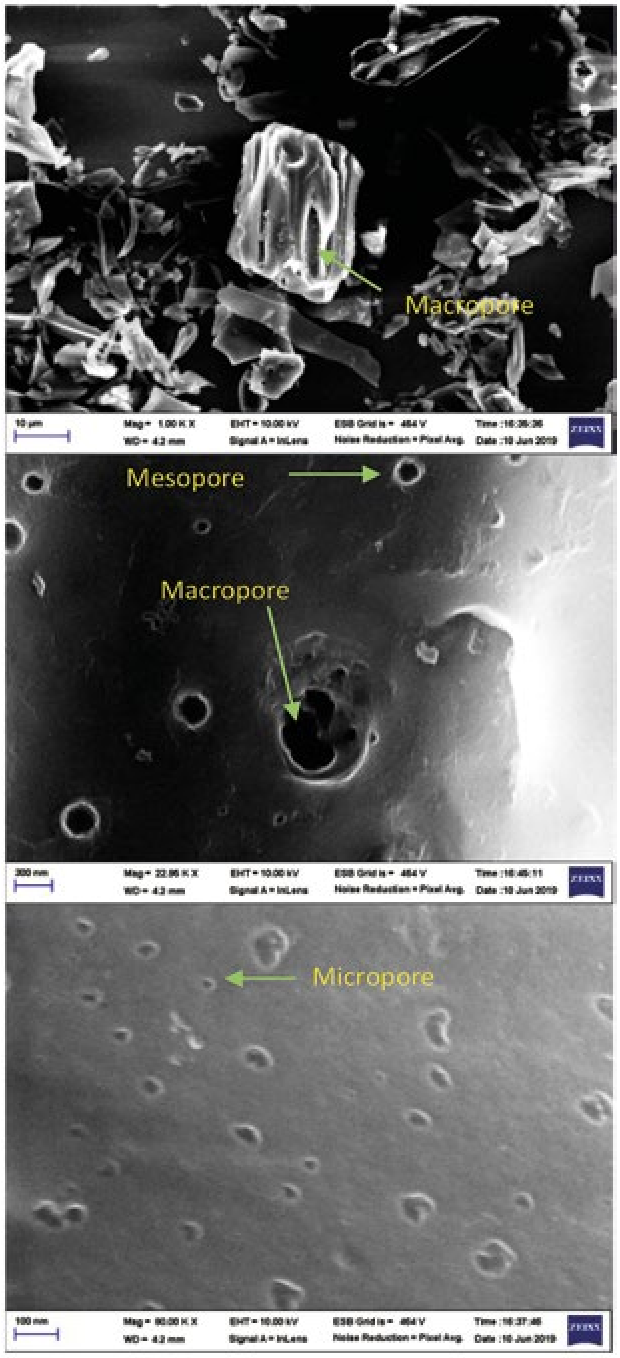

Figure 7 Field emission scanning electron microscope (FESEM) images of pore structures of bamboo biochar

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