Characterization of Blending Hydrogel Biochar as an Adsorbent Derived from Sugarcane Bagasse and Flyash

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***Abstract*—Biomass are a good choice to be used as an adsorbent due to their availabilty in the environemnt and reducing the waste in surroundings indirectly. Large volume of industrial by-products, for instance palm kernel shell, sugarcane bagasse, sawdust, rice husk and others are produced. In this study, biochar from Saccharum officinarum (L), or sugarcane bagasse (SB) was obtained using slow pyrolysis. The raw sample with different ratio of biochar and flyash (FA) was blended with hydrogel. The hydrogel was produced by mixing the biochar with a monomer, that is acrylamide (AAm), a cross-linker, that is methylenebisacrylamide (MBA), and with the presence of an initiator, that is ammonium persulfate (APS). The properties and characteristics of the blended hydrogel biochar have been made through different instrumental methods like field emission scanning electron microscope (FESEM) to obtain the pore structure and distribution, Brunauer-Emmett-Teller (BET) surface area analysis and proximate analysis using Thermogravimetric Analysis (TGA). The blended hydrogel biochar of ratio 50:50 (SB:FA) is found to be a better adsorbent with having larger surface area of 3.7297 m^2/g and its fixed carbon percentage of 24.7655%.**

***Keywords*— adsorbent, BET, Biochar, biomass, characterization, FESEM, hydrogel, slow pyrolysis, sugarcane bagasse, TGA.**

INTRODUCTION

In order to maintain a contemporary lifestyle of a mass production, mass consumption and mass disposal, large amount of natural resources that includes food and energy were used (Suzuki *et al.*, 2016). Due to this, the conversation of energy was promoted which then lead to the introduction of renewable energy as the issue of global warming has increased in severity (Maeda *et al.*, 2016).

One of the mass disposal component is the agricultural wastes. Agricultural waste is a general term used to describe waste produced on a farm through various farming activities, which includes both natural (organic) and non-natural wastes. Agricultural and food industries residues and wastes contribute a significant proportion of worldwide agricultural productivity.

Agricultural materials particularly those containing cellulose shows potential metal biosorption capacity. The basic components of the agricultural waste materials biomass include hemicellulose, lignin, extractives, lipids, proteins, simple sugars, water hydrocarbons, starch containing variety of functional groups that facilitates metal complexation which helps for the sequestering of heavy metals as well as acts as a good adsorbents to remove gaseous pollutants.

The fast development of environmentally friendly technologies is influenced by the desire to achieve the green environmental policy status. A number or researchers have advocated in utilizing biological waste materials as activated carbon precursors (Gaspard & Ncibi, 2013). Biomass, material that possess high surface areas, can be a source of adsorbents comparable wth the commercial activated carbons.

Biomass is the term that refers to any organic matter that has been derived from plants as a result from plant and animal material. For instance, from the industrial, human or animal wastes, and residues resulted from agricultural and forestry processes (Basam, E. N., 2010). Due to it is widely distributed in nature, adsorbents based on the relatively inexpensive biomass aroused broad concern. Furthermore, the other advantages of biomass are that it is available locally, low cost and is an effective material preferred adsorbent (Jia *et al*., 2016). Biomass decompose naturally and releases H_2O, CO_2 and energy during the process. Similar process occurs when biomass is utilized for chemical or energy purposes. Hence, it emits zero net pollution (Langille, J., 2009).

Solid carbon-rich residue acquired from the thermal decomposition of plant derived biomass in the absence of oxygen or in a partial oxygen condition is a common description of biochar. Biomass is not to be confused with biochar, as it is basically defined as substance of a mixture of lignin, cellulose, and hemicellulose in which each of them is then pyrolysed or degraded at various rates and mechanism relying on the pyrolysis temperature, biomass heating rate and pressure.

Certain research indicates that biochar was first found at the Amazon Basin of South America, where the Native Indians began pilling up wood stock in pits and slowly burning them in the absence of air to produce biochar (Thines *et al.,* 2016) in the early stage of civilization.

Biochar, in other terms is a porous substance, it appearance is similar to charcoal that is produced by pyrolysis process of feedstock without the presence of oxygen. It contains stable carbon which remains sequestered for much longer periods than the original biomass. The characteristics of biochar include very low homogeneity, exhibits low adsorption capacity at lower temperature whereas at higher temperature, it has greater surface area and high microporosity (Kolodyńska *et al.,* 2012).

Conventional application of biochar is to improve the overall sorption capacity of oil towards common anthropogenic organic compounds, pesticides, herbisides as well as dyes (Mohan *et al.,* 2014) and hence, influenced the toxicity and transport of such contaminants and decrease their short term risk. The advantages of biochar may broaden to soilless substrates used in the greenhouse industries, and the porous nature of biochar may make it suitable replacement for perlite in greenhouse substrates (Northup, 2013). This application on reduction of greenhouse gas emissions is crucial as carbon dioxide, methane and nitrous oxide are produced which has positive influence on the climate changes.

The application of fly ash nowadays has gained a considerable amount of attention in public and industries, which influence the reduction of environmental problem and enhance the economic

benefit. Fly ash is defined as finely divided mineral residue resulting from the combustion of ground or powdered coal in electricity generating plant (Woolard *et al.,* 2000).

Currently, South Africa generates more than 25 million tons of ash annually, of which nearly 1.2 million tons are utilized for various reasons. The application of fly ash as mentioned by Saakshy *et al.* (2015) includes manufacturing of cement and other building components, back-filling mines, agriculture and other related applications. Research made has revealed that unburned carbon component in fly ash plays an important role in adsorption capacity.

Studies on how to boost the potential of fly ash by researchers are now being focused on presently through proper beneficiation techniques in order to increase its adsorption rate. Bada and Potgieter-Vermaak (2008) stated in their journal that there are several method available to increase fly ash's specific surface area and its adsorption capacity, that include the modification of surface technology involving addition of HCl, the steam activation of unburned carbon in fly ash and sonochemical technique with NaOH.

Instead of using conventional costly activated carbon, researches of fly ash is being conducted for adsorption of nitrogen oxides, oxides of sulphur, organic compound and mercury in air, and cations, anions, dyes and other organic matters in water (Girón *et al.,* 2015). Fly ash is recognized as a promising adsorbent for removal of various pollutants (Kulkarni *et al.,* 2013).

Chemical treatment of fly ash will make conversion of fly ash into a more efficient adsorbent for gas and water cleaning. However, fly ash's chemical characteristics is greatly influenced by crucial factors, which are the geological origin of the coal, method and condition of combustion. Fly ash exhibits good adsorbent properties (Lakdawala *et al.,* 2012) as well as its availability and presence as a low-cost waste product make it a good opportunity and essential step towards reduction of its disposal to environment.

Significant progress has been made in the field of hydrogels as an adsorbent over the past decades. The presence of hydrophilic groups in the polymeric network, which becomes hydrated in aqueous media is what makes the hydrogel structure. Researchers, over the years, have defined hydrogels in many different ways. The most frequent of these is that hydrogel is a water-swollen, and cross-linked polymeric network produced by the simple reaction of one or more monomers (Ahmed, 2013).

Hydrogels are prepared from materials such as gelatin, polysaccharides, cross-linked polyacrylamide polymers, polyelectrolyte complexes, and polymers or copolymers derived from methacrylate esters (Shetye *et al.*, 2015). They can be found in dry or hydrated sheets or as a hydrated gel in drug delivery systems designed for single use and are insoluble in water.

Because of its potentiality to absorb water, hydrogels are under research to explore the fundamentals of swollen polymer network. This ability to absorb water arises from the hydrophilc functional groups that are attached to the polymeric backbone. In the swollen state, they are soft and rubbery, which verifies that they posses a degree of flexibility that is very similar to natural living tissues. They also have broad application in many technical areas, such that as devices for controlled release of protein and drugs, materials for protein separation and contact lenses and dies for encapsulating cells.

METHODOLOGY

Materials

The sugarcane bagasse used in this study was taken at te nearest night market. The bagasse was dried at 105 ºC and stored in a plastic sacks. The flyash used was taken at the nearest coal combustion power stations and was stores in a zipper plastic bags. It has similar chemical composition to that volcanic ash, which contain elements that are also found in rocks and soil (Bold, 2013).

B. Preparation of biochar

Slow pyrolysis

The dried bagasse was cut into a smaller sizes. The experiment was carried out in a laboratory pyrolyzer available in the university. About 100-150 g of hte dried cut bagasse was placed into the pyrolyzer, which is then heated from rrom temperature to 550 ºC at 30 ºC/min, and was maintained for atleast one hour to allow complete pyrolysis. The vapor produced from the pyrolysis process was purged by nitrogen at 100 ml//min by passing the gas through (Morali *et al.,* 2016).

Treatment of biochar

Chemical activation of the biochar was done using zinc chloride (ZnCl) where 500g of dried biochar is well-mixed with 3000mL of concentrated solution of of ZnCl that contains 500g of ZnCl. It then resulted in a slurry and was kept for about 24 h to allow proper soaking (Das *et al.,* 2015). It was filtered using filter pump set and and then was washed with 0.5M of HCl. After that, the mixture is washed with distilled water for several times as to achieved pH value of 7.0.

Preparation of blending hydrogel biochar

In order to prepare the hydrogel, the treated biochar with various weight ratios is basically mixed with a monomer and a cross-linker with the addition of an initiator. 1.0g of acrylamide (AAm) as the monomer was dissolved in 1mL of distilled water and 0.06 of treated biochar with 0.01mmol of methylenebisacrylamide (MBA) as the cross-linker were added to the AAm solution.

After that, 0.2mL of 0.01mmol aqueous solution of ammonium persulfate (APS) was added in order for the initiation of polymerization and was placed in a plastic mould (Karakoyun *et al*., 2011). The hydrogel was dried in an oven at 40 ºC and stored in a dessicator after being cut into desired sizes.

Table 1: Proportions of blended materials in mixtures used in experiment

Sample	SB (%)	FA (%)
HB(100SB)	100	0
HB(80SB)	80	20
HB(60SB)	60	40
HB(50SB)	50	50
HB(40SB)	40	60
HB(20SB)	20	80

*HB(50SB) stands for Hydrogel Biochar with 50% ratio of Sugarcane Bagasse and the other 50% of flyash.

Characterization of biochar

The characterization of original treated biochar and blending hydrogel biochar were carried out to compare with each other. Surface area and pore volume of blending hydrogel biochar was analyzed by N_2 adsorption/desorption isotherm using Model 3Flex Version 3.02 by Brunauer-Emmette-Teller (BET) method. The analysis was completed at UiTM Shah Alam Faculty of Chemical Engineering. Thermal analysis of the blending hydrogel biochar was carried out by using thermogravimetric analysis (TGA) (Model Mettler Star^e SW 14). About 20mg of sample was taken and analyzed with a heating rate of 10 ºC/min to 800 ºC under N_2 atmosphere for biomass with a flowrate 100 mL/min (Varma & Mondal, 2017).

Field emission scanning electron microscope (FESEM) was used to observe the surface morphology of experimentally prepared blending hydrogel biochar. The analysis was completed in UiTM Puncak Alam Faculty of Pharmacy. The specification for the high-resolution imaging and analysis of the non-conductive samples were: acceleration voltage: 10 kV; resolution: 12nm; distance: 10mm and magnification: 200. The samples were then coated with platinum for conductivity (Pan *et al.*, 2017).

RESULTS AND DISCUSSION

Characteristics of treated biochar and blending hydrogel biochar.

Surface Area Analysis

The BET-method is the most commonly used for characterization of catalysts, adsorbents and other porous materials (Mel'gunov & Ayupov, 2017). Its application is not restricted to research and development, but also concerning the control of technology in industrial applications. The Brunauer-Emmett-Teller equation was used by the software in order to calculate the specific surface area of the test samples.

Table 2: Analysis obtained for various ratio of hydrogel biochar

Ratio	HB (20SB)	HB (40SB)	HB (50SB)	HB (60SB)	HB (80SB)	HB (100SB)
BET Surface Area (m^2/g)	0.2865	2.1901	3.7297	0.4703	1.2726	0.4676
BJH Desorption (m^2/g)	0.7171	1.7049	2.7959	0.7364	1.0543	0.54

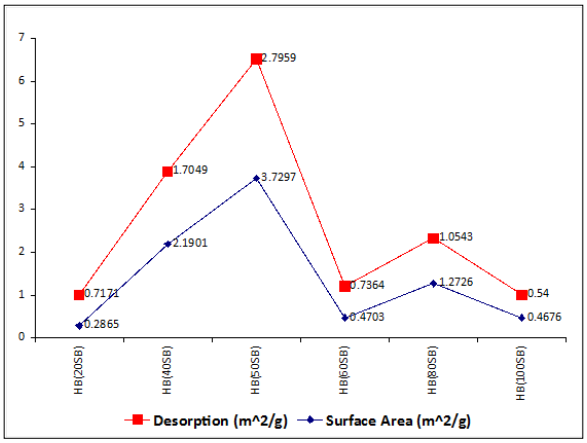


Fig. 1: Graph plotted of the BJH-theory of desorption and BET-method of surface area.

The way of solids burn, react, and dissolve with other materials can be determine by its surface area. It can be seen from the data above that the sample of 50:50 ratio of sugarcane bagasse and flyash has the highest surface area value of 3.7297 m^2/g when compared to the other samples. Flyash is known for its adsorbent properties such as it has large specific suface area, higher content of carbon and fine particle size and a fine size of particle (Saakshy *et al.*, 2016). The specific area of surface of the flyash obtained usually varies from 250 to 850 m^2/g (Chatterjee, 2010).

Shackley et al. (2012) described biochar in more words as "the porous carbonaceous solid produced by the thermochemical conversion of organic materials in an oxygen depleted atmosphere that has physicochemical properties suitable for safe and long-term storage of carbon in the environment". The HB(50SB) sample is used for the other characteriazation analysis (TGA and FESEM) due to it having the largest surface area.

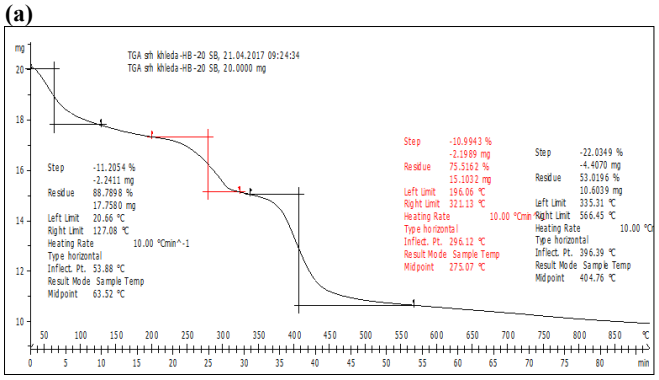
Thermogravimetric analysis

The principle of thermogravimetric analysis is to measure the amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere. The measured weight loss is expressed in a curve diagram that gives information on the thermal stability of the sample.

Basically TGA consists of a simple pan that is supported by a precision balance. During the experiment is conducted, that pan resides in a furnace and is heated or being cooled. The environment of the sample is controlled by the sample purge gas, which it may be inert or a reactive gas that flows over the sample and exits through an exhaust.

The technique can analyze materials that exhibit either mass loss or gain due to decomposition, oxidation or loss of volatiles (such as moisture). About 20mg of each samples with various ratio (HB(80SB); HB(60SB); HB(50SB); HB(40SB); HB(20SB)) was analysed. The weigth loss and the fixed carbon of the sample were calculated using the graph shown below.

(a)



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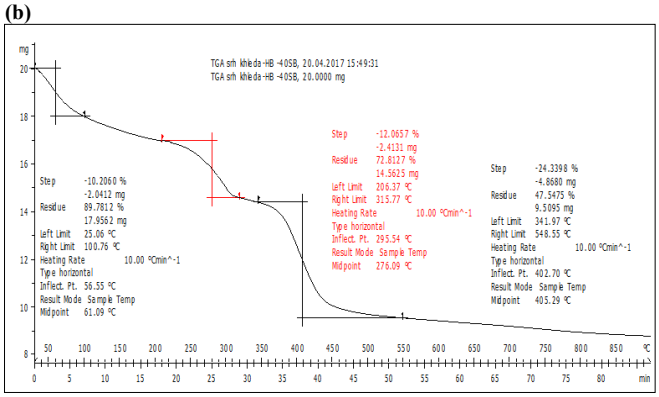

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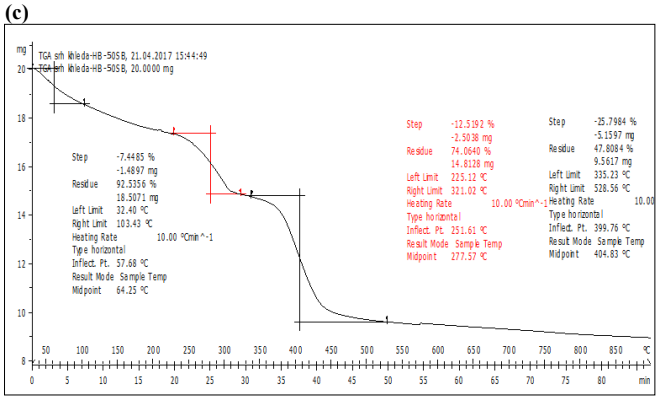
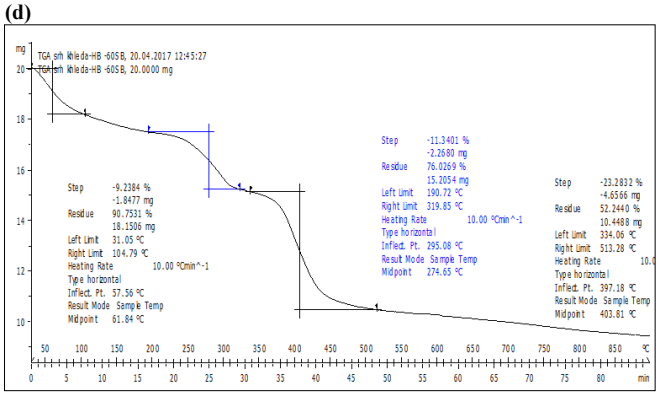
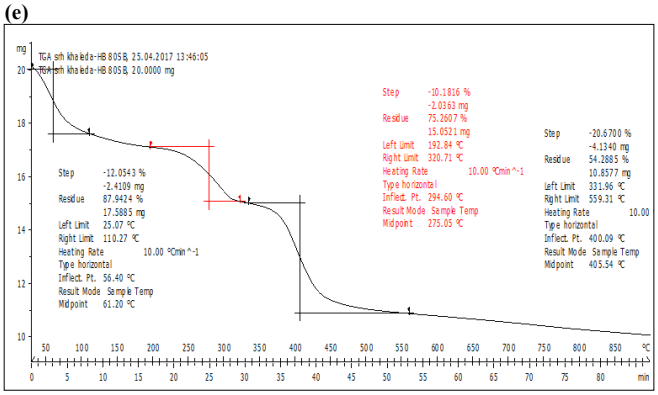


(d)



(e)



(f)

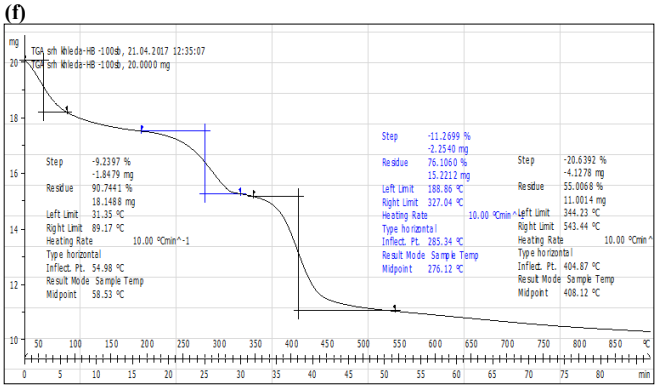


Fig. 2: Thermal analysis of (a) HB(20SB); (b) HB(40SB); (c) HB(50SB); (d) HB(60SB); (e) HB(80SB); and (f) HB(100SB)

Moisture content:

$$Moisture\ content\ (\%) = \frac{A-B}{B} \times 100\%$$
$$= \frac{20mg - 18.5071mg}{20mg} \times 100\%$$
$$= 7.4645\%$$

Where,
A = the weight of sample before heating process
B = the weight of sample after heating process

Volatile matter:

$$Weight\ loss\ (\%) = \frac{A-B}{A} \times 100\%$$
$$= \frac{18.5071mg - 14.8128mg}{18.5071mg} \times 100\%$$
$$= 19.9615\%$$

Where.
A = weight after no moisture
B = weight after the loss of volatile matter

Ash content:

$$Ash\ content\ (\%) = \frac{B}{A} \times 100\%$$
$$= \frac{9.5617mg}{20mg} \times 100\%$$
$$= 47.8085\%$$

Where,
A = the weight of sample before heating process
B = the residue left after heating process

Fixed carbon:

$$Fixed\ carbon = 100\% - moisture\ content\ (\%)$$
$$- volatile\ matter\ (\%) - ash\ content\ (\%)$$
$$= 100\% - 7.4645\% - 19.9615\% - 47.8085\%$$
$$= 24.7655\%$$

Table 3: Data extracted from the TGA graph

Sample	Inital weight (mg)	Weight after heating process (mg)	Weight after loss of volatile matter (mg)	Final weight (mg)
HB(100SB)	20	18.1488	15.2212	11.0014
HB(80SB)	20	17.5885	15.0521	10.8577
HB(60SB)	20	18.1506	15.2054	10.4488
HB(50SB)	20	17.7580	15.1032	10.6039
HB(40SB)	20	17.9562	14.5625	9.5095
HB(20SB)	20	18.5071	14.8128	9.5617

Table 4: The proximate analysis of the blending hydrogel biochar

Sample	Moisture Content (%)	Volatile Matter (%)	Ash Content (%)	Fixed Carbon (%)
HB(100SB)	9.2560	16.1311	55.0070	19.6059
HB(80SB)	12.0575	14.4208	54.2885	19.2332
HB(60SB)	9.2470	16.2265	52.2440	22.2825
HB(50SB)	7.4645	19.9615	47.8085	24.7655
HB(40SB)	10.1740	18.8999	47.5475	23.3795
HB(20SB)	11.2100	14.9499	53.0195	20.8206

The calculation used to obtained values of other sample tabulated above is the same as shown for HB(50SB). Based on the results above, the moisture content of all the samples analyzed ranging from 7.4645% to 12.0575% which stated that the least moisture content is in 50:50 ratio of sugarcane bagasse and flyash.

Adsorbents with high content of volatile matter, fixed carbon and a low ash content were reported as a promising properties of the precursor (Loir *et al.*, 2007). Percentage of the ash content indicates the presence of minerals as impurities in the carbon. It is the remaining residue upon the burn off of the carbonaceous components suring pyrolysis. That being the case, in order to have the most active sites on the surface of the adsorbent, samples with the lowest ash content are preferred. High content of ash ultimately reduce the precentage of fixed carbon.

As for ash content, the value gradually increase with the increment of bagasse ratio it can be assume that the content of ash in the bagasse is higher after undergone the pyrolysis process. Some of the volatile matter present in the biomass should be decomposed into vapors during the pyrolysis process, thus showing a reduction in the percentage of volatile matter in the biochar. A high content of volatile matter often results in more un-burnt gaseous organic compound which then will lead in formation of carbonaceous components in fine particles (Li *et al.*, 2016).

Fixed carbon can be defined as a solid combustible residue that is left after the pyrolysis process and volatile matter is decomposed. Therefore in the the fixed carbon term, the higher value of fixed carbon is desired for the sequestration of carbon as an adsorbent (Barber, 2016). The HB(50SB) sample has the highest fixed carbon value of 24.7655%. This is parallel with the result of BET surface area where HB(50SB) has the highest value.

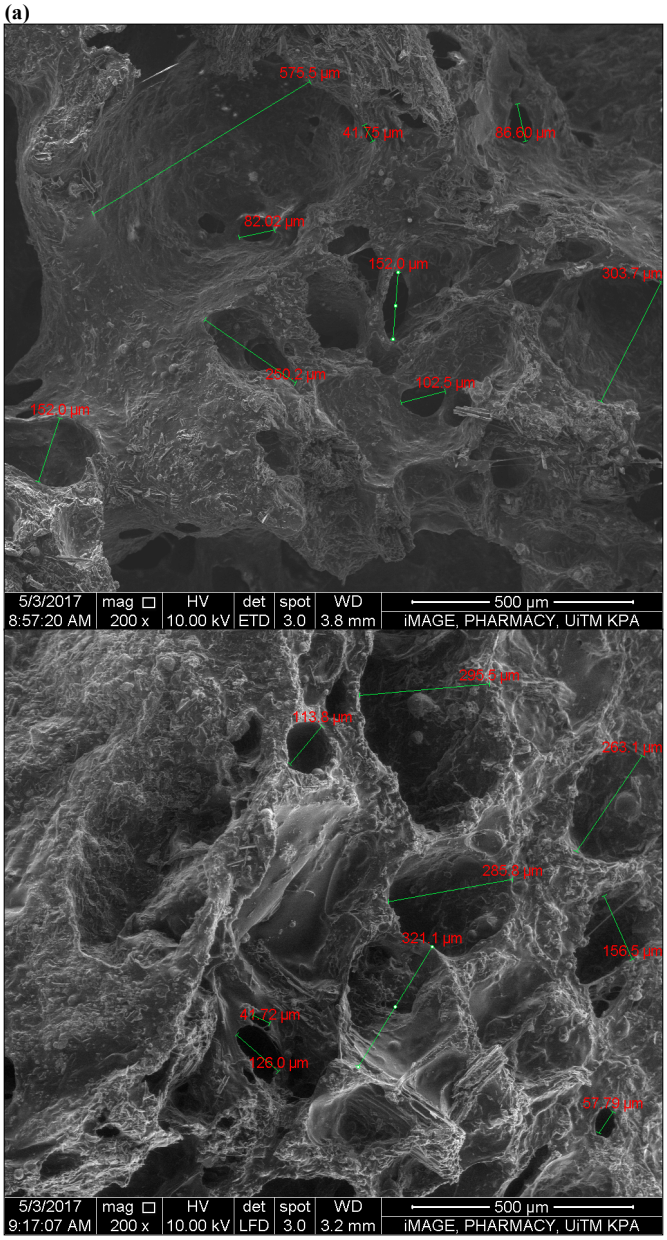
Field emission scanning electron microscope

The adsorptive properties are related directly to the adsorbent porosity, as a high porous carbon can adsorb relatively large amount of organic compound. Hence, HB(50SB) sample with surface area (3.7297 m^2/g) is higher than the other is said to be a good adsorbent. FESEM analysis is usually used to obtain the surface morphology of the sample desired. The images shown below are mainly used for the determination of the structure and distibution of the pores that are present on the surface of the hydrogel biochar.

Table 5: Pores type and its range of size (Llewellyn *et al.*, 2007)

Type of pores	Microporous	Mesoporous	Macroporous
Range of size, (nm)	< 2	2 -50	> 50

(a)



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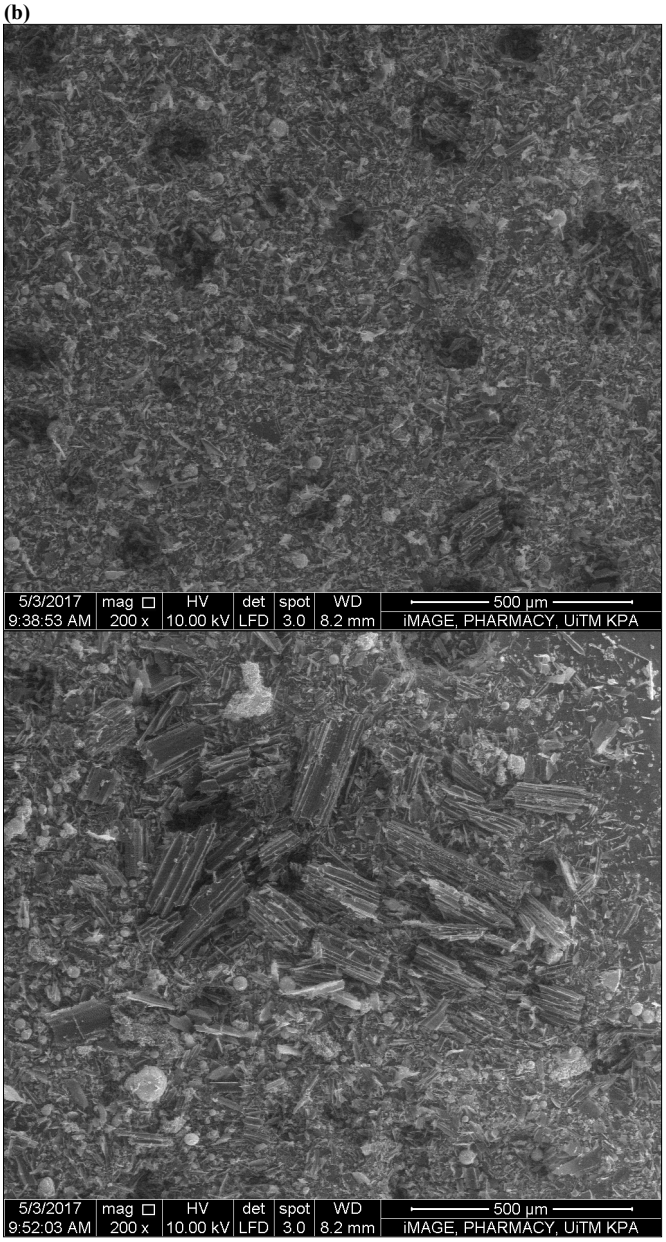

Fig. 3: Comparison of pores structure and distribution between (a) blending hydrogel biochar, and (b) raw biochar

Blending hydrogel biochar (HB(50SB)) with 200x magnification clearly shows the relative porous. The macropores are visible clearly, which will contributes in the easeness of the adsoption process of organic compound to the pore structure. The average range of macropores of hydrogel biochar is ranging between 40nm to 600nm. Conversely, the surfaces of the biochar (without hydrogel blending) are smooth and with the magnification of 200x, no pores can be seen. A research by Mangun *et al.* (1998) found that the capacity of adsorption is greater for materials with high surface area, which means that the material have a larger pore size due to the increment of pore volumes.

Based on the overall findings, the hydrogel biochar with ratio of 50:50 of sugarcane bagasse shows a promising properties of an adsorbent rather than the other four sample with different ratios.

CONCLUSION

The main aim of this study is to prepare blending hydrogel biochar as an adsorbent that is derived from biomass as it is redundant in the environment and is a low-cost material. The characteritics of the prepared samples were analysed using Brunauer-Emmet-Teller equation for its surface area, thermogravmetric analysis in order to determine the proximate

analysis, and field emission scanning electron microscopy to obtain the surface pore structure and morphology. Based on the findings of the experiment, the blending of hydrogel and biochar is a good match as flyash has its own properties that contribute greatly as an adsorbent and biomass, in this case is sugarcane bagasse also posses an adsorbent characteristics besides that it is widely available low-cost material. The BET results shows that HB(50SB) sample has the highest surface area of 3.7297 m^2/g. Meanwhile, the proximate analysis of the same sample shows value of 24.7655% of its fixed carbon percentage and having pore size ranging from 40nm to 600nm, which is macroporous. Therefore, the HB(50SB) of higher surface area has a greater adsorption capacity by having a large pore size and pore volumes. For a future work to expand the findings of this research, other type of raw biomass material can be used, such as livestock. FTIR analysis can be done in order to determine the presence of functional group that contributes to the ability of the biomass as an adsorbent.

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