

# Synthesis of Novel Magnetic Biochar Using Microwave Heating for Removal of Arsenic from Waste Water

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**ABSTRACT:** *Novel magnetic biochar has been successfully synthesized by using microwave technique, using discarded materials such as Empty Fruit Bunch (EFB). The optimized conditions for the best novel magnetic biochar synthesis are at 900 w reaction power, 20 min reaction time, and impregnation ratio 0.5 (biomas:FeCl<sub>3</sub>) The details physical and chemical analyses of novel magnetic biochar were found to be in good agreement with the hypothesis. These newly produced magnetic biochars have a high surface area 890 m<sup>2</sup>/g and that leads to highly efficient in the removal of arsenic (87%) from aqueous solution. As for new invention, magnetic biochar can be directly produced using microwaves heating by a single stage of activation compared to the conventional method.*

**KEYWORDS:** *Heavy metal; CNTs; Adsorption; Separation and purification.*

## INTRODUCTION

Arsenic is well known as a poison from historical time and is abundant on the earth crust. Contamination of ground water and surface water by arsenic from natural as well as an anthropogenic source like wood preservation, glass production, nonferrous metal alloys, and electronic semiconductor manufacturing is one of the most critical water quality problems around the world [1]. Prolong

intake of arsenic may result in cardiovascular, gastrointestinal, hematological, hepatic, renal, neurological, and carcinogenic health problem [2]. Therefore the concentration limit for arsenic is very stringent in most of the countries. According to USEPA and WHO the maximum permissible limit is 10 µg/L. Among others, technique adsorption is evolving as a front line of defense.

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1021-9986/2018/6/111-10 5/\$/5.05

Selective adsorption utilizing biological materials, mineral oxides, activated carbons, or polymer resins, has generated increasing excitement [3-7]. Activated Carbons (AC) made up of different materials are used for removing heavy metals and other toxic substances for long past. However, due to the high cost of AC [8], bio-char in recent year has recognized as a suitable material for agricultural and environmental applications [9-11]. An alternative technology developed recently to remove various pollutants, including phosphate, heavy metals, and organic compounds, from aqueous solutions by using specially modified biochars [12-13], known as magnetic biochar. Magnetic bio-char is produced by thermal conversion of  $\text{FeCl}_3$  pre-treated bio-material. Introducing magnetic biochar, to the commercial sorbent (e.g., active carbon and carbon nanotubes) by chemical co-precipitation is an efficient method, to enable the sorbent to be effectively separated by magnetic separating technique [14]. The thermal conversion of the bio material requires uniform heating to maintain the overall quality of the bio-char. The heating process should also be fast to reduce production cost. Microwave heating can be used to heat the bio-materials fast and uniformly throughout the bulk. The microwave penetrates the material and the microwave energy is converted to heat energy. In this way, heat is generated throughout the bulk of the material. This can reduce the processing time and the overall quality is improved [15-16].

In this work, we have prepared magnetic bio-char from palm Empty Fruit Bunch (EFB) by impregnating with  $\text{FeCl}_3$  and then thermal pyrolysis of the impregnated EFB with the help of microwave heating. This Magnetic Empty Fruit Bunch (M-EFB) char was used to remove arsenic from water solution.

## EXPERIMENTSL SECTION

### *Raw material*

EFB Empty Fruit Bunch (EFB) sample was obtained from the Seri Ulu Langat Palm Oil Mill in Dengkil, Selangor, Malaysia and stored at 4 °C. The EFB was first washed with distilled water to remove water soluble impurities and surface adhered particles and then dried at 105°C for 24 h for dehydration until a constant weight was obtained. Ferric chloride hexahydrate ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ) and sodium arsenate dibasic heptahydrate ( $\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$ ) of analytical grade were purchased from Merck and used as received.

### *Production of novel magnetic Biochar*

The dried biomass was crushed and sieved to a particle size of less than 150  $\mu\text{m}$ . A 20 g of dried biomass was well mixed with 10g of  $\text{FeCl}_3$  solution at an impregnation ratio (weight of activating agent/weight of dried biomass) of 0.5 to 1.15 for 4 h at room temperature. The impregnated samples were next dried at 100 °C and stored in a desiccator. The pyrolysis of biomass was carried out in a HAMiab-C1500 Microwave Muffle System oven. A 20 g of sample was prepared and placed inside the quartz tube (35 mm OD, 38 mm ID and 500 mm length). The power has been set initially to 300 W and later increased to desire power of 1000 W, the reaction time of 30 min with nitrogen gas flow of 0.2 mL/min. After the reaction was finished, the sample was allowed to cool down at room temperature. Then it was collected from the quartz tube and weighted to determine the yield of the product. Finally, the sample was stored in tightly closed bottles. These produced magnetic biochar then washed with distilled water until the pH becomes neutral.

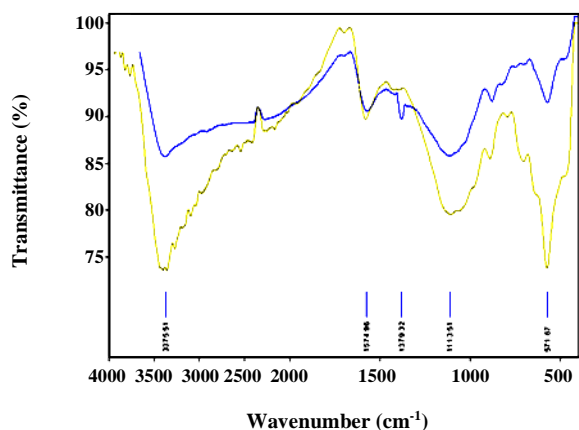
### *Preparation of stock solution for Batch Adsorption*

An Arsenic stock solution of 1000 ppm was prepared from sodium arsenate ( $\text{Na}_2\text{HAsO}_4 \cdot 7\text{H}_2\text{O}$ , Merck). The adsorption of arsenic by novel magnetic biochar was studied at various contact times i.e. 20 min to 5h and 24h. 1g of each novel magnetic biochar produced was added into a 250 mL conical flask containing 1000 ppm of an aqueous solution of arsenic. The sample was agitated at 150 rpm in a rotary shaker at room temperature ( $26 \pm 1$  °C). At the end of each interval of the time mentioned, the novel magnetic biochar was separated from the treated solution by filtration using the ADVANTEC qualitative filter paper no.161. The differences between the initial and the equilibrium metal ions concentrations determine the number of metal ions being absorbed by novel magnetic biochar using Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES) (Perkin elimer).

## RESULTS AND DISCUSSION

### *Characterization of Magnetic biochar using FT-IR*

Produced novel magnetic biochar before and after adsorption of heavy metal was successfully investigated using Fourier Transform InfraRed (FT-IR) spectroscopy



**Fig. 1:** FTIR Absorption Spectra, a) before adsorption of magnetic biochar, b) After adsorption of magnetic biochar.

(Bruker, IFS66v/S)). Fig 1 shows the FT-IR spectra of magnetic biochar before adsorption (a) and after adsorption (b). Results indicated that the functional groups have been successfully impregnated on the surface of magnetic biochar. It could be observed that the hydroxyl group, carboxyl group and methyl group, peak at a range of 3500 to 500  $\text{cm}^{-1}$  [17].

#### Characterization of mantic biochar using FESEM

Field Emission Scanning Electron Microscopy (FESEM)(Zeiss, Auriga) technique was employed to observe the surface physical morphology of the prepared raw biochar and produced novel magnetic biochar samples. Fig. 2 (a), (b) shows the images of raw EFB and the prepared novel magnetic biochar with different magnification scale (1 $\mu\text{m}$  and 100  $\mu\text{m}$ ). As can be seen from Fig. 2 (a), there were very little pores available on the surface of the precursor. However, after activation process under the optimum condition at reaction power 900 w, reaction time 20 min and 0.5 impregnation ratio, pores of different size and different shape were developed as shown in Fig. 2 (b). During chemical activation by  $\text{N}_2$  gas, the diffusion of the Oxidising agent through the carbonaceous matrix involves the removal of impurities and the consumption of carbon to create porosity. The micropores are opened and widened with even a shift to meso- and macropores the removal of the exterior of the particle is significant at high burn-offs [18]. This shows that  $\text{N}_2$  was effective in creating well developed pores on the surfaces of the precursor, hence leading to novel magnetic biochar with large surface area

and porous structure, which had high adsorption capacity on the removal of arsenic from aqueous solution.

#### Characterization of novel mantic biochar using BET Surface Area

Physical characterization of the best quality magnetic biochar produced was analyzed with Autosorb 1 surface area analyzer by nitrogen adsorption at -77 K. prior to analysis, the samples were degassed at 200  $^{\circ}\text{C}$  for 3 h. The surface area was calculated by the Brunauer, Emmett, and Teller (BET) (Brand: Quanta Chrome Model: Autosorb 6B) equation using the nitrogen adsorption data. Using the computer-monitoring system, the adsorbed nitrogen volumes and various equilibrium pressures, and BET surface area of the sample were reported. Results proved that higher surface area with higher pore size distribution. The specific BET surface area of the EFB based-magnetic biochar prepared was found 890 $\text{m}^2/\text{g}$ . The results obtained was agreed with a previous study [19] which reported that BET surface is higher than 350  $\text{m}^2/\text{g}$  after carbonization of lignin at 900 $^{\circ}\text{C}$ . Furthermore, for both precursors, carbonization forms only micropores and mesopores, while macropores are generated only during the activation step. This phenomenon was reported in other studies and may be explained by the fact that the activation not only forms the pores but also widens the size of the existing pores [20-21].

#### Removal of heavy metal using novel magnetic biochar

Fig. 3 represents, the adsorption kinetics for the removal of arsenic using magnetic biochar. In order to study the arsenic removal from the aqueous solution, three different magnetic biochar with iron loading 0.5, 1.1, and 1.1.5 impregnation ratio were studied. Among the three biochars, 0.5 impregnation ratio removing arsenic high efficiently compared to the other two (1.1 and 1.1.5 impregnation ratio). The newly produced magnetic biochars have high surface area 748.5  $\text{m}^2/\text{g}$  and that leads to highly efficient efficiency in the removal of arsenic (87%) from aqueous solution.

#### CONCLUSIONS

Novel magnetic biochar has been successfully produced using microwave heating. The optimized conditions for the best novel magnetic biochar synthesis are at 900 w reaction power, 20 min reaction time,

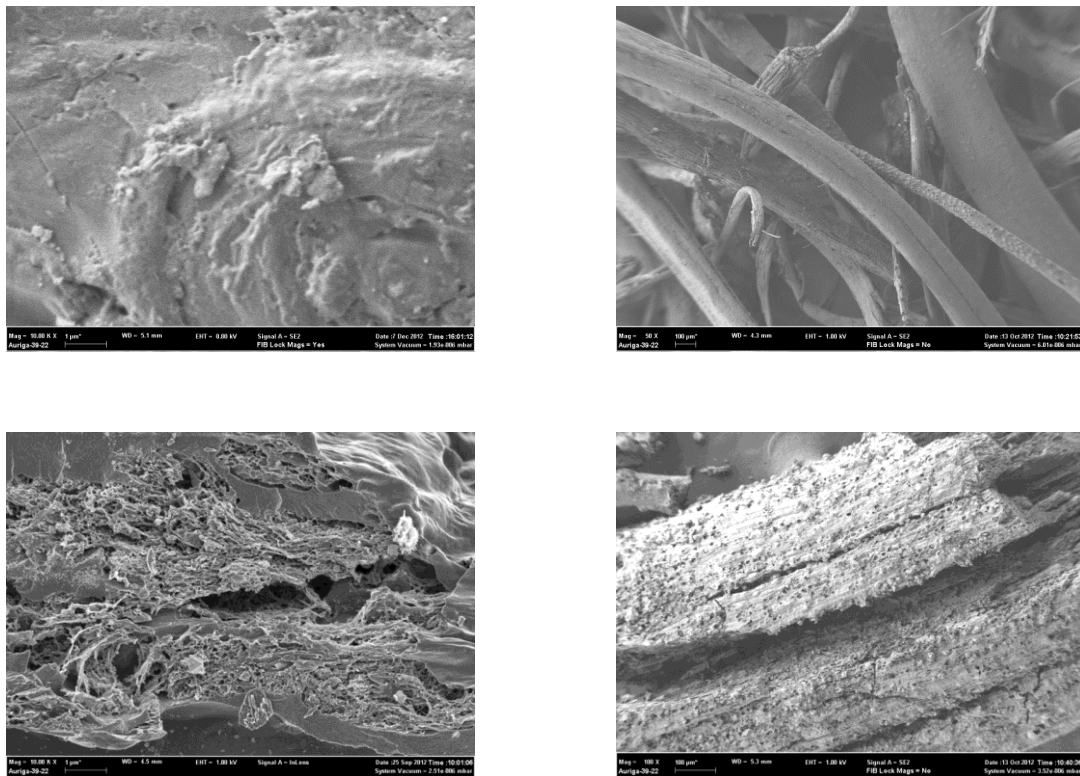


Fig. 2: a) shows a FESEM image of raw EFB at different magnification, b) shows a FESEM image of magnetic biochar produced at reaction power 900 w, reaction time 30 min and 0.5g impregnation ratio, at different magnification.

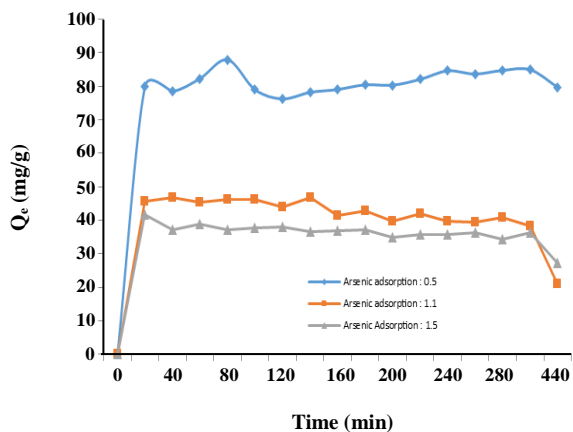


Fig. 3: Adsorption kinetics for the removal of arsenic using magnetic biochar.

and 0.5 impregnation ratio. These newly produced magnetic biochars have a high surface area  $890 \text{ m}^2/\text{g}$  and that leads to highly efficient in the removal of arsenic (87%) from aqueous solution. As for new invention, magnetic biochar

can be directly produced using microwaves heating by a single stage of activation compared to the conventional method. The finding of this study adds a new dimension to the materials as an adsorbent for the removal of arsenic from aqueous solution. This magnetic biochar is recognized as a high-efficient and cost-effective sorbent for different kinds of pollutant removal. In addition, these magnetic properties of biochar could help for many useful applications for replacing activated carbon with magnetic biochar, due to its high surface area, and high porosity and high adsorption capacity.

Received : Dec. 14, 2016 ; Accepted : Apr. 23, 2018

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