

Adsorptive characteristics and microwave dielectric properties of oil palm empty fruit bunch-based activated carbons for dye removal

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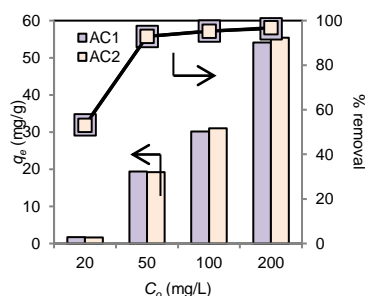
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Graphical abstract



Abstract

The present study was aimed to evaluate the adsorptive characteristics and microwave dielectric properties of activated carbons derived from oil palm empty fruit bunch (EFB) prepared by microwave-assisted potassium hydroxide activation at 300 W and 800 W for 5 min. The resultant products were characterized for dielectric properties, BET specific surface area, surface functional groups and methylene blue adsorption. Results show that the microwave-assisted activation yields activated carbons with surface area of 695 m²/g and 1339 m²/g for heating rates (microwave irradiation power) of 300 W and 800 W, respectively and both exhibit more than 95% removal of methylene blue at concentrations higher than 100 mg/L. The dielectric properties revealed a better propensity of activated carbons towards microwave heating especially at a higher heating rate due to high surface area, and possibly high moisture content and carbon content. A greater heating rate or microwave power could turn EFB into activated carbon with a higher surface area and excellent adsorptive properties for pollution abatement.

Keywords: Activated carbon, dielectric properties, dye adsorption, microwave heating, moisture content, penetration depth

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INTRODUCTION

There has been an increasing interest in the preparation of activated carbon using renewable and cheaper precursor as to compensate the high price of commercial activated carbon. In general, the agricultural wastes are perceived as the important raw material of activated carbon due to high carbon content, abundantly available, sustainable supply and cheap (Saygili *et al.*, 2015). A number of successful attempts to produce activated carbons from agro-wastes such as date stones (Abbas & Ahmed, 2016), date pits (Mahmoudi *et al.*, 2014), langsat empty fruit bunch (Foo & Hameed, 2012a; Njoku *et al.*, 2015), oil palm empty fruit bunch (Zaini & Shaid, 2016), pineapple peel (Foo & Hameed, 2012b), rice husks (Foo & Hameed, 2011a), sunflower seeds oil residue (Foo & Hameed, 2011b) and durian shells (Foo & Hameed, 2012c) have been documented in literature. The strategy to utilize the wastes as the feedstock assists in reducing the costs of waste handling and disposal, and further provides a solution to the environmental problems (Saygili *et al.*, 2015).

The preparation of activated carbon involves two steps, i.e., carbonization and activation. Carbonization aims to enrich the carbon content in order to create rudimentary pores as a result from the release of volatiles in char, while activation helps to develop the pore textures of activated carbon. The raw material conversion into activated carbon can be done through physical activation or chemical activation. In physical activation, the char is reacted with oxidizing gases such as CO₂ and/or steam at temperatures between 700°C and

1000°C (Zaini *et al.*, 2010). Chemical activation, on the other hand utilizes chemical reagent such as KOH, ZnCl₂ or H₃PO₄ that acts as dehydrating agent to promote pyrolytic decomposition and inhibit the formation of tar (Zaini *et al.*, 2009; Saygili *et al.*, 2015). The carbonization step can be skipped in chemical activation, as the impregnated-precursor is readily viable for activation. Chemical activation is more preferred over physical activation due to the fact that the former can be done at a lower temperature (400°C to 600°C) with a higher yield of activated carbon (Zaini *et al.*, 2009; Zaini *et al.*, 2013).

It is widely accepted that the use of conventional heating using furnace for activation is time-consuming and energy-intensive. Recent development in activated carbon preparation demonstrated the feasibility of using microwave in assisting the chemical activation (Foo & Hameed, 2011a-b; Foo & Hameed, 2012a-c). Interestingly, the physicochemical properties of activated carbons produced are similar to that obtained using conventional heating. Compared to conventional heating, microwave heating offers rapid volumetric and selective heating, high heating rates and product yield, and small equipment size and wastes released (Zaini & Kamaruddin, 2013). However, much of available published studies focusing only on the microwave-assisted production of activated carbons from various raw materials with lack of attention on the effect of heating rate, and the fundamentals of dielectric properties.

The dielectric properties (or permittivity, ϵ^*) is expressed as,

$$\epsilon^* = \epsilon' - j\epsilon'' \quad (1)$$

where ϵ' is the dielectric constant (real part of permittivity), that is a measure of how much energy from an external electric field is stored within a material through polarization mechanism, while ϵ'' is the loss factor (imaginary part of permittivity) that represents the ability of material to absorb and dissipate the electromagnetic energy into heat. The loss tangent ($\tan \delta$) is used to describe how efficient the electromagnetic energy stored within a material is converted into heat at a specific frequency and temperature. It is given as,

$$\tan \delta = \epsilon'' / \epsilon' \quad (2)$$

The dielectric properties aid in scrutinizing microwave heating and material interaction, predicting the heating rates, and describing the heating characteristics and behaviour of a material when subjected to a high-frequency electromagnetic field (Zaini & Kamaruddin, 2013). Penetration depth, D_p is used to determine how far the electromagnetic power can go inside a material, and it is given as,

$$D_p = \frac{\lambda_o \sqrt{\epsilon'}}{2\pi\epsilon''} \quad (3)$$

where λ_o is the free space microwave wavelength (for 2.45 GHz, $\lambda_o = 12.2$ cm). The volumetric heating of microwave could be less operative for a material with short penetration depth when only small portion of material thickness absorbs the microwaves. Consequently, the heating would not be uniform due to poor strength of electromagnetic wave at the material core that farther the penetration depth (Zaini & Kamaruddin, 2013; Alias & Zaini, 2015).

The dielectric properties of impregnated agro-wastes such as K_2CO_3 -impregnated cempedak peel (Alias *et al.*, 2017a), NaOH-impregnated cempedak peel (Alias *et al.*, 2017b), KOH-impregnated palm kernel shell (Zaini *et al.*, 2015a), and $ZnCl_2$ -impregnated palm kernel shell (Zaini *et al.*, 2015b) at different concentrations depicted a promising role of activating agents as microwave absorber in chemical activation. However, the interaction between heating rate (or microwave power) and dielectric properties of activated carbons is not well understood, and has to be established to enrich the present literature. Therefore, the objective of the present work is to evaluate the dielectric properties of empty fruit bunch-based activated carbons prepared using microwave power of 300 W and 800 W. Potassium hydroxide was used as the activating agent, while methylene blue dye was used to probe the performance of activated carbons. The changes and relationships between the activated carbons characteristics and dielectric properties were discussed to shed some light on factors that can provide positive effects in microwave-assisted activation at varying heating rates.

EXPERIMENTAL

Materials

Empty fruit bunch was obtained from Kluang plantation at Johor state of Malaysia. Methylene blue dye (molecular weight = 319.85 g/mol) was purchased from HmbG Chemicals. Potassium hydroxide was supplied by QRec. All chemicals are of analytical-grade reagents.

Microwave-assisted activation

Oil palm empty fruit bunch (EFB) was ground to an average size of 1 mm. EFB was impregnated with potassium hydroxide (KOH) at a solid weight ratio (KOH : EFB) of 1.5:1. The desired weight of KOH pellets was dissolved in distilled water, and then mixed with the desired weight of dried EFB. The mixture was homogeneously stirred and heated for 30 min, and then dried in an oven at 110°C for 24 h. Five grams of impregnated sample was placed in a crucible inside a Teflon container. The cap of the container was tightened, and the container was put in a microwave chamber. An exhaust tube was connected from the Teflon cap through the microwave cavity and directly to the fume hood. The microwave-assisted activation was performed using a 2.45 GHz multimode microwave applicator (Samsung Me711K), at microwave heating rates of 300 W and 800 W for 5 min. The resultant activated carbons were washed with distilled water and dried in an oven at 110°C for 24 h prior to use. The

activated carbons were labelled as AC1 and AC2, respectively. EFB-based char was also prepared for comparison by conventional heating, at 500°C for 2 h in a muffle furnace.

Characterization of activated carbons

A Fourier transform infrared spectroscopy combined with attenuated total reflectance (IRTracer-100, Shimadzu) was used to determine the surface functional groups of EFB-derived materials. The specific surface area of activated carbons were determined using a Pulse ChemiSorb 2705 (Micrometrics) at a liquid N_2 temperature of 77 K. The surface area was calculated using a single-point Brunauer-Emmett-Teller (BET) method.

The dielectric properties of EFB and its derived samples were measured at various microwave frequencies (1 – 6 GHz) using an open-ended coaxial probe technique. The measurement system consists of a coaxial probe (HP 85070D) attached to a Vector Network Analyzer (VNA model HP 8720B). The measurement of each sample was repeated at least three times to ensure good reproducibility of data.

Adsorption of methylene blue

The adsorption of methylene blue onto EFB-based activated carbons and char was performed using a bottle-point technique. The initial pH of methylene blue solution was not adjusted, and was measured as 5.9±0.2. Thirty mg of activated carbon was brought into intimate contact with 30 mL of methylene blue solution of different concentrations (1 – 200 mg/L). Next, the solution mixture was allowed to equilibrate for 72 h. After that, the residual concentration was measured using a visible spectrophotometer (Halo Vis-10) at a wavelength of 615 nm. The adsorption capacity, q_e (mg/g) was calculated as, $q_e = (C_o - C_e) \times V / m$, where C_o and C_e (mg/L) are the initial and equilibrium concentrations, respectively, V (L) is the volume of methylene blue solution, and m (g) is the weight of char.

RESULTS AND DISCUSSION

Characteristics of activated carbons

Table 1 shows the yield and BET surface area of EFB-based char and activated carbons. The carbonization (to produce char) and microwave-assisted activation processes have resulted in the decrease of yield. The yield of char is 75%, while that of activated carbons are 67% and 61%, respectively. The weight loss during material heating either by means of conventional heating or microwave is generally due to the release of volatile products with the increase of temperature, and this becomes more intensified under the presence of dehydrating agent (potassium hydroxide) which accelerates the elimination reaction and gasification of surface carbon. The decrease of yield in microwave-assisted potassium hydroxide activation is more pronounced at a higher microwave power of 800 W. This could be resulted from a greater heating rate as the microwave power increases which also infers that the activation was held at a higher temperature for the same 5 min retention.

Table 1 Yield and BET surface area of EFB-derived char and activated carbons.

Sample	Yield (%)	pH	Surface area (m ² /g)
EFB	100	7.8	28
Char	74.7	6.8	37
AC1	66.9	8.1	695
AC2	60.6	7.8	1339

From Table 1, the order of specific surface area is, EFB < char < AC1 < AC2. The surface area of char upon carbonization slightly increases due to the initiation of rudimentary pores. AC2 exhibits a higher surface area than AC1 as the heating rate enhances the development of new pores and active sites, hence increasing the pore volume and surface area at a higher temperature. The combined effect of volumetric and internal heating of microwave accelerates the

activation process in a rapid manner (5 min) (Hesas *et al.*, 2013). Generally, the specific surface area of AC1 and AC2 are within the acceptable range of commercial activated carbon (Hidayu *et al.*, 2013). AC2 demonstrates a greater surface area than ZnCl₂-activated EFB carbon (surface area of 866 m²/g) prepared by conventional heating (Zaini & Shaïd, 2016). In addition, the surface area of AC2 is comparable with that of other KOH-activated carbons prepared via conventional heating, but the ones produced through microwave-assisted activation as demonstrated in this work, offers a far better yield in a very short time (Shu Hui & Zaini, 2015).

Attempt has been made in this work to activate EFB under irradiated water environment at 800 W. The underlying theory of irradiated water was introduced in the previous works (Zaini *et al.*, 2014a-b). The mixture of EFB and KOH solution was added into an uncapped Beatson bottle inside a Teflon container. The subsequent activation procedures are the same as described earlier. After 5 min activation, the solution was partly dried, while the EFB still retained its light brown colour. Surprisingly, no value of surface area was recorded by the resultant product. It is suggested that the available pores of the pristine EFB are clogged by potassium salt during the microwave-drying, thus decreasing the surface area from 28 m²/g to null (Zaini & Shaïd, 2016). Furthermore, the role of irradiated water, with or without the presence of dehydrating agent, that was claimed to mimic that of steam activation could not be well justified. It is of common knowledge that water is an excellent microwave absorber that can turn microwaves into heat (Zaini *et al.*, 2015c). However, the heat that is propagated during the irradiation process (microwave drying) may not exceed the water boiling point (100°C). The so-called 'irradiated water environment' can only be made possible if the KOH solution bearing EFB is completely dried and sufficient time is allowed for the intercalation of potassium cations within the EFB matrix to create new pathways for the porous structure under the presence of microwaves (Zaini *et al.*, 2014c). In fact, the activation process is promoted due to the formation of microplasmas and hot spots on the dried solids surface that can reach up to several hundreds and thousands degree centigrade (Alias & Zaini, 2015; Zaini *et al.*, 2015c).

Fig. 1 shows the FTIR spectra of EFB, char, AC1 and AC2. The EFB spectrum exhibits several peaks located at 3310 cm⁻¹, 2920 cm⁻¹, 1610 cm⁻¹ and 1040 cm⁻¹, that correspond to the characteristics of O-H (hydroxyl), -CH₂ (alkyl), C-O-C (ester, ether and phenol) and C-O (anhydrides). Three major functional groups in EFB are hydroxyl groups, carbonyl groups and carboxyl groups [14]. On the other hand, the char spectrum displays a peak with weak band at 3117 cm⁻¹ which is attributed to -CH₂ (alkyl). A number of peaks formerly seen in EFB spectrum are disappeared due to the elimination of volatiles and heat-sensitive functional groups at 500°C (Zaini *et al.*, 2016).

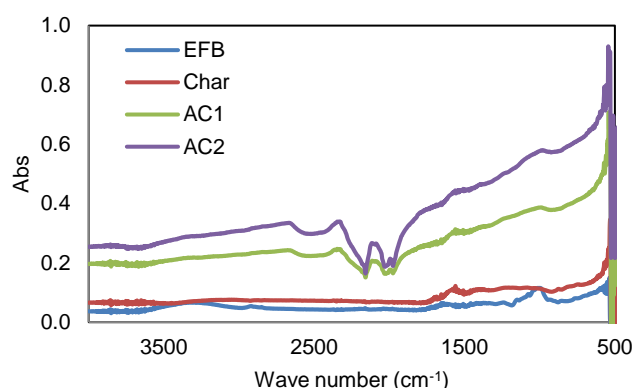


Fig. 1 FTIR spectra of EFB, char, AC1 and AC2.

From Fig. 1, AC1 and AC2 display similar FTIR spectra, indicating identical quality of surface functional groups. The absorption bands at 2660 cm⁻¹ and 2330 cm⁻¹, 1570 cm⁻¹ and 980 cm⁻¹ are the characteristics of O-H (carboxylic acids), C-H (alkyls) and C-O (anhydrides), respectively. Yet, AC2 exhibits peaks with greater intensity as compared to AC1, which also suggests a greater quantity of functional groups in AC2.

Dielectric properties and penetration depth of activated carbons

Fig. 2 shows the profiles of dielectric properties of EFB, char, AC1 and AC2 at different microwave frequencies.

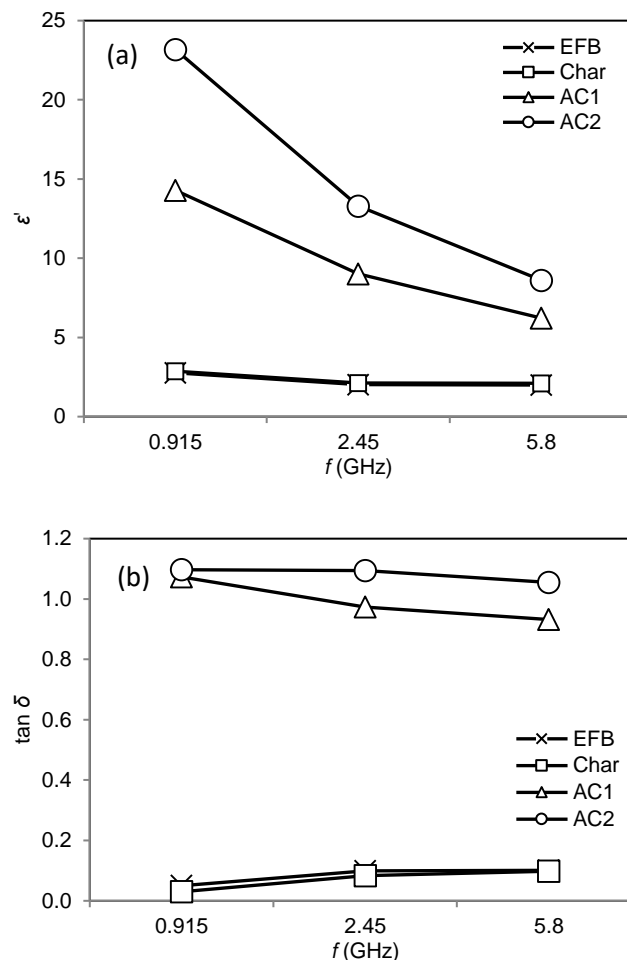


Fig. 2 Profiles of dielectric properties of EFB and its derived materials (a) dielectric constant, and (b) loss tangent.

From Fig. 2(a), AC2 displays a higher dielectric constant (ϵ'), followed by AC1, char and EFB, where the profiles of EFB and char are fairly overlapped. This pattern is true with the increase of microwave frequencies. Also, for all materials studied, the ϵ' decreased with increasing frequency as a result of polarization effect due to varying electric field (Salema *et al.*, 2013). The presence of moisture in AC1 and AC2 could be the main reason for a higher ϵ' at any frequency despite the rapidly diminished conductive effect of microwave heating (Alias & Zaini, 2015). AC1 and AC2 possess a higher surface area compared to char and EFB. A greater surface area as a result of a higher heating rate of microwave-assisted KOH activation also implies a larger pore volume to accommodate naturally physisorbed water from the surrounding. The moisture content offers the flexibility of the free ions in the material to move and contribute to high loss ($\tan \delta$) or conductivity loss (Omar *et al.*, 2011). Other factors such as ash content, carbon content and functional groups could also offer positive effects on ϵ' (Alias & Zaini, 2015; Alias *et al.*, 2017b). Hence, ϵ' is a complex function that varies especially when there is a change in the intrinsic properties of the material during microwave-assisted activation. The increase of carbon content in the form of graphitic structure upon activation indeed plays an important role in the dielectric properties (through orientation polarization) because of the presence of aromatic rings. The delocalized π -electrons can move freely in a broad region and might create ionization to the surrounding (Salema *et al.*, 2013; Zaini & Kamaruddin, 2013). On the contrary, the EFB-based char exhibits an

inferior dielectric properties probably due to the under-developed graphitic structure even though the carbon content increased upon carbonization (Zaini *et al.*, 2015c).

The profiles of loss tangent ($\tan \delta$) are shown in Fig. 2(b). AC1 and AC2 exhibit higher values of $\tan \delta$ compared to char and EFB, suggesting that they are more efficient to be heated under microwaves. In general, sample with high $\tan \delta$ normally has a better energy absorption properties, energy storage characteristic and a higher heating rate (Zaini *et al.*, 2015a). Also, it shows the underlying roles of KOH to promote surface area in EFB-based activated carbons, and inherent moisture content in enhancing the efficiency of microwave heating. Water is known as natural polar and prominent microwave absorber, and has been used as a benchmark for other dielectric materials (Sosa-Morales *et al.*, 2010). However, the pattern of $\tan \delta$ for all samples are inconsistent with increasing frequency due to the decrease in interfacial polarization (Zaini *et al.*, 2015a) and/or gradual decrease in the dipole movement that produces heat within the material via molecular polarization (Salema *et al.*, 2013; Zaini *et al.*, 2015b).

EFB can be heated under microwave but may not be as rapid as that of high loss material (absorber). However, once it is transformed into rich graphitic carbon structure (activated carbon), the $\tan \delta$ gets much higher. The EFB-based activated carbons are good microwave absorbers for microwave-assisted activation due to high values of $\tan \delta$ ($\tan \delta > 0.1$). On the other hand, EFB and char are classified under low microwave absorbing dielectric materials ($\tan \delta < 0.1$). Accordingly, these samples may not be suitable for microwave-assisted activation, unless an impedance matching (tuning) system between the load (sample) and microwave power source to ensure maximum transfer of power is installed (Zaini & Kamaruddin, 2013). Consequently, the selection of microwave frequency for chemical activation should be carefully selected to improve the effectiveness of the activation process. As such, multiple adjustment of frequency during the course of activation can be proposed.

Fig. 3 shows the penetration depth of EFB, char, AC1 and AC2 at different microwave frequencies. Penetration depth, D_p , is defined as the depth into material where the power reduced to $\sim 1/e$ of the original intensity, and can be used to evaluate the microwave heating uniformity. From Fig. 3, AC1 and AC2 show a smaller D_p probably due to high moisture content that trapped within the pore textures. The microwave heating of the material with high moisture content may be localized only on the surface due to short D_p . This is where the hotspots are mainly centered in heating the moisture (Motasemi *et al.*, 2014).

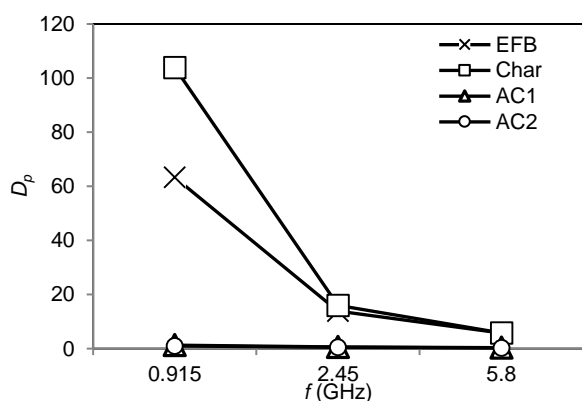


Fig. 3 Penetration depth of EFB and EFB-derived samples.

A non-uniform heating can also happen if the size of the sample to be heated is larger than the D_p of the microwave. Homogenous heating may not take place as the energy is absorbed only near the surface of the material (Sait & Salema, 2015). Nevertheless, the D_p for AC1 and AC2 are longer than their particle size, which infers that the uniform heating can take place. On the other hand, a high D_p as depicted by char indicates that the material is somewhat transparent to the microwaves with low or no microwave absorption. However, it

enables even the thicker bed of char to be processed due to its high penetration depth. The penetration depth also provides the preliminary knowledge and understanding on the size of the target material in the manufacture of activated carbon using microwaves. It is shown in Fig. 3 that, with increasing frequency, the D_p of all the samples decreases. This might be due to the difference of wavelength of the frequencies. For instance, a longer wavelength of 32.75 cm (0.912 GHz) penetrates more than a shorter wavelength of 12.24 cm (2.450 GHz) (Sait & Salema, 2015).

Methylene blue adsorption

Fig. 4 represents the methylene blue removal by EFB-based materials. In general, the removal capacity of methylene blue increased with increasing concentration, indicating a favourable adsorption process. As the concentration increases, the driving force for the dye molecules to lodge on the pores and active sites also increases. AC1 and AC2 show a higher capacity of 1.6 mg/g than char that yields only 0.78 mg/g at $C_o = 20$ mg/L. In addition, AC1 and AC2 display an increasing pattern of dye percentage removal with a comparable performance of 53% at $C_o = 20$ mg/L. On the other hand, char recorded a higher performance of 35% at $C_o = 10$ mg/L, after which the percentage removal began to drop, indicating the maximum capacity or saturation point has been reached.

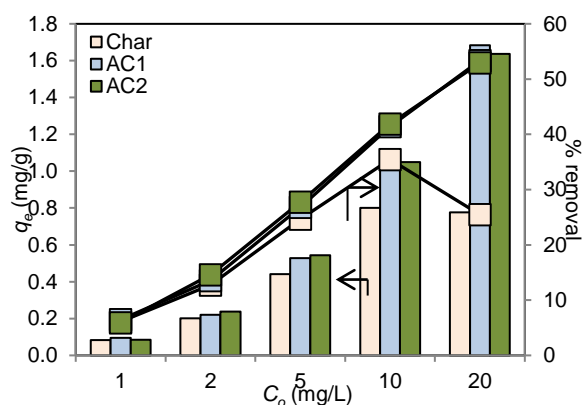


Fig. 4 Removal of methylene blue by EFB-based materials.

Fig. 5 shows the adsorption of methylene blue onto AC1 and AC2 at higher concentrations. AC1 and AC2 display a similar pattern of increasing adsorption capacity with increasing concentration. The adsorption capacity of 55 mg/g was recorded at $C_o = 200$ mg/L. Furthermore, the percentage removal shows an exceptional increase of more than 95% at concentrations greater than 100 mg/L. In an earlier work, Mohd.-Nasir *et al.* (2015) reported a maximum (saturation) capacity of 32 mg/g of methylene blue adsorption onto untreated EFB at $C_o = 237$ mg/L, which accounts for 13.5% removal. It implies that the EFB-based activated carbons are promising adsorbent for dye adsorption as they can still accommodate more dye molecules beyond the concentrations studied.

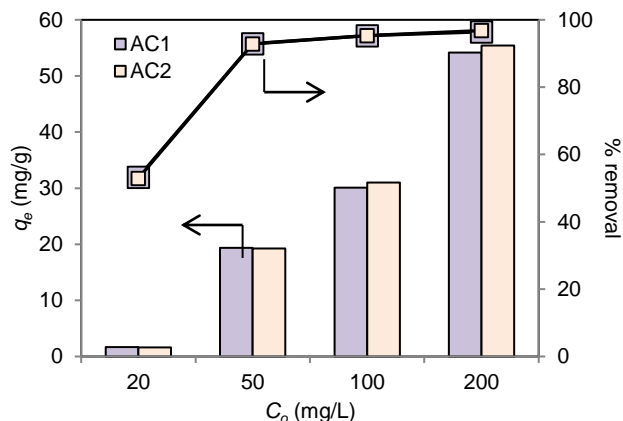


Fig. 5 Removal of methylene blue by EFB-based activated carbons at higher concentrations.

CONCLUSION

Oil palm empty fruit bunch was converted into activated carbons by microwave-assisted KOH activation at heating rates (microwave irradiation power) of 300 W and 800 W. Activation at 800 W produces activated carbon with 61% yield and a surface area of 1339 m²/g. Activated carbons demonstrate higher dielectric constant and loss tangent due to their high surface area and possibly high moisture content, hence possess better propensity to be heated under microwaves. Nevertheless, an impedance matching system and multiple adjustment of frequency can be proposed in microwave-assisted activation to enable maximum transfer of power and uniformity of the heating process. Activated carbons produced in this work display a comparable performance of more than 95% dye removal at $C_0 = 200$ mg/L. It shows a promising application of EFB-based activated carbons prepared by microwave-assisted activation for dye removal from wastewater.

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