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Parametric and Adsorption Kinetic Studies of Reactive Black 5 Removal from Textile Simulated Wastewater Using Oil Palm (*Elais guineensis*) Empty Fruit Bunch

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ABSTRACT

The potential of using Empty Fruit Bunch (EFB), an agrowaste material, as a low-cost biosorbent for the removal of Reactive Black 5 (RB5) from aqueous solution was investigated in this study. The influences of solution pH, contact time, initial concentration and biosorbent dosage were studied in batch experiments at room temperature. Adsorption equilibrium was achieved after 30 min of agitation. The maximum adsorption uptake of RB5 dye occurred at pH 2, resulting in a rapid adsorption (more than 50% RB5 uptake) for the first 5 min of contact. Lower solution pH values showed better adsorption because the dye molecules tend to adsorb on positively charged adsorbent sites. Furthermore, higher biosorbent dosages increased the dye uptake by up to 90% due to the availability of more active adsorption sites. Both the pseudo-second-order kinetic model and the Langmuir model indicated that monolayer coverage on the adsorbent was dominant and that chemisorption was the rate-determining step.

Key words: EFB, oil palm, reactive black 5, biosorption

INTRODUCTION

Water occupies approximately two-thirds of the Earth's surface; more than 70% of the Earth's surface is covered by water. However, of the available water on Earth, only 2.5% is freshwater. Freshwater is characterized as having low concentrations of dissolved salts and other solids. Of this freshwater, nearly 70% is frozen in the icecaps of Antarctica and Greenland. Most of the remaining fresh water is located in deep underground aquifers as groundwater or exists as soil moisture that is unfit for human use. Thus, only 1% of the existing freshwater is directly accessible for human use (Gleick, 2000).

Water is of great importance to us and water pollution has become a major concern. One important water pollutant is the

dye that is used by industries, such as printing, paper-making, plastic, coating, food and textile industries. These industries use dyes and pigments to color their products (Elizalde-Gonzalez *et al.*, 2007). The discharge of wastewater from these industries is worrisome. It has been reported that 10-15% of the dye that is used by industries is released to the environment after the dyeing process (Chowdhury *et al.*, 2011).

The degree of water pollution is determined by the Biochemical Oxygen Demand (BOD), the chemical oxygen demand (COD), the suspended solid content, malodor, toxicity and color (Ali and Sreerishnan, 2001). Wastewater that is contaminated with dyes is colored with reduced light penetration, which affects aquatic life and increases the toxicity of the wastewater, making it unfit for human use

(El-Geundi, 1997). In Malaysia, the textile industry is ever growing. Currently, it is reported that the textile industry is contributing about 22% of the total volume of industrial wastewater generated in the country. There are over 100,000 commercially available dyes with a production of over 7×10^5 Mt year⁻¹ (Hameed *et al.*, 2008). Most dyes and color effluents have toxic carcinogenic and mutagenic effects that affect the human health and the environment. Thus, the removal of these dyes has become a major concern and significant effort has been made to search for a proper and more efficient method for treating dye-containing effluents.

The primary functions of dyes are to provide the chosen raw material with a coating of long-lasting color. Therefore, dyes are designed to be resistant to soap, water, light and oxidizing agents. Furthermore, synthetic dyes have complex aromatic structures that increase their stability and resistance to biodegradation. Hence, finding an efficient dye-removal method for treating dye-containing effluents is challenging (Khataee *et al.*, 2013; Akar *et al.*, 2009).

The conventional treatment of dye-containing effluents may include combinations of physical, chemical or biological processes. These processes include coagulation/flocculation, advanced oxidation, ozonation, membrane filtration, electroflotation, electrokinetic coagulation, electrochemical destruction, ion exchange, irradiation, precipitation and biological treatment. However, these processes also have several disadvantages. The most significant disadvantages include high reagent concentrations, high energy requirements, low selectivity, high capital, high operational costs and the generation of secondary wastes that are difficult to remove or degrade (El Haddad *et al.*, 2014). An effective alternative process for treating dye-containing effluents is by adsorption. Currently, a popular and widely used adsorption technique is the use of activated carbon as the adsorbent. This method is popular due to the high adsorption capacity, surface area, high surface reactivity and microporous structure of adsorbents. However, this method also has problems, including high operating costs and problems with regeneration (Akar *et al.*, 2009).

Consequently, interest in using inexpensive and widely available materials for the adsorption of dyes has increased in recent years. A considerable number of studies have been conducted to search for inexpensive adsorbents that are abundant in nature or formed as by-products or waste materials from another industry (Khalaf, 2008). Different agricultural biomasses, such as tree ferns, peat and rice husk, peanut hulls, sugarcane dust, apple pomace and wheat straw, bark, palm kernel fiber, banana peel and orange peel, coir pith, linseed cake and sawdust have been evaluated for the removal of different types of dyes. Some of the biosorbents that have shown promising results have been compared to the adsorption capabilities of EFB is as shown in Table 1. However, the search for a more effective and economical biosorbent material continues (Akar *et al.*, 2009).

The objectives of this study are to determine the suitability of *Elaeis guineensis* (Oil palm) Empty Fruit Bunch (EFB) as a biosorbent for the removal of Reactive Black 5 (RB5) from textile effluents; to study the adsorption kinetics and isothermal behaviors of EFB in the removal of RB5 from textile effluents and to conduct a parametric study of biosorption relative to the pollution concentration, adsorbent dosage and solution pH.

MATERIALS AND METHODS

Sample preparation: The EFB that was used for this study was obtained from a local oil palm plantation in Beaufort, Sabah, Malaysia. The EFB was washed several times in distilled water to remove unwanted impurities, such as dirt, pesticide residue and other inorganic compounds. After the cleaning procedure, the EFB was thoroughly dried and cut into small pieces of approximately 2-3 cm. Next, the small pieces were placed in the oven overnight to dry at a temperature of 130°C. This step was conducted to reduce the moisture content in the EFB. Next, the EFB were ground, passed through a mechanical sieve with a mesh size of 2-3 mm and stored in an airtight container. Figure 1a-d show the EFB preparation procedures of washing with distilled water, drying and sieving, respectively.

Table 1: Other biosorbents studied for dye removal capabilities (Yagub *et al.*, 2014)

Adsorbents	Dye name	pH range	Removal range (%)
Modified-Alumina	Crystal violet	2.6-10.8	20-80
Activated-clay	Methylene blue	2-9	60-95
Activated-carbon	Methylene blue	2-11	Increase
Kaolin	Crystal violet	2-7	65-95
Activated-rice husk	Acid Yellow 36	2-9	80-45
Bentonite	Acid Blue 193	1.5-11	Decrease
Fly ash	Methylene blue	2-8	36-45
Pine leaves	Methylene blue	2-11	20-80
Fe ₂ O ₃	Acid red 27	1.5-10.5	98-27
Pine cone	Congo red	3.55-10.95	60.5-5.75
Pine cone	Methylene blue	3.47-7.28	63.83-94.82
Tobacco Stem Ash	Methylene blue	2.08-7.93	60-81
Modified sawdust	Methylene blue	2-11	Increase
Oil Palm EFB	Reactive Black 5	2-11	55-6



Fig. 1(a-d): (a) Obtained empty fruit bunch, (b) Washing with distilled water, (c) Drying in an oven at 130°C and (d) Sieving process

Preparation of the RB5 stock solution: RB5 was purchased from Aldrich and was used without further treatment.

Initial dye concentration: First, 50 mL of RB5 solutions with initial concentrations (C_i) of 5, 10, 15, 20 and 25 mg L⁻¹ were prepared in a series of 250 mL Erlenmeyer flasks. Then, 0.1 g of EFB was added to each erlenmeyer flask and the flasks were then placed in an orbital shaker and shaken for 60 min at a constant speed of 150 rpm until equilibrium was achieved. Next, aqueous samples were taken from the solutions using a syringe membrane filter and the concentrations were analyzed using a UV-Vis spectrophotometer. The final concentration (C_e) was measured and the amount of adsorbed dye (q_i in mg g⁻¹) was calculated using the following equation:

$$q_i = (C_i - C_e) \frac{V}{W} \quad (1)$$

Where:

- $C_i = C_i$ (mg L⁻¹) is the initial dye concentration
- $C_e = C_e$ (mg L⁻¹) is the final dye concentration
- $V = V$ is the volume of the dye solution (L)
- $W = W$ is the mass of the adsorbent that was used (g)

All the experiments were conducted at room temperature and repeated in triplicates, with the average calculated and tabulated into graphs.

Biosorbent dosage: The initial RB5 concentration was set as a constant. Then, the effects of the amount of biosorbent were studied over a concentration range of 0.4-4.0 g dm⁻³.

Solution pH: To observe the effects of the solution pH on the uptake by adsorption, initial solution pH values of 2-11 were used. The acidic and basic environments of the solutions were adjusted by adding 0.1 M HCl and NaOH, respectively. The initial dye concentration (C_i) was fixed at 6.0 mg L⁻¹, with a biosorbent dosage of 0.002 g mL⁻¹. The solution temperature was maintained at 30°C. The final dye concentration (C_e) was measured and the percent of dye removal was calculated using the following equation:

$$\text{Dye removal(\%)} = \frac{C_i - C_e}{C_i} \times 100 \quad (2)$$

Where:

- $C_i =$ Initial concentration (mg L⁻¹)
- $C_e =$ Final concentration (mg L⁻¹)

Adsorption isotherm

Langmuir isotherm: Langmuir (1918) defined the Langmuir isotherm equation as follows:

$$q_e = \frac{q_{\max} K_L C_e}{1 + K_L C_e} \quad (3)$$

Where:

- q_e = Amount of adsorbate adsorbed at equilibrium (mg g^{-1})
- q_{max} = Maximum monolayer adsorption capacity of the adsorbent (mg g^{-1})
- C_e = Equilibrium concentration of the adsorbate (mg L^{-1})
- K_L = Langmuir adsorption constant related to the free energy adsorption (L mg^{-1})

This simplest physically plausible isotherm is based on the three following assumptions (Alrozi *et al.*, 2012):

- Maximum adsorption proceeds when the surface is covered by a monolayer of the adsorbate (monolayer adsorption)
- All surface sites are equivalent and can accommodate, at most, one adsorbed atom (the solid surface is uniform)
- The ability of a molecule to adsorb at a given site is independent of the occupation of neighboring sites (no intermolecular interactions are present)

Freundlich isotherm: Freundlich (1906) defined the Freundlich isotherm equation as follows:

$$\frac{x}{m} = kC^{1/n} \quad (4)$$

Where:

- x = Mass of the adsorbate adsorbed (mg)
- m = Mass of the adsorbent (g)
- p = Equilibrium pressure of the adsorbate (mg L^{-1})
- c = Equilibrium concentration of the adsorbate in solution (mg L^{-1})
- K, n = Constants for a given adsorbate and adsorbent at a particular temperature

Adsorption kinetic studies: Batch adsorption kinetic experiments that were identical to the equilibrium experiments were conducted. Aqueous samples were collected at preset time intervals and the dye concentrations were measured. The adsorption kinetic experiments were performed by placing 200 mL of RB5 solutions with different initial concentrations of 5-25 mg L^{-1} in contact with 0.1-0.5 g of EFB powder in a 250 mL Erlenmeyer flask at room temperature. The samples were extracted at fixed time intervals, filtered and analyzed.

RESULTS AND DISCUSSION

FTIR analysis of the EFB: The FTIR spectra of the EFB before and after adsorption are shown in Fig. 2. In the obtained spectra, several functional groups can be identified on the surface of the EFB sample. A comparison of the spectra of the EFB samples before and after adsorption and at different pH

levels was made. From this comparison, it was clear that a few significant peaks became more intense after adsorption and a few minor peaks shifted or disappeared.

The characteristic wide peak from 3300-3420 cm^{-1} shown in Fig. 2 resulted from the overlap of the O-H and N-H stretching bands (Thirugnanasambandham *et al.*, 2013). Before adsorption, the peak occurred at 3420 cm^{-1} . However, after adsorption, this peak shifted to approximately 3350 cm^{-1} . The peak at 2982 cm^{-1} that occurred before adsorption was attributed to the C-H group, which shifted to approximately 2880 cm^{-1} after adsorption (Pirbazari *et al.*, 2014). Another peak was observed at 1641 cm^{-1} before adsorption and was attributed to the amide II; N-H bending and C = O stretching of acetyl groups caused shifts to 1623 and 1607 cm^{-1} , respectively (Pirbazari *et al.*, 2014). Finally, the peak at 1050 cm^{-1} was greatly intensified after adsorption. This peak resulted from the S = O group.

After the dye adsorption process, a shift in the positions of the O-H, N-H and C = O group peaks occurred. This shift indicated that the RB5 molecules forms temporary chemical bonds with the O-H, N-H and C = O groups of the EFB biosorbent. The changes that were observed in the FTIR spectra confirmed that these functional groups were involved on the surface of the EFB during and after the adsorption process.

Scanning Electron Microscopy (SEM): The SEM images of the EFB are shown in Fig. 3a-b. The SEM image was taken at 1000x magnification using a SEM EVO 10 MA. Figure 3a shows the ridges and Fig. 3b shows the pores that were observed on the surface of the EFB. Micropores can be detected inside the ridges and based on this scale, the pore size was estimated to be between 10 and 15 μm . However, the micropores are approximately 2 μm . The main difference that can be observed between Fig. 3a-b was the sizes of the pores that are found in different areas of the EFB. A small amount of debris (EFB fiber) can be observed on the surface of the EFB before adsorption.

Effects of solution pH on the adsorption process: Figure 4a shows the effect of solution pH on the adsorption of RB5. From Fig. 4a, it can be observed that the adsorption of EFB was favorable at lower pH values. At pH 2, the percent of removal at equilibrium was 55.06%, whereas at pH 11, the percent of removal at equilibrium was only 6.34%. The solution pH greatly affects the adsorption process, whereby the nature of the dye (cationic or anionic) and the charge of adsorbents play a key role (Anastopoulos and Kyzas, 2014). The adsorption of the RB5 dye by the EFB adsorbent is unfavorable under higher pH conditions because the RB5 dye is an anionic dye. Thus, the RB5 dye tends to be adsorbed on positively charged adsorbent sites. However, under higher pH conditions, the number of positively charged adsorbent surface

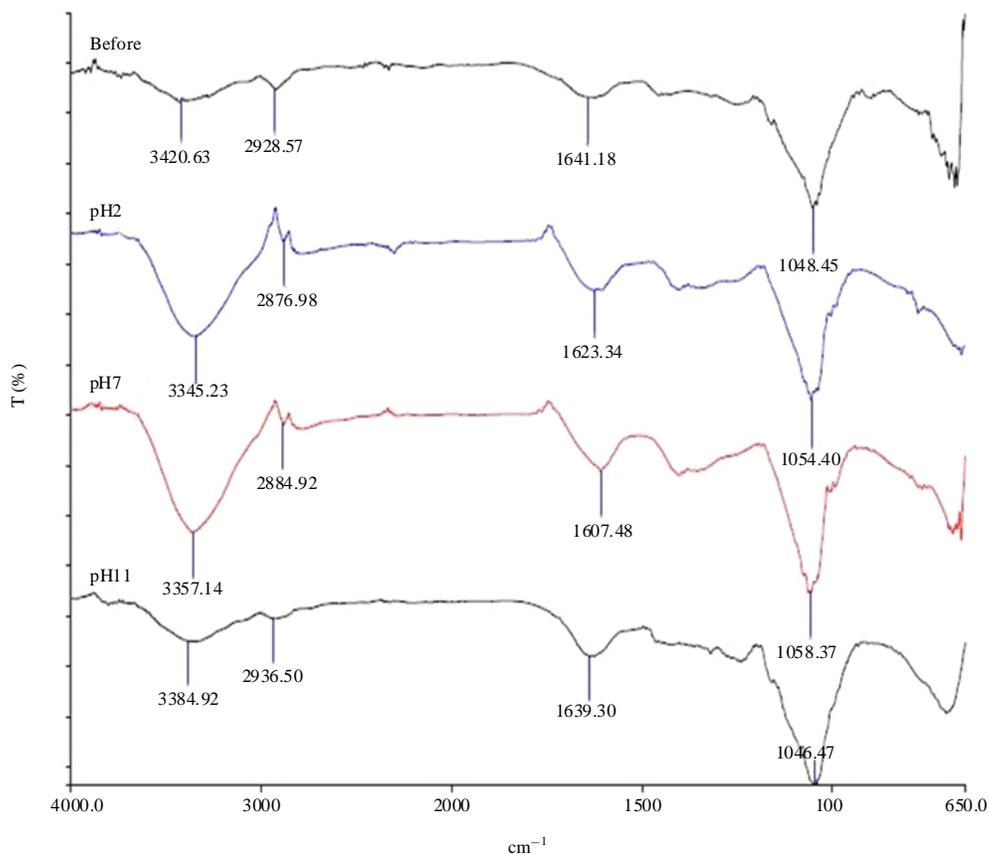


Fig. 2: FTIR spectra of the EFB before and after RB5 adsorption at different pH levels



Fig. 3(a-b): SEM images of the different parts of the EFB which highlights the different pore sizes found on the surface

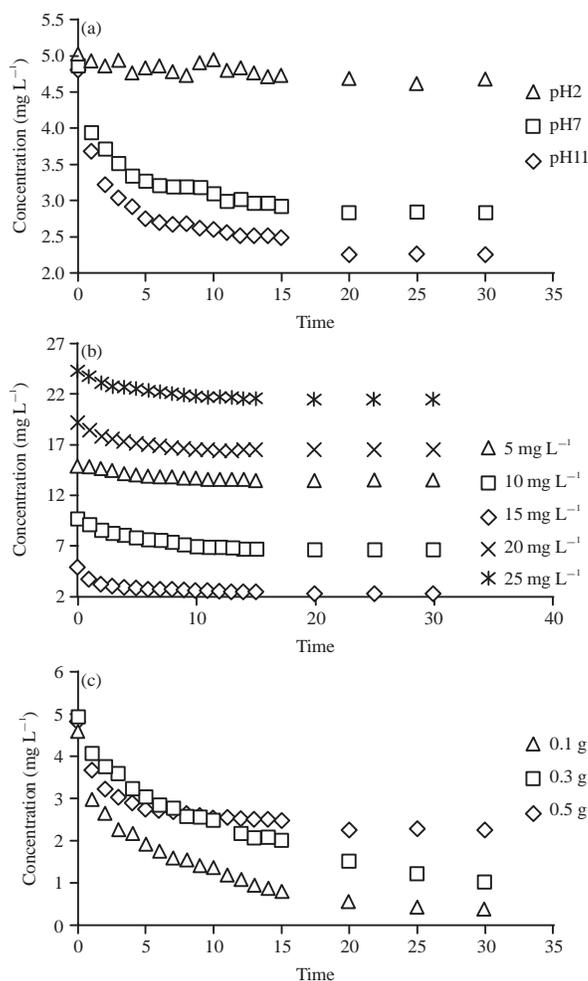


Fig. 4(a-c): (a) A plot of adsorbed RB5 versus time at different (a) pH values using empty fruit bunch, (b) Initial concentrations using EFB and (c) EFB biosorbent doses

sites decreases and the number of negatively charged surface sites increases. Furthermore, this condition is unfavorable for the adsorption of the negatively charged RB5 dye anions due to electrostatic repulsion (REF). In the presence of excess OH⁻ ions, which compete with RB5 dye anions for the adsorption sites on the EFB, the adsorption of the RB5 dye anions at basic pH may also decrease. As the pH decreases, the percent of RB5 dye removal increased significantly due to significant changes in polarity; the number of negatively charged active sites increased and more RB5 anions were able to bond to the adsorption active sites of the EFB.

Effects of initial dye concentrations on the adsorption process: In Fig. 4b, the effects of the initial RB5 dye concentrations on the dye uptake by the adsorbents in acidic conditions (pH level = 2) can be observed. In addition, the RB5 dye adsorption rate was fast during the initial stage and slowed down until equilibrium was

reached after approximately 15 min, when nearly constant values were recorded. In the equilibrium state, none or a limited amount of dye was removed from the dye solution by the biosorbent. This phenomenon likely occurred because a large number of the biosorbent surface sites are available for the adsorption of RB5 molecules during the initial stage. However, the number of available sites decreases with time because the sites are occupied by the RB5 molecules. Thus, the number of sites for the RB5 molecules decreases and the remaining surface sites are not easily occupied due to the repulsion between the solute molecules of the solid and bulk phases.

An increase in the initial dye concentration leads to an increase in the adsorption capacity of the biosorbent for the dye because the driving force of the concentration gradient increases. The adsorption capacity at equilibrium increases from 2.2471-21.4529 mg L⁻¹ as the initial RB5 concentration increases from 5.0-25.0 mg L⁻¹.

Effects of biosorbent dosage on the adsorption process:

Figure 4c shows the effects of biosorbent dosage on RB5 dye uptake by the adsorbent under acidic conditions (pH levels = 2) and a constant concentration of 5 mg L⁻¹. As observed in Fig. 4c, the percent removal of RB5 increases as the biosorbent dosage increases from 0.1-0.5 g. The adsorption is greater when a higher biosorbent dosage is used. The percent removal of the RB5 dye increased from 55.06-92.18%, when the biosorbent dosage was increased from 0.1-0.5 g. The RB5 dye adsorbed decreased when the number of biosorbent surface sites decreased. However, when a higher biosorbent dose was used, the number of biosorbent surface sites significantly increased. This increase not only allowed more RB5 dye molecules to attach themselves to the biosorbent surface sites but also effectively reduced the repulsion between the solute molecules of the solid and bulk phases.

Adsorption isotherm: The adsorption isotherm for RB5 on EFB was determined and summarized as shown in Table 2. A correlation coefficient of 0.9999 was obtained for the Langmuir model and 0.0009 was obtained for the Freundlich model. This proves that the adsorption follows the Langmuir adsorption isotherm, which indicates monolayer adsorption. The Langmuir model is based on the assumption that the adsorption energy is constant and independent of the surface coverage. In this case, the adsorption occurs on localized sites with no interactions between the adsorbate molecules. In addition, the maximum adsorption occurs when the surface is covered by a monolayer of the adsorbate (Amri *et al.*, 2012).

BET isotherms: A graph of volume (cc g⁻¹) against relative pressure (P/Po) was used to determine the BET isotherm type (Fig. 5c). The BET isotherm exhibited a Type III adsorption isotherm, which indicates the absence of zero gradient portions. This result indicates the absence of single-layer adsorption. The nitrogen gas uptake at the beginning gradually increased with the absence of an initial peak. Thus, the adsorption mechanism transitioned from single molecule adsorption to multi-layered adsorption in the EFB. However, the gas uptake sharply increased at the end of the graph, which indicated that capillary condensation occurred.

This finding contradicts the Langmuir isotherm results, which indicated monolayer adsorption. The contrasting findings potentially resulted from the heterogeneous nature of the EFB surface. A cooperative multi layer adsorption induced by small van der Waals perturbations at some distance from the surface may have existed (Halsey, 1948).

From the BET isotherm, the surface characteristics are determined and recorded in Table 3.

Adsorption kinetics: Studies regarding the adsorption kinetics of wastewater treatment are important because they not only

provide valuable insights into the reaction pathways and mechanisms of sorption reactions but also describe the solute uptake rate that controls the residence time of sorbate uptake at the solid-solution interface (Ho and McKay, 1998). To analyze the adsorption kinetics of RB5 onto EFB, two kinetic models are applied to analyze the experimental data that were obtained. A pseudo-second-order kinetic model closely fit the observed adsorption kinetics.

Pseudo-second-order kinetic model: The pseudo second order model is based on the assumption that the rate-limiting step may be chemical sorption, which involves valence forces through the sharing or exchange of electrons (Ho and McKay, 1998). The pseudo-second order kinetic rate equation was expressed as follows:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \tag{5}$$

Where:

k₂ (g/mg.h) = The second-order adsorption rate constant

To determine the suitability of the pseudo-second-order kinetic model for describing the experimental data, a graph of t/q_t against t was plotted and is shown in Fig. 6.

Based on Fig. 6, the coefficients (R²) for the pseudo-second-order model were closer to unity (R²>0.9797), which indicates a better fit with the pseudo-second-order model. The values of the correlation coefficients (R²) for the pseudo-second-order equation were 0.9797<R²<0.9951. Thus, the adsorption process of the RB5 dye using EFB as a biosorbent is described by a pseudo-second-order kinetic model. This was consistent with a recent review by Anastopoulos and Kyzas (2014) whereby most agricultural adsorbents for dye adsorption follows both Langmuir and

Table 2: Adsorption isotherm parameters

Isotherm	Isotherm constants
Langmuir isotherm	
Q _o (mg g ⁻¹)	7.34210
K _L (L mg ⁻¹)	1.50500
R _L	0.02590
R ²	0.999900
Freundlich isotherm	
K _f	8.24520
n	13.7174
R ²	0.000900

Table 3: Surface characteristics of the EFB

Surface characteristics	Values
Total surface area (S _{BET})	2.1790 m ² g ⁻¹
Total pore volume (V _T)	0.01502 cc g ⁻¹
Average pore diameter	275.7 Å
Single point surface area	1.875 m ² g ⁻¹
Langmuir surface area	5.0720 m ² g ⁻¹

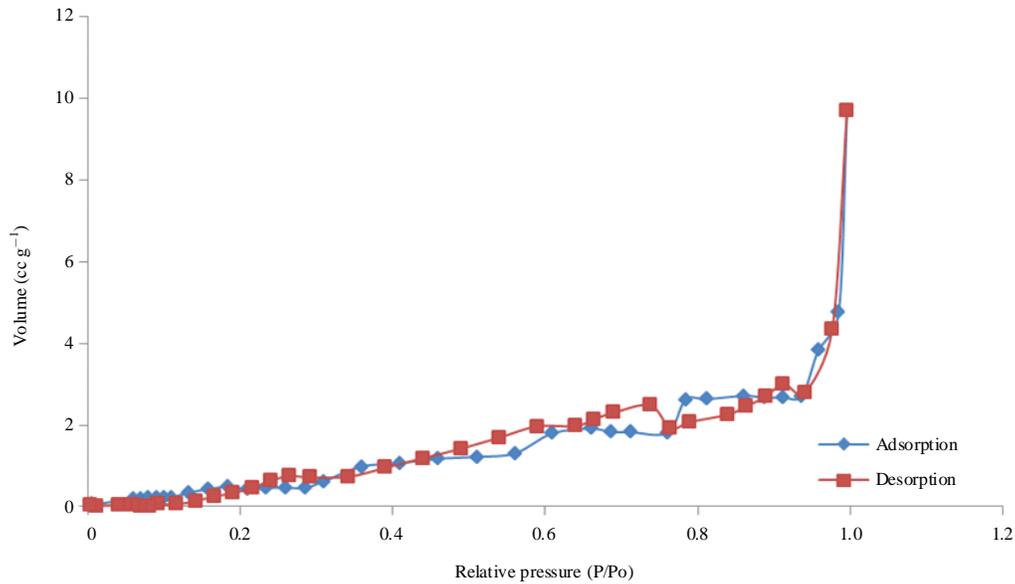


Fig. 5: N₂ adsorption-desorption graph of the EFB

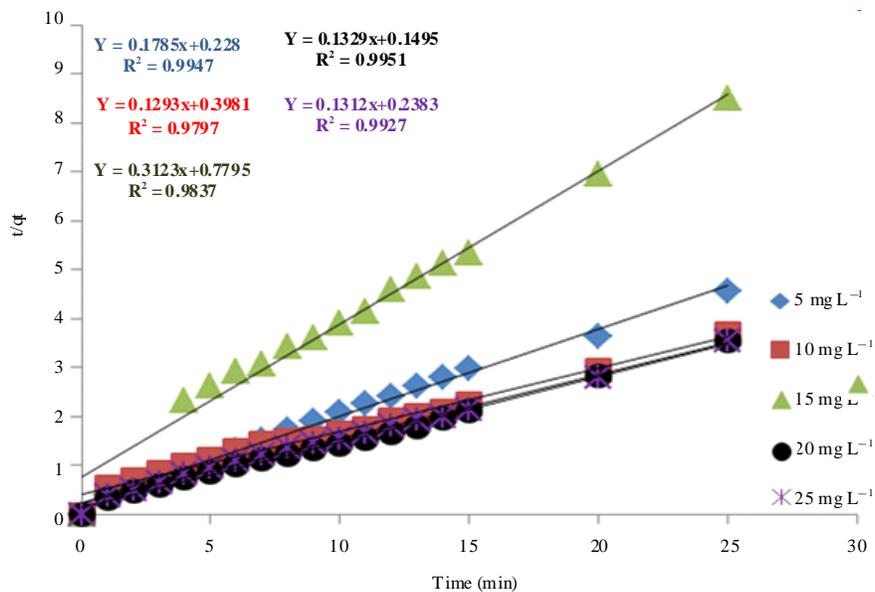


Fig. 6: Pseudo-second order kinetic plots for the biosorption of RB5 onto EFB

Pseudo-Second order models (Anastopoulos and Kyzas, 2014). This finding suggests that chemisorption is most likely the rate-limiting step that controls the overall adsorption process.

CONCLUSIONS

The use of EFB as an adsorbent is advantageous to oil palm rich countries like Malaysia as it allows an opportunity for agriwaste disposal and at the same time provides a waste to wealth possibility of utilizing this advantage. The overall

result demonstrates that EFB can be alternative adsorbent for the removal of dye from aqueous solutions. The spent biosorbent can be ashed and used as fertilizer as it a plant residue. This will in turn also destroy the adsorbate, reducing the need for any further treatment and cost.

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