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Adsroption of Bismarck Brown R Dyes Using Mesoporous Silica MCM-48

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Abstract

Bismarck Brown R (BBR) dye has been adsorption using mesoporous silica (MCM-48). We synthesized the adsorbent using Ludox HS-40 as a silica source and surfactants of Cetyl Trimethylammonium Bromide (CTAB) and Triton X-100. The characterization of MCM-48 was performed using Fourier transform infrared (FTIR), X-ray diffraction (XRD), and scanning electron microscopy (SEM). Various contact times were used to study the adsorption kinetics, and concentrations were used to study the adsorption isotherm. The optimum contact time of Bismarck Brown R dye was one hundred twenty minutes, and the adsorption followed a pseudo-second-order model. Based on the equation Langmuir and Freundlich adsorption isotherms, the adsorption capacity values of each are 158.7301 mg g^{-1} and 4.3601 mg g^{-1}. Our results showed that the material can be used as a new dye adsorbent.

Keywords: Mesoporous silica, MCM-4, adsorption, Bismarck Brown R, isotherms.

INTRODUCTION

The Indonesian textile industry has continued to experience increased production in recent years. A side effect of the increase is the ever-increasing amount of waste. Waste from the textile industry It is generally environmentally harmful if disposed of directly without prior processing. One of the hazardous textile wastes is azo dye waste. Azo dyes are widely used in fiber dyeing because they have a stronger dyeing power than other dyes. The most commonly used azo dye is *Bismarck Brown* R (BBR). BBR is a cationic dye characterized by a very dark brown color. BBR dyes have carcinogenic effects on humans and aquatic organisms.

The effect of BBR dyes in a short time or for a long time, in contact with eyes and skin, can cause severe irritation with redness at the contact site (Sole & Chipman, 1986). When swallowed, it causes testicular irritation, which includes nausea, vomiting, diarrhea, discomfort, and mouth or throat redness. If not stopped immediately, it might irritate the throat, causing chest tightness and coughing (Mittal, Thakur, & Mittal, 2013). As a result, a method is required to reduce the dyes presence before they are released into the environment.

The dye waste treatment method can be done in several ways such as coagulation (Kaur, Rani, & Mahajan, 2012), electrocoagulation, photodegradation (Tahreen, Jami, & Ali, 2020), and adsorption (Ashraf, Abulibdeh, & Salam, 2019; Herrera-González, Peláez-Cid, & Caldera-Villalobos, 2017; Kasperchik, Yaskevich, & Bil'dyukevich, 2012). The method chosen to be used in sewage treatment is the adsorption method because it is easier, cheaper, and more practical (Ayub, Sharma, & Tripathi, 2014; Kumar, Malik, & Purohit, 2017). The adsorption process can proceed smoothly if the adsorbent has a high surface area. Mesoporous silica (MCM-48) is one of the adsorbents that offers these benefits. MCM-48 material has a three-dimensional structure to avoid the occurrence of pore-blocking (Schumacher, Ravikovitch, Du Chesne, Neimark, & Unger, 2000; Taba, Budi, & Puspitasari, 2017). The mesoporous material was synthesized using silica as a source of ludox HS-40 and surfactants such as CTAB and triton X-100 (Pajchel & Kolodziejski, 2018; Wei, Liu, Lu, & Liu, 2010; Zhai et al., 2004). The synthesized mesoporous silica was washed using HCl-ethanol to remove some of the surfactants while producing a porous MCM-48 material (Taba, Shintadewi, Zakir, & Budi, 2020). The number of washing processes will affect the amount of surfactant that remains in MCM-48. In this experiment, two items of washing were carried out MCM-48 washing twice. The synthesized MCM-48

material can be used to treat BBR dye waste pollution by simulating adsorption experiments.

METHODOLOGY

Materials and Instrumentals

The chemicals used for the synthesis were ludox HS-40 as a silica source, cetyltriethylammonium Bromide (CTAB, Sigma) as a surfactant, and triton X-100 (Aldrich) as a surfactant, H_2O (distilled water) as a solvent, NaOH (sodium hydroxide). The solutions of CH_3COOH (acetic acid), C_2H_5OH (ethanol), HCl (hydrochloric acid), Whatman No.42 filter paper, universal pH paper and Bismarck Brown R (C.I. 21010) used had a wavelength of 415 nm. All materials used are of a high standard and analytical grade.

Instruments for characterization were Fourier Transform Infra-Red (FTIR) brand Prestige-21 (Shimadzu), X-Ray Diffraction (XRD) brand MAXima 7000 (Shimadzu), Scanning Electron Microscopy (SEM) brand JEOL JSM-6510LA, Spectrophotometer UV-Vis brand Spektronik 20 D^+, and other laboratory equipment.

Synthesis of MCM-48

The synthesis of mesoporous silica MCM-48 was carried out under hydrothermal conditions as reported by Ryoo (Ryoo, Joo, & Kim, 1999) followed by a study by Taba (Taba et al., 2017). In the first stage, the sodium tetrasilicate solution was dissolved by heating until it became clear. The surfactant was stirred for 2 hours in a water bath set at 80 ^oC. Then the two solutions were mixed in a polypropylene bottle and shaken for 15 minutes until the solution solidified. The mixture was then heated in an oven at 100 ^oC for 24 hours and shaken every hour. Then, the pH was adjusted, namely, pH 10 using 30 % acetic acid, and heated again in an oven at 100^oC for 24 hours. After the heating process, the mixture in the bottle was filtered using Whatman filter paper No. 42.

The residue was neutralized with distilled water and then dried in an oven until it reached a constant weight. The dried MCM-48 material was washed twice using HCl-ethanol and then characterized using XRD, FTIR, and SEM. In general, the synthesis procedure of MCM-48 can be illustrated in Figure 1.

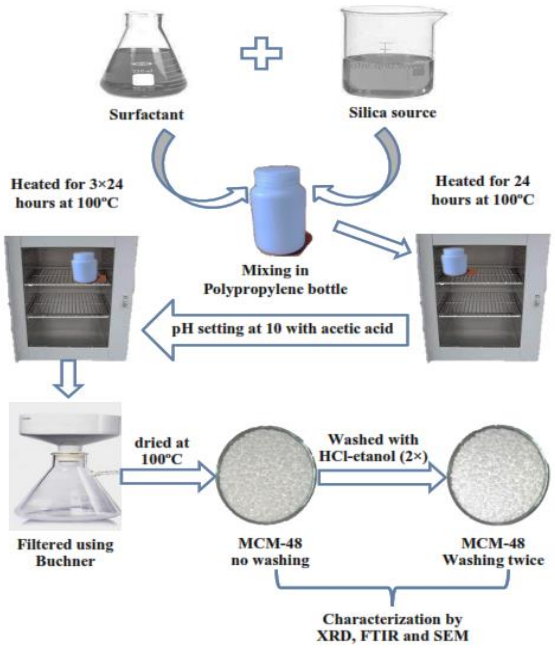

Figure 1. Illustration scheme of preparation, washing, and characterization of MCM-48

Adsorption

BBR adsorption by MCM-48-W2 material used a variation of contact time and concentration. 1000 mg/L BBR mother liquor was prepared to prepare the standard series and the sample solution. Adsorption using BBR solution with a concentration of 200 mg/L with an adsorbent Weight of 0.1 gram MCM-48-W2.

Then 0.05 L of 200 mg/L BBR solution was contacted and then stirred using a multi stirrer with time variations of 10, 20, 30, 45, 60, 75, 90, 120, 150, and 180 minutes. After that, it was filtered using Whatman filter paper No. 42. The filtrate was measured using a UV-Vis spectrophotometer at a wavelength of 415 nm. Concentration variations were carried out using 200, 300, 400, 500, 600, 700, 800 and 900 mg/L as sample solutions. The solution was pipetted as much as 0.05 L each into an Erlenmeyer containing 0.1 gram of MCM-48-W2 adsorbent and then stirred using a multi-stirrer at the optimum time.

Then filtered using Whatman filter paper No. 42, and the filtrate obtained was measured using a UV-Vis. The absorption BBR, (q) can be calculated from the difference in the concentration of BBR before and after adsorption using Equation 1.

$$q_e = \frac{C_o - C_e}{w} V \quad (1)$$

where q_e is the amount of BBR adsorbed (mg/g), C_o is the initial concentration and C_e is the final concentration of BBR in solution (mg/L), V is the volume of the solution (L), and W is the mass of the sorbent (g).

Adsorption Kinetic and Isotherm Models

The process of adsorption kinetics aims to determine the rate of adsorption of the adsorbent on the adsorbate. The adsorption kinetics will be discussed according to the quasi-first-order kinetics model developed by Lagergren and the Pseudo-second-order kinetics model (Herrera-González et al., 2017). The Pseudo-first-order kinetic model proposed by Lagergren is based on the increase in the adsorbent adsorbed on the solid as a function of time, usually expressed by Equation 2.

$$\ln (q_e - q_t) = \ln q_e - k_1 t \quad (2)$$

Then a graph is made between $\ln(q_e - q_t)$ versus t, it can be calculated with the values of q_e and k_1, where q_e is the amount of adsorbent adsorbed at equilibrium (mg/g), q_t is the amount of adsorbent adsorbed at time t, and k_1 is the rate constant first-order adsorption (min^{-1}). Meanwhile, the pseudo-second-order kinetics model is based on the adsorption rate in the solid phase, which is expressed by Equation 3.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (3)$$

The graph is made between (t/q_t) versus t. So that the values of q_e and k_2 can be calculated, where q_e is after that graph is made between (t/q_t) versus t. So that the values of q_e and k_2, can be calculated, where q_e is the amount of adsorbent adsorbed at equilibrium (mg/g), qt is the amount of adsorbent adsorbed at the time (mg g^{-1}), and k_2 is the second-order adsorption rate constant ($g.mg^{-1}.min^{-1}$).

The adsorption isotherm aims to identify the interaction between the adsorbent and the absorbate to clarify the adsorption behavior. Langmuir isotherm predicts monolayer coating on a homogeneous surface, and Freundlich applied to heterogeneous multilayer layer represents non-ideal and reversible adsorption. Langmuir and Freundlich's models are written in Equations (4) and (5), respectively.

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \frac{C_e}{Q_o} \quad (4)$$

Where C_e is the adsorbate concentration at equilibrium (mg L^{-1}). q_e is the amount of adsorbed BBR dye per gram of adsorbent (mg g^{-1}). Q_o is adsorption capacity (mg g^{-1}), and b is adsorption intensity (L mg^{-1}).

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (5)$$

Where q_e is the amount of adsorbed BBR dye per gram of adsorbent (mg/g), Ce is the concentration of the adsorbate at equilibrium (mg/L), K is the adsorption capacity (mg g^{-1}), and n is the adsorption intensity.

RESULTS AND DISCUSSION

To obtain MCM-48, the raw materials used were CTAB as a surfactant, NaOH as a catalyst, and Ludox as a silica source (Longloilert, Chaisuwan, Luengnaruemitchai, & Wongkasemjit, 2011). The shape of the micelle geometry that is formed depends on the concentration of the template used. Initially, a Liquid-crystal mesophase (micelle) was created from CTAB and triton. It gradually became a cluster of micelles forming a cubic structure and was followed by migration and polymerization of silicate anions by the addition of ludox with the help of NaOH as a catalyst to produce the framework for MCM-48 (Huang, Wang, & Wu, 2018; Rath, Rana, & Parida, 2014).

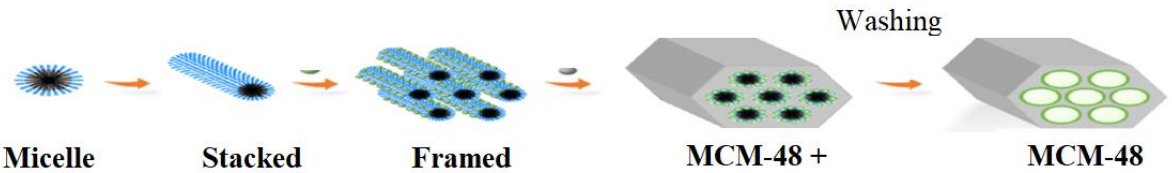

Figure 2. Illustration of MCM-48 formation mechanism and surfactant removal for porous MCM-48 results

An illustration is shown in Figure 2. The synthesized MCM-48 material still contains a surfactant, so it is necessary to remove some surfactants by washing. Washing with HCl-ethanol is expected to give better results in removing surfactants while activating the active silanol group so that MCM-48 produces open pores.

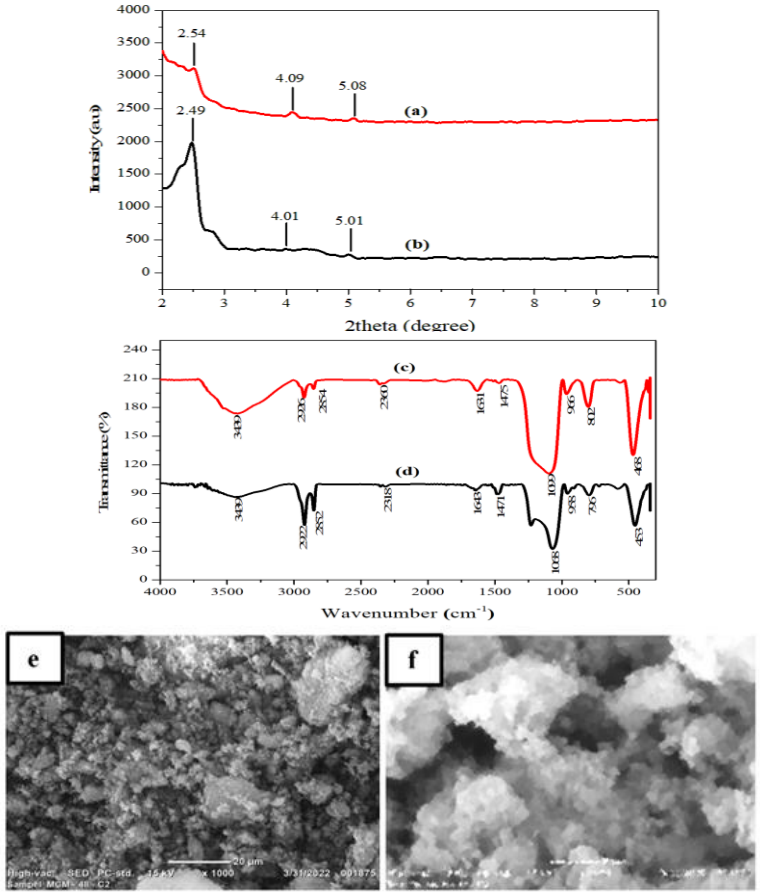

Figure 3. Characterization of MCM-48 (a). Diffractogram of MCM-48 after washing twice with HCl-ethanol, (b). Diffractogram of MCM-48 without washing (c). Spectrum Infrared of MCM-48 after washing twice with HCl-ethanol, (d). Spectrum Infrared of MCM-48 without washing, and SEM image of MCM-48 washing twice (e). Magnification 1000 times and (f). SEM image of MCM-48 washing twice magnification 8000 times.

Characterization of MCM-48

Characterization of MCM-48 before and after washing twice with HCl-ethanol is shown in Figure 3. Diffractogram of MCM-48 without washing (Figure. 3a) a shoulder peak appeared with moderately strong intensity at 2θ = 2.49^o supported by peaks at 2θ = 4.01^o and 5.01^o as well as several other peaks with weak intensity. whereas for MCM-48 after washing twice, the shoulder peak appeared at 2θ = 2.54^o followed by a peak at 2θ = 4.09^o and 5.08^o as well as several other peaks of weak intensity. After the washing process (Figure 3b), the peak shifted towards a larger 2θ due to constriction and condensation of the pores. This result is consistent with what was reported in a previous study that a strong intensity shoulder peak appears in the 2θ = 2^o region and a weak-intensity peak at 2θ = 3^o-5^{o,} which is characteristic of the three-dimensional structure of MCM-48 (Jang, Park, Ko, Lee, & Margandan, 2009; Sayari, 2000; Solovyov, Belousov, Dinnebier, Shmakov, & Kirik, 2005; Xia, Su, Ma, Ge, & Zhu, 2005). Characterization with FTIR will detect the functional groups found in MCM-48. The FTIR spectrum of the MCM-48 washing twice samples running at a wave number of 4000-400 cm^{-1}.

Table 1 The amount of BBR dye adsorbed by MCM-48 washing twice at equilibrium can be calculated based on the straight-line equations of the pseudo-first-order equation (Figure 4b) and pseudo-second-order (Figure 4c) using equations (2) and (3).

Pseudo-first-order			Pseudo-second-order			
k_1 (min^{-1})	R^2	q_e (mg g^{-1})	k_2 (g mg^{-1} min^{-1})	R^2	q_e (mg g^{-1})	$q_{experiment}$ (mg g^{-1})
-0.0001	17.4423	0.7237	0.0023	48.3091	0.9974	48.0406

Samples of MCM-48 after washing twice (Figure 3c) and (Figure 3d) showed without washing, absorption peaks in the 2922 and 2852 cm^{-1} regions experienced a shift to 2926 and 2854 cm^{-1} regions, which are C-H vibrations of symmetrical stretching and asymmetry followed by a shift in the C-H buckling vibration at the absorption peak of 1471 cm^{-1} to 1475 cm^{-1}. The next shift is the absorption peak shift from 796 cm^{-1} to 802 cm^{-1}. This shift is followed by a shift at 453 cm^{-1} to 468 cm^{-1} which is the Si-O strain and buckling vibration of the Si-O-Si originates from the silicate lattice. Another shifting absorption peak has been observed.

The shift occurred from wave number 1065 to 1099 cm^{-1}. This area is an O-H bending vibration. this is followed by another shift in the absorption peak from 958 cm^{-1} to 966 cm^{-1} as Si-O stretching vibration of Si-OH. These results confirm that the MCM-48 surface does have a silanol group, as expected. The IR spectrum closely matches MCM-48 reported in previous studies (Taba et al., 2020; Wang, Lu, Yang, Xiao, & Wang, 2012).

MCM-48 particles is irregular at magnifications 1000 and 8000 times. The picture shows that the particle size is not uniform, and several forms of clumps exist. However, if observed in certain sections, the particle shape is almost spherical as previously reported results that MCM-48 has a fine spherical particle shape (Beck et al., 1992; Endud & Wong, 2007; Mokri et al., 2019). The resulting SEM image is not so good due to the condition of the tool. That is not optimal for scanning both at low and high magnifications.

Adsorption

The effect of contact time on the adsorption of BBR dye by the adsorbent of MCM-48 washing twice can be determined by varying the adsorption time. Figure 4 shows the amount adsorbed as a function of the contact time. Based on the graphic in Figure 4a, the amount of adsorbed BBR dye increases from 5 minutes to 120 minutes. This is because a longer contact period allows for greater diffusion or attachment of solute molecules to the adsorbent, resulting in a greater amount of adsorbed

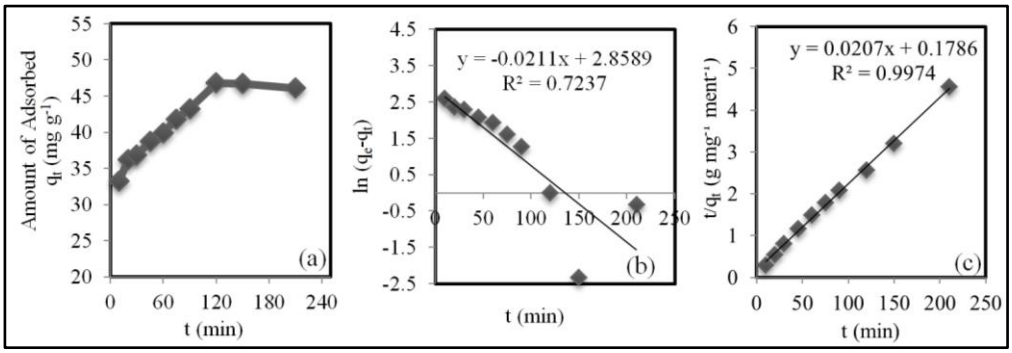

Figure 4. Adsorption of BBR dye by MCM-48 washing twice (a). Effect of contact time on the adsorption BBR dye, the adsorption kinetics model of BBR (b). Pseudo-first-order and (c). Pseudo-second-order

The peaks at 2926 and 2854 cm^{-1} are associated with the C-H stretching from surfactant that has not been removed completely. Figure 3e and 3f show that the shape of the synthesized material (Yun et al., 2011). However, after 150 minutes, the amount of adsorbed BBR dye decreased, indicating that the adsorbent surface had been saturated. It is clear that from 90 minutes to

120 minutes, there is a very significant increase, indicating that 120 is the optimum time for adsorption with an adsorption value of 46.8171 mg g^{-1}. The period to attain equilibrium is greater, two hours because the adsorption combines physical and chemical adsorption. From the contact time data, the adsorption kinetics of BBR dyes can be determined as shown in Figures 4b and 4c below. Comparison of reaction constants, respectively. Pseudo-first-order and pseudo-second-order for BBR dye adsorption by MCM-48 washing twice are presented in Table 1.

The adsorption value BBR dye based on the equation of pseudo-first-order was 17.4423 mg g^{-1} (R^2 = 0.7237) and pseudo-second-order was 48.3091 mg g^{-1} (R^2 = 0.9974). The correlation coefficient of pseudo-second-order is close to 1 and the calculated q_e value is close to the experimental q_e value (48.0406 mg g^{-1}). This proves that the adsorption of BBR by MCM-48-W2 follows the pseudo-second-order reaction kinetics model.

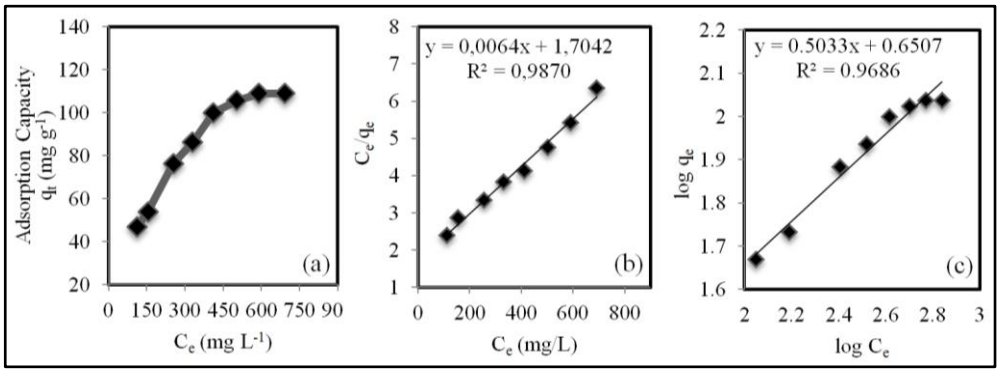

Figure 5. Adsorption of BBR dye by MCM-48 washed twice (a). Effect concentration on the adsorption BBR dye, (b) Langmuir Isotherm and (c) Freundlich Isotherm

process between the adsorbent and the adsorbate. The number of substances adsorbed part unit weight of the adsorbent is then expressed as a curve (Rajendaran, Zaini, Arsad, & Nasri, 2019). The amount of BBR dye adsorbed by MCM-48 washing twice as a function of concentration can be seen in Figure 4 and 5. The amount of BBR dye adsorbed increases with increasing concentration value. However, in this experiment, it was impossible to determine the maximum amount adsorbed because the adsorption conditions did not yet have a saturation point. Therefore, the adsorption capacity was calculated using two adsorption isotherms, namely Langmuir and Freundlich isotherms by making their respective curves as shown in Figure 4b and 4c. Based on the equation of the Langmuir isotherm, it can be calculated the value of adsorption capacity (Q_o), correlation coefficient (R^2), and adsorption intensity (b). Likewise, with the Freundlich isotherm, we can calculate the value of adsorption capacity (K_f),

Table 2. The amount of BBR dye adsorbed by MCM-48 washing twice can be calculated based on the straight-line equations of the Langmuir (Fig. 5a) and Freundlich isotherm (Figure 5b) using Equations (4) and (5).

Isoterm Models	Parameters	Values
Langmuir	R^2	0.9870
	Q_o (mg g^{-1})	158.7301
	b (L mg^{-1})	0.0037
Freundlich	R^2	0.9686
	k (mg g^{-1})	4.4740
	n (g L^{-1})	1.9868

Varying concentrations can determine the effect of concentration on the adsorption process. The adsorption isotherm can identify the interaction correlation coefficient (R^2), and adsorption intensity (n). The correlation coefficient can be used to determine the most appropriate isotherm in the

adsorption process. The R^2 value in Figure 5 shows that the adsorption follows Langmuir and Freundlich isotherms because the value of R^2 is closer for both isotherms. The comparison of The values of the two isotherms can be seen in Table 2.

CONCLUSION

The optimum condition of contact time for BBR dye adsorption by MCM-48-W2 was obtained at 120 minutes with an adsorption value of 48.0406 mg g^{-1} and followed a pseudo-second-order model. The adsorption capacity for MCM-48 washing twice against BBR dye was 158.7301 mg g^{-1} (Langmuir isotherm) and 4.4740 mg g^{-1} (Freundlich isotherm).

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