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Sustainable Environment Research xxx (2018) 1e7

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Removal of Procion Red MX-5B from songket's industrial wastewater in South Sumatra Indonesia using activated carbon- 1

Fe3O4 composite

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article info Article history: Received 8 January 2017 Received in revised form 11 December 2017 Accepted 17 January 2018 Available online xxx Keywords: 11

Procion Red MX-5B Activated carbon Fe3O4 Composite abstract Songket is traditional costume in South Sumatra, Indonesia. This study investigates the feasibility of using activated carbon-Fe3O4 composite to adsorb the Procion Red MX-5B dye from songket's industrial wastewater. The adsorbent was characterized using the

surface area analyzer, X ray Diffraction, Scanning Electron Microscopy, Energy Dispersive X-ray Analysis, Fourier Transform Infrared and Vibrating Sample Magnetometer. The effects of 7

pH, weight of composite and the contact time were evaluated to determine the adsorption efficiency. The kinetic and isotherm were carried out to evaluate the adsorption behavior of composite. The toxicity level of songket's industrial wastewater was measured using Tilapia fishes as the biological indicator. The 24-h LC50 was calculated using Probit analysis method. The results show that the adsorption process of Procion Red MX-5B using activated carbon-Fe3O4 composite follows a pseudo first order kinetic and the experimental data show a good correlation with Freundlich isotherm. Songket's industrial wastewater has the 24-h LC50 for Tilapia of 5.6% ± 0.6. After treatment using acti- vated carbon-Fe3O4 composite at pH 6 and contact time of 50 min, the adsorbent can reduce concen- tration of the Procion Red MX-5B by 94% and chemical oxygen demand by 96%. The experimental results indicate that the activated carbon-Fe3O4 composite is effective as an adsorbent for the treatment of songket's industrial wastewater.

1. Introduction Songket is the cultural heritage of Indonesia, particularly in

South Sumatra. Songket is used as cloth by the people of South Sumatra for parties, traditional ceremonies and other official events. Currently, the songket industry is a domestic industry that is growing in the community of South Sumatra [1]. The process of songket manufacture consists of several stages of dyeing, weaving and finishing. The dyeing process produces wastewater contain- ing synthetic dye. The dyes used in the dyeing process are the azo *

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Chinese Institute of Environmental Engineering. classification (eN]Ne). The Azo compounds can be bonded with aromatic or aliphatic compounds [2]. Aromatic-azo compounds are stable and have light colors. The azo dyes also act as a reactive dye. Reactive groups which form part of the dye are easily sepa- rated. The dye often used in the dyeing process of songket in- dustry is the Procion Red MX-5B dye [3]. The Procion Red MX-5B dye is classified as aromatic-azo which has the molecular formula C19H10Cl2N6S2Na2O7. The presence of a synthetic dye

in water can impede the penetration of sunlight into the water and reduce the supply of oxygen in the 16

water. Decomposition of azo dyes by bacteria can produce aromatic amine compounds which are by far more toxic than the dye itself [4]. The azo dye when discharged in water, can survive long enough and accumulate, this accumulation has toxic effects on aquatic organisms [5]. In addition, it is a carcinogenic substance which stimulates the growth of cancer [6]. https://doi.org/10.1016/j.serj.2018.01.004 2468-2039/ 4

Some characteristics of

songket's industrial wastewater include the following; it is made of synthetic dye, chemical oxygen demand (COD) in high content and low pH. The concentration of

Procion Red MX-5B from one of the songket's industrial wastewater in South Sumatra 1

is 1237 mg L 1, COD is 3820 mg L 1, and pH is 5.8. Therefore, songket's industrial wastewater treatment is necessary prior to its discharge to the environment. The wastewater treat- ment using magnetic materials is an efficient method to remove pollutant. The advantage of this method of rapid adsorption and separation processes is that it can be performed quickly using a permanent magnet, without requiring any filtration. One of the magnetic materials that effectively act as the adsorbent is magne- tite (Fe_3O_4), which has a superparamagnetic property [7,8]. The nanomagnetic ability of Fe_3O_4 has been used to adsorb the Procion Red MX-5B [3], heavy metals include Cu(II), Cr(VI), Ni(II) and Cd(II) [9]. To increase the adsorption capacity, the modification is per- formed between two or more adsorbents called composite. In recent years, many researchers have developed magnetic com- posite, for example, magnetic rectorite/magnetite for the adsorp- tion of Methylene Blue and Methyl Orange [10],

chitosan coated magnetic hydroxyapatite for the **adsorption of Reactive Blue** dye [11], **and** 25

zeolite/magnetic that reduces Cu(II) [12]. The magnetic properties of the composite are more easily attracted by a magnet. Activated carbon is an excellent adsorbent for wastewater treatment because of its high adsorption capacity, large surface area, high surface reactivity degree and low cost [13,14]. Palm shells have the main content of cellulose and hemicellulose component [15], so

it can be used as raw **material for activated carbon. The** 32

palm shell is the largest part of the palm oil industrial waste. In Indonesia on 2016, the palm field reached 11.9 Mha with a total production of 33.2 Mt of oil palm fruit [16]. The purpose of this study is to prepare activated carbon with Fe_3O_4 to adsorb Procion Red MX-5B and carry out toxicity tests on fish from songket's industrial wastewater. Toxicity test used to determine the negative effects of a substance to biota (LC50), which is the value of the toxic substance exposure concentration that causes 50% mortality of the total biota tested [17]. In this study, toxicity test of songket's industrial wastewater was performed us- ing Tilapia fish as test animal. Tilapia is used as test animals because they have the capability of surviving in poor water quality with low oxygen levels and low or high pH [18]. Tilapia fish is also consumed as a food source for humans. 2. Materials and methods 2.1. Chemicals Activated carbon from palm shells is made using H_3PO_4 as an activator. Chemical reagents were used such as H_3PO_4, $FeCl_3$, $FeCl_2$, NaOH from Merck, Germany. Procion Red MX-5B dye from Sigma Aldrich, CAS Number 17804-49-8. The songket's industrial waste- water from songket industry in Palembang, South Sumatra. 2.2. Preparation of activated carbon-Fe_3O_4 composite Activated carbon was synthesized from palm shells which carbonized at 500 C for 2 h. The carbonization result was milled to obtain size of 200 mesh (0.075 mm). A total of 100 g of palm shell powder was soaked with 300 mL of 5% H_3PO_4 solution, heated at 400 C while flowed with N_2 at 150 cm3 min 1 for 1 h. The product activated carbon was washed using 0.1 M NaOH solution, followed by distilled water until pH neutral. Activated carbon is dried at the oven at 110 C for 2 h. Activated carbon-Fe_3O_4 composite was obtained by using co- precipitation. $FeCl_2$ and $FeCl_3$ dissolved in the molar ratio of 1:2 in 200 mL of demineralized water. Then, activated carbon was added to solution.

The amount of active carbon was adjusted to obtain mass ratio of 29

active carbon and Fe3O4 of 1:1 and 2:1. The mixture is stirred with a magnetic stirrer, under N2 bubbling and added with NaOH 1 M at a temperature of 70 C until the pH reached about 11 and black precipitate is formed. The activated carbon-Fe3O4 composite can be formed as given: FeCl2 þ 2FeCl3 þ 8NaOH þ activated carbon/4H2O þ 8NaCl þ activated carbon Fe3O4 (1) The composite is separated using a permanent magnet, then washed using demineralized water until neutral and dried at 105 C in the oven. Surface area of composites was analyzed using micromeritics ASAP 2020 analyzer based on BET equation. Magnetic properties were analyzed using Vibrating Sample Magnetometer (VSM) on Lakeshore 74004.

X-ray Diffraction (XRD) pattern was analyzed using XRD Shimadzu XD610 with Cu- Ka radiation 22

(l ¼ 0.154 nm) in the 2q range 10e80 . Morphology and element content was ob- tained

using Scanning Electron Microscope-Energy Dispersive X- ray Spectrometry (SEM -EDX), JEOL JEM 1400 and Fourier Transform Infrared Spectroscopy (FTIR) 12

Shimadzu 5400 to know the func- tional group of composite. 2.3. Adsorption study Adsorption study was done using Procion Red MX-5B by batch experiments. Determination of the concentration of Procion Red MX-5B was performed at a wavelength of 545 nm using UVeVis Spectrophotometer Shimadzu 2550. The effect of pH on the sorp- tion process was tested within pH range of 3e9 with the initial concentration of Procion Red MX-5B 150

mg L 1 in the volume of 50 mL and the weight of the 28

composite of 30 mg, the

pH was adjusted using 0.1 M HCl and 0.1 M NaOH [19e21]. The effect of 14

composite weight was carried out in the weight range of 10e60 mg while the various contact time ranged from 10 to 80 min. The ex- periments were conducted three times at room temperature and the error bar represented by the standard deviation. The formula expresses the amount of dye adsorbed: qe ¼ ðCo CeÞ W V

(2) where qe is adsorption capacity (mg g 1), C0 and Ce are initial concentration of dye and equilibrium liquid phase concentration (mg L 1), V is volume of solution (L), and W is weight of composite (g). The 5

similar treatment for songket's industrial wastewater using activated carbon-Fe3O4 composite was also conducted to reduce concentration of Procion Red MX-5B and COD. Analysis of COD followed the Standard Test Methods (ASTM D1252-06) for COD [22]. 2.4. Toxicity test The experiment was conducted in 25 L glass aquaria consisting of songket's industrial wastewater with concentrations of 0, 2, 4, 6, 8, 10%, and composite treated songket's industrial wastewater. The Tilapia fish were found from the local fishery and acclimated for ten days in the laboratory. Each aquarium filled with 20 Tilapia fish with approximately equal weight and length. Distilled water is used as dilution and control conditions of experiment include pH, temperature, and total hardness according to APHA [23]. Constant air flowed into the glass aquaria using a pump. Table 1 BET surface area, pore volume and saturation magnetization of activated carbon and activated carbon-Fe3O4 composite. Materials Surface Pore volume Saturation area (m2 g 1) (cm3 g 1) magnetization (emu g 1) Activated carbon Composite 2:1 Composite 1:1 386 0.20 322 0.13 272 0.06 e 13 16 Observations were carried out for 24 h to observe the number of death fish. The death fish were separated immediately from the glass aquaria. The Probit analysis method to determine the LC50 was carried out using SPSS 20.

3. Results and discussion 3.1. Characterization of adsorbent The 23

activated carbon-Fe3O4 composites were synthesized with a weight ratio of activated carbon and Fe3O4 at 2:1 and 1:1 respec- tively. The value of the magnetization is influenced by the amount of Fe3O4 in the composite; therefore, the reduction of Fe3O4 will affect the value of magnetization [24]. The higher value of magnetization of the composite favors easy attraction by a magnet. The increasing magnetic properties have an impact on surface area reduction, thus reducing the ability of composite to adsorb the pollutants [25,26]. The surface area, pore volume and magnetitic saturation of the activated carbon and composites are shown in Table 1. Composite with ratio 2:1 has a large surface area than composite 1:1, while both of composite have insignificant magnetic properties difference. This study shows that composite with ratio 2:1 and 1:1 have a saturation magnetization of 13 and 16 emu g 1, respectively. Subsequent experiments were using the composite weight ratio activated carbon and Fe3O4 of 2:1. Fig. 1 is the morphology of activated carbon and activated car- bon-Fe3O4 composite with 10,000 magnification by SEM. Com- posite has less pore than activated carbon

which indicates the presence of nano-sized Fe3O4 particles on the surface of 31

activated carbon. EDX study presented in Table 2 shows the elemental composition of the adsorbent. The EDX result showed that the chemical composition of activated carbon has C of 98.8%. After the formation of a composite with a weight ratio of 2:1, it decreased to 58.8% and percentage of Fe and O was 24.3 and 16.1%, respectively. The composite that has the lowest carbon with a weight ratio of 1:1 is 38.4%. It is clear, that the presence of Fe3O4 in the composite cause of the decrease a surface area. The subsequent experiments were using the composite weight ratio activated carbon and Fe3O4 of 2:1. The characterization results using XRD (Fig. 2) shows that acti- vated carbon is amorphous with a wide peak. The peak of activated carbon-Fe3O4 composite confirmed the existence of Fe3O4 peak at 2q value of 30.2, 35.6, 43.3, 52.8, 57.2, and 62.9 which indicate

cubic spinel structure of peaks indexed as (220), (311), (400), (422), (511) and (440) 19

base on 2q values and relative intensity for Fe3O4 of JCPDS 19-0629 [27]. The change of sharp peaks in the composite indicates that the formation of the composite has been successful. The FTIR spectra of activated carbon-Fe3O4 composite and Fe3O4 are presented in Fig. 3. The bands at 3402.2 cm 1 in the composite indicated vibrations of OeH stretching and binding water. The Fig. 1. Morphology of (a) activated carbon, (b) activated carbon-Fe3O4 composite (2:1) and (c) activated carbon-Fe3O4 composite (1:1). Table 2 The elements percentage of activated carbon and activated carbon-Fe3O4 composite. composites and contact time. Fig. 4a shows the effect of pH on adsorption Procion Red MX-5B in the variation of pH at 3e9. The adsorption experiments were used for 50 mL of Procion Red MX-5B C 98.8 58.8 38.4 O 0.6 16.1 32.6 contact time of 30 min pH is an important parameter in the Fe e 24.3 28.6 adsorption process because it affects adsorption capacity. The result Na e 0.5 0.3 P 0.4 0.3 0.1 indicated the removal of the dye is optimum in lower pH. The pKa Si 0.2 e e of Procion Red MX-5B is 2.5. At the pH solution above the pKa, the dye has a negative charge of two sulfonate groups. Thus, causing electrostatic attraction between the negative charge dye and the 1700 positive charge of activated carbon-Fe3O4 composite [11,30,31]. In 311 alkaline solution, there is competing between negative charge dye 1500 with the hydroxide ion. Thus, the adsorption capacity decreased. 1300 a) 220 440 400 511 422 Fig. 4b shows the influence of adsorbent weight on the adsorp- tion of Procion Red MX-5B. The adsorption process used Procion Red Intencity (a.u) 1100 a MX-5B

with a concentration of 150 **mg L 1,** in **50 mL at** 6

pH 6 and Element (%) Activated carbon Composite 2:1 Composite 1:1 with initial concentration 150 mg L 1, 30 mg of composite and contact time of 30 min. The adsorption of dye increases with in- 900 crease in the weight of composite at 10e40 mg, afterward the 700 adsorption decreased. We can say that the increased adsorption of dye is not comparable with the addition weight of the composite, so 500 b) the amount of the dye adsorbed per weight of adsorbent decreased. 300 Pore size, adsorbent reactivity, and the amount of adsorbate molecules determines the optimum contact time [32]. The 100 adsorption experiment for contact time was conducted at pH 6, the 0 10 20 30 40 50 60 2θ (Degree) 70 80 90 weight of composite 40 mg and contact time in the range 10e90 min with an initial concentration of Procion Red MX-5B Fig. 2. X ray diffraction pattern of (a) activated carbon-Fe3O4 composite and (b) acti- 150 mg L 1 at room temperature (25 C). It can be seen in Fig. 4c vated carbon. that adsorption capacity is dependent on contact time, the longer contact time between the dye and composite increases the peaks in the region 2850e2950 cm 1 and 1550e1680 cm 1 were adsorption capacity. In this study, it reaches equilibrium at the contact time of 50 min. The optimum contact time depends on the associated CeH stretching and C]C, respectively [28]. The char- acteristic of the composite can be seen of the peak at 576.3 cm 1 type of adsorbent and adsorbed molecules. In the adsorption pro- attributed to FeeO stretching. In the pure Fe3O4, it appears at wave cess of the CoFe2O4-carbon composite to the Malachite Blue Dye number 582.5 cm 1. This peak, similar to another result at peak optimum at 5 min [33], and the optimum time of sodium-Fe3O4 581.0 cm 1 for composite bentonite-Fe3O4 [29]. polymer

composites for adsorption Methylene Blue, Neutral Red and Methyl Orange 30

are 30, 90 and 15 min, respectively [10]. 3.2. Effect of pH solution, weight of adsorbent and contact time 3.3. Adsorption kinetics Variables studying the adsorption of Procion Red MX-5B using activated carbon-Fe3O4 composite with a weight ratio of activated Studies of adsorption kinetics can indicate how fast the carbon and Fe3O4 of 2:1 include pH solution, the weight of adsorption reaction or the effectiveness of adsorption. The 140 120 Transmitant (%T) 100 80 a) 2850-2950 1577.6 576.3 3402.2 60 40 b) 3400.4 582.5 20 4500 4000 3500 3000 2500 2000 1500 1000 500 0 Wavelength (cm-1) Fig. 3. FTIR spectra of (a) activated carbon-Fe3O4 composite and (b) Fe3O4. (a) 200 Adsorption capacity (mg g-1) 190 180 170 160 150 140 130 1 2 3 4 5 6 7 8 9 10 pH solution (b) 260 Adsorption capacity (mg g-1) 240 220 200 180 160 140 120 100 0 20 40 60 80 Weight of composite (mg) (c) 250

Adsorption capacity (mg g-1) 200 150 100 50 0 26

0 10 20 30 40 50 60 70 80 90 100 Contact time (min) Fig. 4. Effect of different parameters on adsorption capacity of activated carbon-Fe3O4 composite (initial concentration of Procion Red MX-5B 150 mg L 1, and contact time 30 min). (a) pH solution with 30 mg composite, (b) weight of composite at pH 6, (c) contact time at pH 6 and 40 mg composite. adsorption kinetics models evaluated were

pseudo first order and pseudo second order. The pseudo first order reactions illustrate that the rate of adsorption of 10

the dye adsorbed is proportional to the time as shown below [34]: ddqtt ¼ k1ðqe qt Þ

(3) where qe and qt are the concentration adsorbed in equilibrium and absorbed per time (mg g 1), t is 15

time and k1

is the pseudo first order rate constant of adsorption (min 1). The integration of the equation with limits from t ¼ 0 to t and 13

q ¼ 0 to qt , can be expressed [19,34e36]. lnðqe qtÞ ¼ k1t þ ln qe (4) The pseudo second order kinetics for Procion Red MX-5B with activated carbon-Fe3O4 composite was determined the following form [34]: ddqtt ¼ k2ðqe qt Þ2 (5) where qt is concentration adsorbed at time t

(mg g 1) and k2 is the constant of pseudo second order reactions (g mg 1 min 1). The adsorption of dye 9

usually follows the pseudo first order or second order. The adsorption of Congo Red using magnetic cellu- lose composite flowed the pseudo first order, while Carmin Indigo and Reactive Orange 16 dye adsorption using zeolite-Fe2O3 com- posite follows the pseudo second order [34]. According to the value of R2, the adsorption process of Procion Red MX-5B using activated carbon-Fe3O4 composite follows the pseudo first order reaction as shown in Fig. 5. The value of coefficient of determination (R2) on the first order of

0.994 is higher than second order of 0.732 while the value of k on first order is 0.057 min 1. 3.4. Adsorption isotherms Adsorption isotherm is used to understand the phenomenon of adsorption. It is important to understand

how the molecule **is distributed between** in **the liquid and solid** phase. **The** 6

Langmuir isotherm assumes that adsorption occurs only on a single surface (monolayer).

Langmuir isotherm equation **is** derived **based on the** assumption **that** an equilibrium between **the** adsorbed **molecules** on **the** 27

surface of the adsorbent and the molecules that are not adsorbed. The Langmuir equation is expressed as: qe ¼ qKmþax:CCee (6)

where qe is the is **amount of dye adsorbed (mg g 1),** 21

qmax

(mg g l) **is the** maximum **adsorption capacity, Ce (mg L** l) **is equilibrium concentration of dye in solution,** K (L **mg** 1) **is** 8

a constant related to binding site. The slope and intercept of linear plots for K=qmax and 1/qmax. The Linear form of Freundlich equation expressed in the equa- tion [32]: qe ¼ KF Ce1=n (7) 5 4.5 4 3.5) 3 ln (− 2.5 2 1.5 1 0.5 0 0 10 20 30 40 50 60 t (min) Fig. 5. Pseudo first order kinetics for adsorption Procion Red MX-5B on activated carbon-Fe3O4 composite. 1.1 1 0.9 0.8 0.7 Probability 0.6 0.5 0.4 0.3 y = 0.1034x - 0.0648 0.2 R^2 = 0.9732 0.1 0 -0.1 0 1 2 3 4 5 6 7 8 9 10 11 -0.2 Songket's industrial wastewater concentration (%) Fig. 6. Probit analysis of songket's industrial wastewater. where KF is related to the adsorption capacity of the adsorbent and 1=n is another constant related to the surface heterogeneity. The slope and intercept of linear plots of qe against Ce yield the values of 1=n and 2

KF . Adsorption parameters based on Langmuir isotherm measure- ments are: R2 of 0.801, K constant of 0.012 and the maximum ca- pacity of adsorption of 278 mg g 1. The R2 of Freundlich isotherm is 0.996 which is higher than Langmuir isotherm. Therefore, Freundlich isotherm has better linearity than Langmuir. The KF and n values of Freundlich isotherm are 3.18 and 1.15, respectively. Since the favorable adsorption process described by the n value ranges from 1 < n < 10 [29], in this study, the n constant lies between in the range. It is obvious that adsorption isotherm tends to follow Freundlich isotherm. This also shows that adsorbent surface is more heterogenous. 3.5. Toxicity test Evaluation of the toxic effects of songket's industrial wastewater in this study conducted using Tilapia as test animals [37]. Physically, the death fish suffered damage at the gills. The gill damage can be a lamella wall thickening, degradation of cell or tissue damage which cause the gill function to become unnatural and disturbing the process of respiration and eventually cause death [38]. Toxicity test illustrated in Fig. 6. The results of Probit analysis using SPSS 20 show that the value LC50-24 h was 5.6 ± 0.6%. Assessment of acute toxicity level is: if 100% LC50 > 75% ¼ toxic mild, 75% LC50 > 50% ¼ toxic, and LC50 50% is very toxic [39]. Thus, the songket's industrial wastewater is classified as very toxic. The concentration of the Procion Red MX-5B contained in the wastewater is very high at 1237 mg L 1. Textile dye content of 1 mg L 1 has caused the water to become colored [5]. Typically, textile wastewater contains a synthetic dye ranging from 100 to 200 mg L 1 and COD in the range 150e12.000 mg L 1 [40]. Table 3 The quality of songket's industrial wastewater before and after adsorption using activated carbon-Fe3O4 composite. Parameters Before adsorption After adsorption (mg L 1) (mg L 1) Efficiency (%) Procion Red MX-5B 1237 70 94 COD 3824 150 96 The observation of treated songket's industrial wastewater for 24 h of exposure shows that no dead animal was found. Their result showed that toxicity of songket's industrial wastewater decrease after treatment using activated carbon-Fe3O4 composite. Table 3 shows the characteristic of songket's industrial waste- water before and after treatment using activated carbon com- posite-Fe3O4. It indicates that the activated carbon composite- Fe3O4 has a significant effect on the treatment of wastewater from songket industrial. 4. Conclusions Activated carbon-Fe3O4 composite with the molar ratio of acti- vated carbon: Fe3O4 ¼ 2:1 has the potential for the treatment of songket's industrial wastewater. Separation can be performed quickly and easily using permanent magnets without filtering. The kinetics and adsorption isotherm used to describe the adsorption process of Procion Red MX-5B is the first order reaction and Freundlich isotherm. The songket's industrial wastewater which has a concentration of Procion Red MX-5B of 1237 mg L 1 and COD of 3824 after treatment using composite activated carbon-Fe3O4 decreased to

70 mg L 1 and 150 mg L 1, respectively. The 33

songket's industrial wastewater has LC50-24 h value of 5.6 ± 0.6% to Tilapia fish. There was no death recorded on the Tilapia fishes during the 24 h observation in the songket's industrial wastewater after treatment using activated carbon-Fe_3O_4 composite. Acknowledgements The authors

would like to thank the Sriwijaya University for financial support “Unggulan Kompetitif Universitas Sriwijaya” 18

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