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Congo Red Dye Adsorption using Magnesium Hydroxide from Seawater Bittern

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Abstract

The study of $Mg(OH)_2$ from seawater bittern as an adsorbent for Congo red (CR) from an aqueous solution has been performed. This study aimed to determine the effect of pH, contact time, and initial CR concentration on CR adsorption by $Mg(OH)_2$. The adsorption kinetics and isotherms of CR on $Mg(OH)_2$ in an aqueous solution were also studied. The optimum adsorption was obtained at pH 8 within 90 minutes of contact time with an adsorption capacity of 46.3 mg/g for an initial CR concentration of 29 mg/L. The adsorption process followed the Freundlich isotherm model with an n value of 2.579 and the pseudo-second-order kinetic model with a k2 value of 0.0021 g $mg^{-1}min^{-1}$.

1. Introduction

Dye environmental contamination has increased recently, especially water pollution. The textile industry contributes around 2% of industrial wastewater production volume [1]. Most color waste that still contains constituent parts, such as the azo structure, benzene, hydroxy, amine, or thiol groups from polymers, is directly discharged into the environment without any prior dye removal process. Moreover, these dyes cause severe environmental contamination since they cannot be degraded by anaerobic bacteria. Dye-contaminated water should not be consumed or used for daily life [2, 3]. Congo red is a reactive dye often used in the textile industry, mainly in cotton fabrics. This dye is very soluble in water and produces a red solution that cannot be degraded biologically (recalcitrance) [4, 5].

Several methods can be employed to treat textile waste, including adsorption, precipitation, evaporation, solvent extraction, ion exchange, and reverse osmosis [6]. The adsorption method has more advantages when compared to other methods because the process is relatively simple, and the efficiency is relatively high [7]. According to Stumm and Morgan, adsorption mainly occurs on solid surfaces with an abundance of functional groups such as –OH, –NH, –SH, and –COOH and is generally driven by interactions between metals and surface functional groups of the adsorbent through complex formation interactions [8].

The adsorption process is influenced by several factors, including pH, reaction time, and adsorbate concentration. pH can affect the active site of the adsorbent surface, which plays an active role in the adsorption of the adsorbate. The pH may impact the absorbance measurement since the adsorbate has a varied maximum wavelength depending on the pH, which changes the final wavelength [9]. Reaction time or contact time can affect the adsorption process; as the contact time increases, more adsorbent particles interact with the adsorbate, increasing the amount of adsorbate adsorbed [10]. Another factor affecting the adsorption process is the adsorbate concentration. The amount of substance accumulated on the adsorbent surface will increase if the adsorbate concentration in the solution is greater [11].

Recently, several studies have developed magnesium hydroxide ($Mg(OH)_2$) as a dye adsorbent. In the research of Ramesh and Sreenivasa [12], $Mg(OH)_2$ was used as an adsorbent for indigo carmine dye with the adsorption percentage at pH 6–7 (333 K) was 20% higher than at pH 12–13. Yunessnia lehi and Akbari [13] employed $Mg(OH)_2$ in membrane capsule form as an adsorbent for dye wastewater in carpet industries, in which the dye molecules were adsorbed on the surface of the

nanoparticles, resulting in dye concentration decreased. Liu *et al.* [14] aimed a study on $Mg(OH)_2$ as an adsorbent for methyl orange. Then the study by Liu *et al.* [15] showed that the adsorption of congo red on $Mg(OH)_2$ and MgO occurs through surface exchange until the surface function sites are fully occupied. The dye molecules are diffused for further adsorption reactions (complexation interactions).

The alkaline precipitation method can be employed to produce $Mg(OH)_2$ from seawater bittern. Some precipitacion agent can be used NaOH, $CaCO_3$, NH_4OH [16]. Bittern, known as old water, is a waste generated from the remaining crystallization of the salt-making process in the form of a concentrated liquid. Generally, this bittern has not been widely used and is discarded at the final stage of production, although it is abundant in magnesium and other elements. The compounds contained in bittern are magnesium sulfate ($MgSO_4$), sodium chloride (NaCl), magnesium chloride ($MgCl_2$), potassium chloride (KCl), calcium chloride ($CaCl_2$) [17]. These compounds will have benefits and economic value if they are processed further, especially the magnesium content.

As mentioned above, it is expected that employing $Mg(OH)_2$ can solve the environmental pollution caused by Congo red dye. The $Mg(OH)_2$ in this study was synthesized from seawater bittern, which has not been widely used. Therefore, it is anticipated that $Mg(OH)_2$ will not only solve the pollution problem but also raise the economic value of seawater bittern.

2. Methodology

2.1. Equipment and Materials

Materials used were bittern obtained from PT. Garam Mas, NaOH and HCl (analytical grade, produced by E-Merck). The adsorbate used in this study was the anionic dye Congo red (molecular formula $C_{32}H_{22}N_6O_6S_2$, a molecular weight of 696.66 g/mol and a surface area of 55.6 Å^2). Figure 3 shows the molecular structure of Congo red dye.

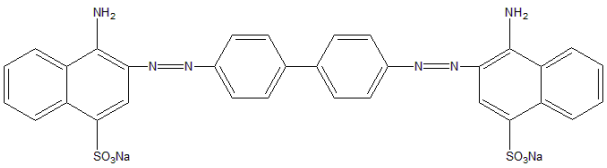

Figure 1. Congo red dye molecular structure

The equipment used include XRD (X-ray Diffractometer, Panalytical/Xpert 3 Powder), FTIR (PerkinElmer Spectrum Version 10.4.00), SEM-EDX (JEOL JSM-6510LA), glassware, analytical balance, oven, orbital shaker, universal pH indicator and UV-Vis spectrophotometer (FLUOStar Omega Microplate).

2.2. Chemical and Materials

The adsorbent used in this study was $Mg(OH)_2$ from bittern obtained from PT. Garam Mas, Rembang, Indonesia. The $Mg(OH)_2$ synthesis process was conducted following the research of Jumaeri *et al.* [18] with modifications. $Mg(OH)_2$ was synthesized via precipitation method by mixing 500 mL of 31°Be bittern with 1000 mL of 2 M NaOH. The mixture was precipitated until it reached pH > 10. After completely settled, the mixture was filtered to obtain a solid. The solid $Mg(OH)_2$ was washed using distilled water to remove NaCl. Distilled water is used for washing because the ionic solid NaCl dissolves easily in water solvents [19]. After washing, the $Mg(OH)_2$ was dried in an oven at 110°C for 2 hours, and the final mass obtained was weighed. The obtained bittern from PT. Garam Mas and several methods such as precipitation process, agglomeration, and synthesis are shown in Figure 1. Solid $Mg(OH)_2$ was then characterized using XRD, FT-IR, and SEM.

Figure 2. (a) bittern sample from PT. Garam Mas, Rembang (b) precipitation process (c) agglomeration of $Mg(OH)_2$ (d) synthesis result

The adsorbate used in this study was a congo red dye solution. The stock solution was prepared by dissolving 1 g of CR with 1 L distilled water and stored in a glass bottle for further use. The maximum wavelength of 20 mg/L CR with predetermined pH variation was analyzed at a wavelength ranging from 410 to 700 nm. After the maximum wavelength was obtained, a calibration curve was generated by analyzing standard solutions of CR (0, 1, 5, 10, 15, 20, and 25 mg/L) with a predetermined pH variation at the previously determined maximum wavelength.

2.3. Congo red dye adsorption

A total of 0.01 g $Mg(OH)_2$ was added to 25 mL of Congo red dye solution under various conditions (pH, contact time, and initial concentration) and then shaken using an orbital shaker at 200 rpm. After the adsorption process, the solution was centrifuged for 2 minutes at a speed of 1000 rpm, thus separating the solid from the solution. The dye concentrations after adsorption were determined using a UV-Vis spectrophotometer at the maximum wavelength according to the pH conditions of each solution. The concentration of dye obtained was calculated for its adsorption capacity by the following equation:

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

Where the adsorption capacity or the number of dye molecules adsorbed at equilibrium (mg/g) is denoted as q_e, C_o and C_e are the adsorbate concentration (mg/L), m is the mass of adsorbent (g), and V is the adsorbate volume (mL) [20].

3. Results and Discussion

3.1. Characterization of Magnesium Hydroxide

The results of magnesium hydroxide characterization are shown in Figure 3. Figure 3(a) is the diffraction pattern obtained in the experiment. From the figure, it can be seen that the diffraction peaks are at 2θ: 18.80° (001); 37.99° (101); 50.89° (102); 58.71° (110); and 62.17° (111). The resulting diffraction peaks are similar to those of the reference and JCPDS No. 44-1482, indicating that the synthesized $Mg(OH)_2$ has high purity. Furthermore, Figure 3(b) shows the IR spectra with the interpretation offered in Table 1. Figure 3(c) shows the surface morphology of the $Mg(OH)_2$ sample: small, non-uniform grains.

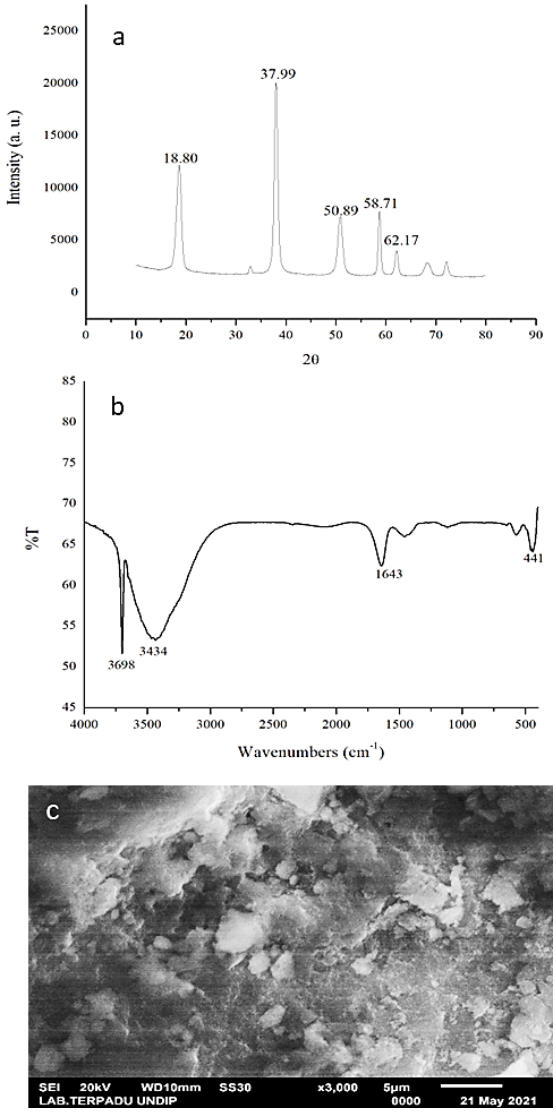

Figure 3. (a) diffraction pattern (b) IR spectra (c) surface morphology of $Mg(OH)_2$ synthesized from seawater bittern

Table 1. Interpretation of the infrared spectrum of $Mg(OH)_2$ from bittern

Absorption frequency area (cm^{-1})		Interpretation
Result	1/λ	
3698	3698	Stretching vibration O-H from $Mg(OH)_2$
3434	3432	Bending vibration H-O-H
1643	1641	Bending range vibration of the molecule H_2O
441	440	Range vibration MgO

3.2. Effect of pH

The wavelength of the CR solution was determined at various desired pH variations before the adsorption process. The degree of acidity (pH) of the solution plays an essential role in the adsorption process, affecting the ionic form of the adsorbed dye and the surface charge of the adsorbent. Therefore, pH can encourage or inhibit the adsorption process of a solution [21]. A total of 25 mL of CR dye with a concentration of 20 mg/L at a certain pH was adsorbed using 0.01 g of $Mg(OH)_2$ for 60 minutes in an orbital shaker at a speed of 200 rpm. After the adsorption process, the absorbance of CR was determined using a UV-Vis spectrophotometer at the maximum wavelength that had been obtained. The results of the adsorption of CR dyes at varied pH are shown in Figure 4.

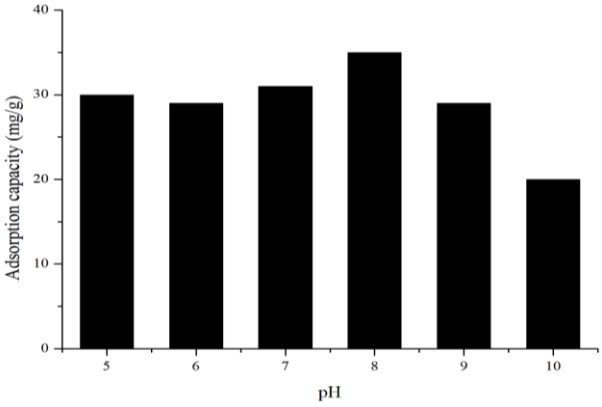

Figure 4. Adsorption of CR dye at various pH

The results showed that the optimum absorbance of CR was at pH 8 and a wavelength of 499 nm. The adsorbed CR was 34.57 mg/g. This is in line with previous studies on the synthesis of MgO and its adsorption properties. In that study, it was suggested that the adsorption of ionic dyes might be related to isoelectric attraction and surface complexation. MgO has an isoelectric point of 12.1-12.7, not significantly different from $Mg(OH)_2$, which has an isoelectric point of 12 [22, 23]. When the isoelectric point of the adsorbent is above the pH of the CR solution, the adsorbent will have more positively charged surfaces, absorbing more anionic dye molecules. The positive charge on the adsorbent is related to its structure, where $Mg(OH)_2$ has a solid form called brucite. Brucite $Mg(OH)_2$ is a layered hydroxide with the simplest structure, consisting of Mg^{2+} at octahedral sites surrounded by six hydroxyl groups. Therefore, $Mg(OH)_2$ readily adsorbs CR dye at pH 8. For the next experiment, the optimum

adsorption was carried out at pH 8 and the optimum wavelength of 499 nm.

3.3. Effect of Contact Time

Determination of contact time aimed to discover the time required for CR dyes to achieve optimum adsorption when adsorbed on $Mg(OH)_2$. Generally, a longer contact time will result in a higher amount of adsorbate being adsorbed because more and more adsorbent particles interact with the adsorbate [10]. The adsorption of CR dye was carried out at contact times of 15, 30, 45, 60, 75, 90, and 120 minutes with an orbital shaker speed of 200 rpm. Adsorption was implemented at pH 8, with an initial concentration of 20 mg/L in 25 mL and 0.01 g of $Mg(OH)_2$. The connection between adsorption capacity and contact time is presented in Figure 5.

Figure 5 shows that the adsorption capacity of CR has increased with increasing contact time. The optimum contact time was reached at 90 minutes with an adsorption capacity of 38.37 mg/g. In the early stages of the adsorption process, many unoccupied active sites are still on the surface of the adsorbent particles, which allows the adsorbed adsorbate molecules to remain present at the active site, thus increasing the adsorption capacity [24].

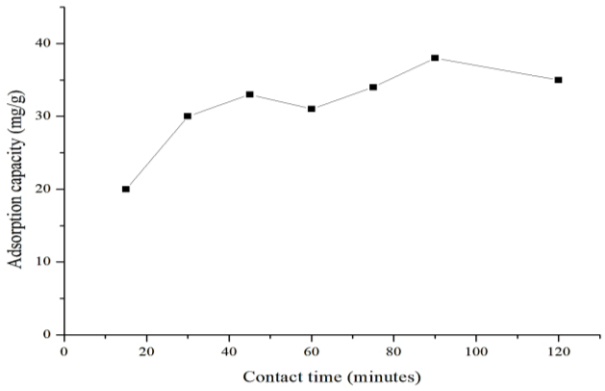

Figure 5. Effect of contact time on adsorption of CR dye

3.4. Effect of Initial Concentration

The initial concentration was determined to ascertain the optimum concentration of CR when it is adsorbed by $Mg(OH)_2$. The adsorption of CR was performed on an orbital shaker at 200 rpm for 90 minutes of contact time and pH 8 to study the effect of initial concentration. The initial concentrations of the CR solution were 5, 10, 15, 20, 25, and 30 mg/L. The results of the initial concentration variations are shown in Figure 6.

Figure 6 (a) shows that the adsorption capacity has not yet reached the optimum value at the initial concentration variation. In contrast, previous studies on the adsorption of CR dye on $Mg(OH)_2$ adsorbent revealed that 1820 mg/g was the optimal adsorption capacity. Adsorption capacity increases with increasing CR concentration, but the adsorption percentage decreases [15]. The adsorption capacity increases with increasing initial concentration because mass transfer between the dye solution and the adsorbent surface is encouraged at higher concentrations and increases the number of collisions between the dye molecules and the adsorbent.

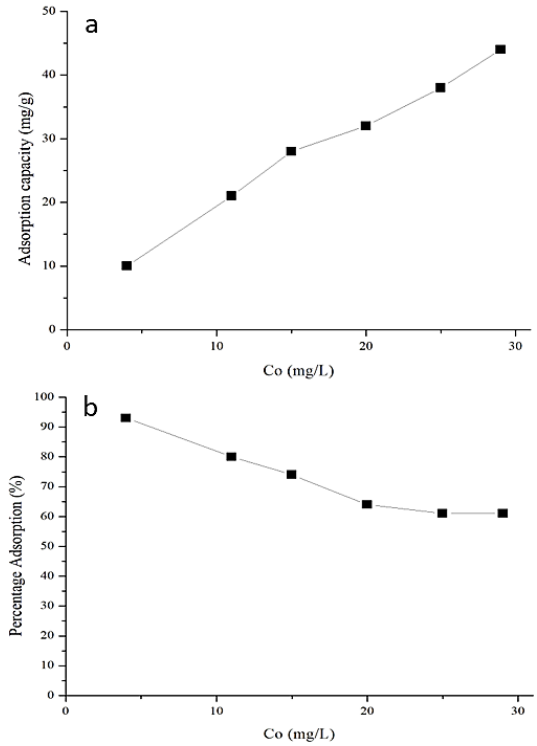

Figure 6. (a) adsorption of CR dye at various initial concentrations (mg/g) (b) adsorption percentage of CR (%)

The active site on the surface of the adsorbent is more at a lower solution concentration when compared to a higher solution concentration. The color removal was more effective when the solution concentration was low because more dye molecules were adsorbed on the adsorbent surface. At higher solution concentrations, the dye molecules must compete with one another for the active sites on the adsorbent surface, causing some dye molecules not to be adsorbed and remain in the solution [25].

3.5. Adsorption Isotherms

The adsorption isotherm model describes the interaction between the adsorbent and the adsorbate. Langmuir and Freundlich's isotherm model was used to evaluate the data rather than adsorption [25]. The adsorption isotherm determines the type of adsorption that occurs by analyzing the magnitude of the linear regression value generated by the graph on each isotherm so that the characteristics of the adsorption system between the solution and the adsorbent surface can be identified [26, 27]. The study of adsorption isotherms was produced by varying the initial concentration of CR dye from 5 to 30 mg/L at obtained optimum pH and contact time. Langmuir and Freundlich isotherms can be calculated according to equations (2) and (3), respectively:

$$\frac{C_e}{q_e} = \frac{C_e}{Q_m} + \frac{1}{Q_m K_L} \quad (2)$$

$$Log\ q_e = Log\ K_f + \frac{1}{n}\ Log\ C_e \quad (3)$$

Where q_e is the number of dye molecules adsorbed at equilibrium (mg/g), C_e is the equilibrium concentration of adsorbate (mg/L), K_L and K_f are Langmuir's constants

(L/g) and Freundlich's constants (mg/g), Q_m is the maximum capacity of the adsorbent (mg/g), and n is the adsorption intensity [28]. Based on equations (2) and (3), the linear curve of the Langmuir and Freundlich isotherm model on the adsorption of CR dye using $Mg(OH)_2$ adsorbent is presented in Figure 7. Based on the slope and interseptal values in Figure 7, the adsorption isotherm parameter values can be seen in Table 1. The data from Tabel 1 shows that the correlation coefficient R^2 for the Freundlich equation is higher than the Langmuir model. This indicates that the adsorption pattern occurring on $Mg(OH)_2$ with CR dye is multilayer with the Freundlich adsorption isotherm model.

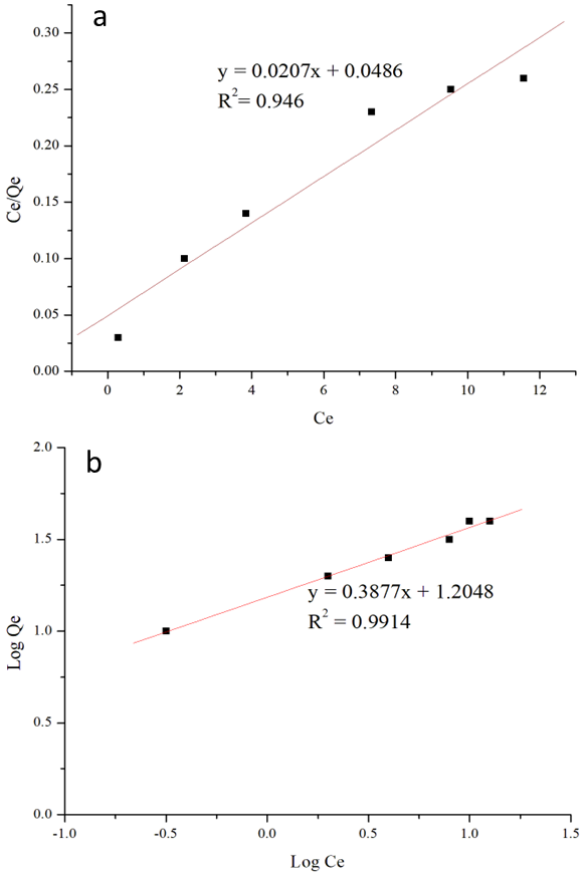

Figure 7. Langmuir (a) and Freundlich (b) adsorption isotherms of CR

Table 1. Results of adsorption isotherm parameters

Adsorption Isotherm Parameters			
Langmuir	Results	Freundlich	Results
R^2	0.946	R^2	0.991
Q_m	48.300	n	2.579
K_L	0.426	K_f	16.020

3.6. Adsorption Kinetics

Adsorption kinetics aims to understand and investigate the mechanism of the process that controls the rate of adsorption. It is used for the selection of optimum operating conditions. The pseudo-first-order and pseudo-second-order are the most widely used models to study the adsorption kinetics of dyes and measure the rate of adsorption [29]. The adsorption kinetics of CR dye with $Mg(OH)_2$ from seawater bittern was tested using the Lagergren pseudo-first-order model, as in equation (4), and the pseudo-second-order Ho shown in equation (5).

$$Log(q_e - q_t) = Log\ q_e - \frac{k_1}{2.303}t \quad (4)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \quad (5)$$

Where q_e and q_t are adsorption capacity at equilibrium and at time t (mg/g), k_1 and k_2 are rate constants of pseudo-first-order and pseudo-second-order [30, 31]. Based on equations (4) and (5), the kinetic curve of CR adsorption with the $Mg(OH)_2$ is presented in Figure 8. From the slope and interseptal values in Figure 8, it can be concluded that the constants k_1, k_2, and q_e are listed in Table 2.

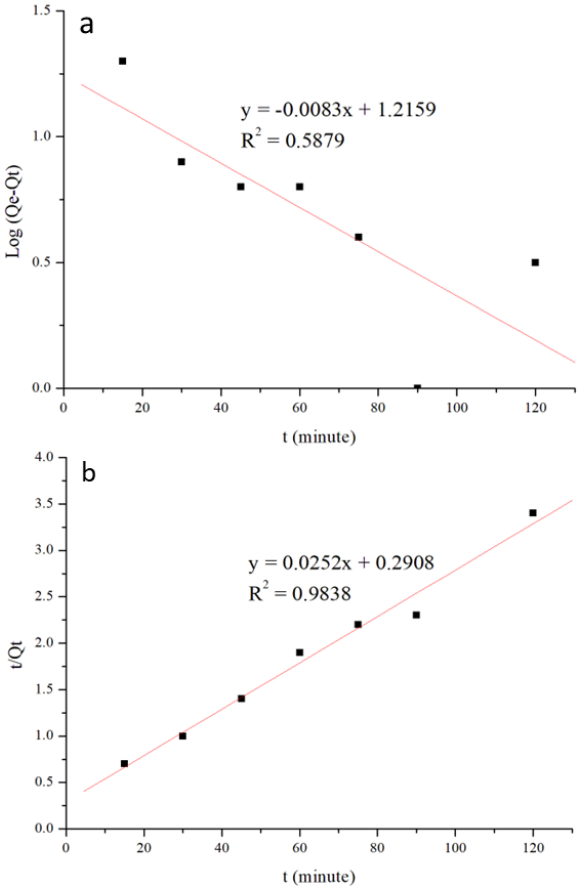

Figure 8. The curves of (a) pseudo-first-order (b) pseudo-second-order

Table 2. Calculation results of adsorption kinetics parameters

	Orde 1	Orde 2
R^2	0.588	0.984
k_1 (min^{-1})	0.019	-
k_2 ($g\ mg^{-1}\ min^{-1}$)	-	0.002
q_e ($mg\ g^{-1}$)	16.440	36.680

Based on the analysis of the adsorption kinetics data in Table 2, it is known that the adsorption kinetics model suitable for showing the CR dye adsorption process is ideal for the pseudo-second-order model with a correlation coefficient (R^2) of 0.9838. Similar results were found that the CR adsorption was best explained by a pseudo-second-order model [15]. The value of R^2 in

pseudo-second-order shows that the adsorption occurs chemically, forming chemical bonds between the adsorbate molecule and the adsorbent surface [30].

4. Conclusion

The diffraction peak characteristics of $Mg(OH)_2$ synthesized from seawater bittern were similar to those of JCPDS No. 44-1482, with O-H and H-O-H functional groups and inhomogeneous small grain morphology. The synthesis results showed that $Mg(OH)_2$ is highly pure. The adsorption of CR dye on $Mg(OH)_2$ synthesized from bittern resulted in an adsorption capacity of 46.3 mg/g at an optimum pH 8, 90 minutes of contact time, and an initial concentration of 29 mg/L. The initial concentration variation has not yet reached the optimum adsorption capacity. The Freundlich isotherm pattern showed that the adsorption pattern of CR dye is multilayer. The results of the adsorption kinetics on $Mg(OH)_2$ followed a pseudo-second-order kinetics pattern. These results conclude that $Mg(OH)_2$ synthesized from bittern is an effective adsorbent for removing CR dyes in aqueous solutions.

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