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Research Paper

Performance of Graphite for Congo Red and Direct Orange Adsorption

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Abstract

To investigate its ability, graphite material was characterized by X-Ray diffraction (XRD), Fourier Transform Infrared Spectrophotometer (FTIR), Thermo Gravimetry Differential Thermal Analyse (TG-DTA), and Brunauer Emmet Teller (BET). Adsorption capability was evaluated by using Congo Red dye and Direct Orange dye solutions. The experiment was studied by using kinetic, isotherm, and thermodynamic parameters. The result of XRD characterization showed that graphite has four characteristic peaks which are 26,405°(002), 44,50°(101), 54,45°(004), and 77,32°(006). FT-IR characterization showed that the vibration at 3432 cm^{-1}, 1635 cm^{-1}, and 1381 cm^{-1}. TG-DTA analysis showed that graphite has only one decomposition peak at 760 °C. BET analysis showed that the surface area of graphite was 11,558 m^2/g. The optimum pH of CR and DO dyes were in pH 6 and pH 2 with contact time of 120 minutes. Kinetic adsorption model of CR and DO dye onto graphite follow pseudo-second-order model with a liner regression coefficient close to one. Isotherm parameter of CR and DO adsorption onto graphite follow Langmuir isotherm model with spontaneous endothermic process. The desorption process of graphite was used HCl to remove the dyes. Regeneration process of graphite showed that the capacity of adsorption decreased after it was reused 3 times.

Keywords
graphite, characterization, adsorption, dyes

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1. INTRODUCTION

In recent years, the development of world industry give its impact on the environment to the worst. The changed in color of water became a sign of water quality which became lower than before. It commonly can be caused by too much utilization of dyes so that water chemical contain increased.

Dye is a synthetic chemical contained which contained unsaturated organic substance, chromophore as the color agent, and ausochrome as the binding agent for fiber (Naimah et al., 2014; Agustina and Amir, 2012). It has aromatic structure (Saraswati et al., 2015), non-degradable, and stable (Gupta et al., 2005). Besides that, dyes also can be toxic (Prameswari et al., 2013) and carcinogenic (Sari et al., 2017), especially for humans. One of the examples is congo red and direct orange. Congo red dye has a high toxic degree so that it can be poison for other living creatures (Mulyaningtias and Syafiq). Direct orange also is one of dyes which can caused allergy, skin irritation, cancer, and mutagenic diseases to living organisms (Anouzla et al., 2009). It was mostly resistant to biodegradation (Safa and Bhatti, 2011). This explanation showed that dyes can caused water pollution which can be andgerous for human living.

Some methods can be used for solving water pollution (Risna, 2013). The methods can be carried out physically or chemically, such as filtration, reduction, electrochemical treatment, ion exchange, coagulation, flocculation, membrane separation, and adsorption (Farda, 2013; Yaacoubi et al., 2014; Taher et al., 2018). Having a complicated processes and expensive costs (Ruan et al., 2016) became a consideration to choose the suitable method so that adsorption became the suitable method which can be easily used for solving water pollution (Herlina et al., 2017). Adsorption is a process in which gas or liquid molecules come in contact with a solid surface (adsorbent) or adsorbed on the surface due to physical force (Suryawan, 2004). Adsorption is the simplest method which is the easiest to implement. Besides that, this method also is kind of inexpensive and effective method which can removed the pollutants (Futalan et al., 2011).

Nowadays, many materials were investigated as an adsorbent, one of them was graphite. Graphite is a carbon-rich porous solid material with high surface area, porosity levels, and a stable carbon matrix. Generally, this mate-

rial can be obtained from the thermal pyrolysis process of carbon-containing biomass under limited oxygen conditions (Fessenden and Fessenden, 1986; Li et al., 2016; Gholami et al., 2020). It has a two-dimensional hexagonal structure that has good electrical and thermal conductivity (Ciesielski and Samori, 2014) (Geng et al., 2012). The advantages of this material were generally renewable, cheap, environmentally friendly, has excellent stability, and can be used as a sustainable adsorbent (Li et al., 2016; Xue et al., 2016).

These reasons have led to graphite being extensively developed in materials science, especially as an adsorbent (Xue et al., 2016). In this work, graphite was applied as congo red and direct orange removal agents. The physicochemical of graphite was characterized by XRD, FTIR, thermalgravimetric, and surface area using BET method. The adsorption study was determined using kinetic, isotherm, thermodynamic, and regeneration study.

2. EXPERIMENTAL SECTION

2.1 Material

The chemicals were provided from Merck and Sigma-Aldrich such as fabricant graphite, sodium hydroxide (NaOH), hydrogen chloride (HCl), Congo Red (CR) dye, Direct Orange (DO) dye, and aquadest. XRD analysis was performed using the Rigaku Miniflex X-Ray diffractometer. BET surface area analysis was conducted Quantachrome adsorption-desorption apparatus. FTIR analysis was carried out using Shimadzu Prestige-21 at wavenumber 400-4000 cm^{-1} wavenumbers. Dyes concentration analysis was performed using a UV-Vis Biobase spectrophotometer.

2.2 Determination of Maximum Wavelength

As much as 20 mL dye solution 50 mg/L was measured by UV-Vis spectrophotometer. The measuring was investigated on 450 -700 nm at normal pH. The maximum wavelength for CR dye and DO dye were 497 nm and 500 nm.

2.3 Adsorption Direct Orange and Congo Red Experiment

2.3.1 Optimum pH

0.02 g graphite was added into 20 mL CR dye 50 mg/L. The adsorption studies was carried out by optimize pH parameters. pH value for adsorption studies was adjusted from 2-11 by added 0,1 M HCl or 0,1 M NaOH solution. The adsorption process was carried out with a magnetic stirrer for 2 hours. After that the dyes solution was measured by UV-Vis using maximum wavelength of dyes to determine optimum pH. This experiment was also done for DO dye solution.

2.3.2 Optimum Contact Time

0.02 g graphite was added into 20 mL CR dye 50 mg/L which was already adjusted optimum pH. Contact time of adsorption parameter were adjusted from 0 – 180 minutes. The solution of dyes was measured by UV-Vis to get the equilibrium time of adsorpstion. This experiment was also done for DO dye solution.

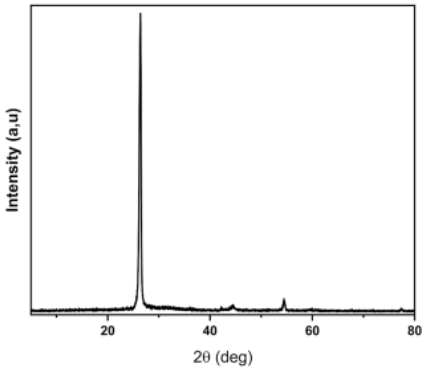

Figure 1. X-ray Powder Diffraction Patterns of Graphite

2.3.3 Optimum Consentration and Temperature

Consentration and temperature of adsorption paramenter was studied by adding 0.02 g graphite into 20 ml dye with concentration 50, 75, 100, 125, and 150 mg/L. The pH value was adjusted optimum pH at temperature 303 – 333K. the mixture then was stirred by magnetic stirrer in optimum contact time. Then the filtrate solution was measured by UV-Vis.

2.3.4 Desorption and Regeneration Direct Orange

The desorption process was studied to determine adsrobent efficiency. 0,05 g graphite was added into 50 ml dye 100 mg/L and stirred during 120 min. Then, the filtrate was separated and measured by UV-Vis spectrometer. After that, the adsorbents was dried and 0,01 g residue was shaken into 10 mL solvents for 120 min. The filtrate was measured by UV-Vis spectrometer. The regeneration studies was carried out by the adsorption-desorption process in three circles.

3. RESULTS AND DISCUSSION

3.1 Characterization of Composite Ca-Al/Graphite

The characterization of graphite was investigated by XRD, FT-IR, BET, and TG-DTA analysis. The XRD pattern of graphite was shown in Fig 1. The XRD spectrum of graphite showed four distinct characteristic diffraction peak at 26,405°(002), 44,50°(101), 54,45°(004) and 77,32°(006). Peak 26,405°(002); 54,45°(004) and 77,32°(006) were reflection of polyarene layers and peak 44,50°(101) as the reflection of determined the longitudinal dimension La of the structural elements (Popova, 2017). Peak 26,405°(002) also showed that graphite has an amorphous structure (Yunasfi, 2017).

Fig 2. shown FTIR analyse of graphite by using spectrophotometer IR instrument. The board vibration at 3402 cm^{-1} was indicated hydroxyl group in graphite. The lower

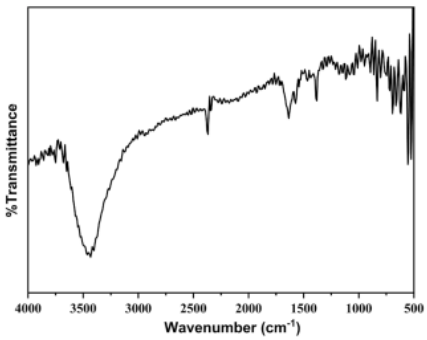

Figure 2. FT-IR Spectrum Of Graphite

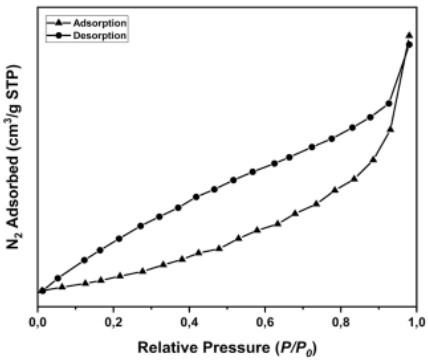

Figure 3. Adsorption-Desorpstion of Graphite

vibration at 1635 cm^{-1} is presences of OH bending. The vibration at 1381 cm^{-1} indicated the =C–H at graphite.

The adsorption-desorption isotherm of graphite were investigated by the adsorption-desoprtion isotherm of nitrogen gase at liquid nitrogen temperature. Fig 3 shown that the adsorption-desoprtion curve of graphite belongs to type IV and H3. This curve has a hyteresis which indicated mesoporous material. This kind of hyteresis is given by adsorbents with slit-shaped pore. The calculation of surface area BET and BJH were listed in Table 1.

Fig 4 provided the thermalgravimetric of graphite. This analysis was investigated by thermal analyzer at 20°C-900°C temperature using nitrogen gase. Graphite material has only one decomposition peak at 760 °C. This decomposition peak was denoted that graphite was pure graphite without other ingredients, include water. All of the graphite was decomposed be oxide.

3.2 Adsorption Study of Graphite

3.2.1 Determination of Optimum pH

The adsorption study of CR and DO onto graphite was investigated using several variables such as adsoption pH, contact time, dye concentration and adsorption temperature. Based on the experiment of adsorption pH variable using

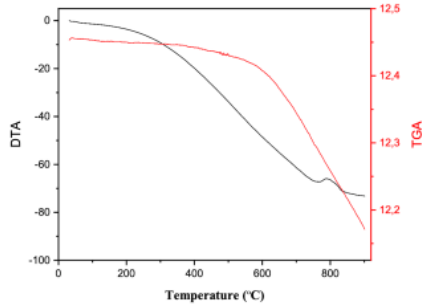

Figure 4. Thermalgravimetic Pattern Of Graphite

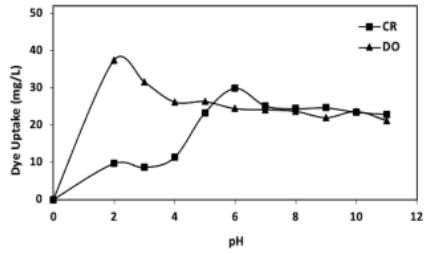

Figure 5. Graph of Optimum pH CR and DO Dyes

0,2 g graphite into 50 mg/L dyes which was stirred during 2 hours. The pH variation was adjusted from 2 – 11. Fig 5 showed the graph of pH variation. The optimum pH of CR dye was obtained in pH 6. Whereas the optimum pH of DO which can be reached was in pH 2. This optimum pH was used to determined the effect of contact time which calculated using kinetic parameter.

3.2.2 Effect of Contact Time of Graphite Adsorption

The experiment of contact time was conducted in initial concentrations 10, 50, 100, 150, 200 mg/L. As much as 0,2 g graphite was added into 20 mL dyes solution which haa been adjusted to optimum pH. The mixture was stirred by variation time 0, 10, 20, 30, 40, 50, 60, 70, 80, 90, 120, 150, 180, and 200 minutes. Fig 6 showed that the dye uptake increased rapidly with the time and the concentration. Both of CR and DO dyes reached 7 equilibrium after 120 minutes.

The kinetic adsorption was determined by pseudo first order (PFO) and pseudo second order (PSO). Therefore to predicted the mechanism process of graphite were used like the following equation :

$$Log(qe - qt) = logqe - \left(\frac{k_1}{2.303}\right) t \quad (1)$$

$$\frac{t}{qt} = \frac{1}{k_2 qe^2} + \frac{1}{qe} t \quad (2)$$

Table 1. Morphology Analysis of Graphite Using BET and BJH Method

Adsorbent	Surface Area (m2/g)	Volume Pore (BJH) (cc/mg)	d-Pore (BJH)(nm)
Graphite	11.558	0.027	3.169

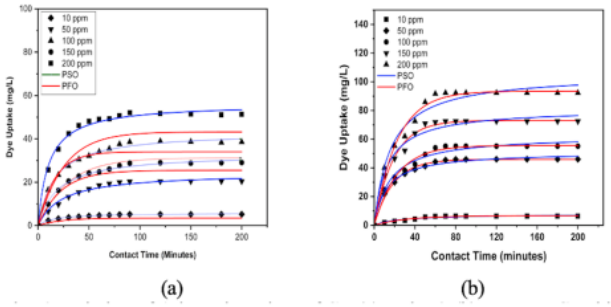

Figure 6. Variation of Adsorption Time of CR (a) and DO (b) Dyes on Graphite

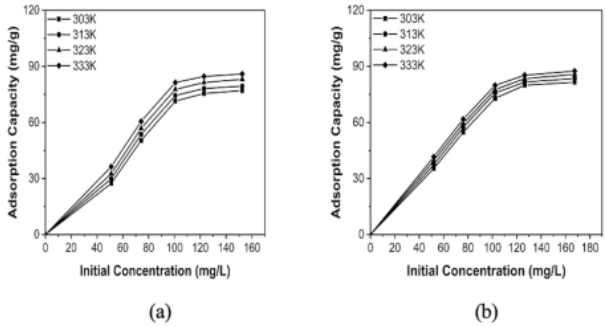

Figure 7. The Effect of Concentration And Temperature of CR (a) and DO (b) Dyes Adsorption onto Graphite

where qe is dye adsorbed capacity at equilibrium (mg/g), qt is the dye adsorbed capacity at time t (min), k is rate constant of PFO and PSO. The correlation of rate constant were determined using these equations and the results were listed in Table 2.

Table of kinetic parameter showed that correlation coefficient of CR and DO dyes adsorpstion in every concentration were belongs to PSO. The adsorbed dye capacity calculation (Qe_{scalc}) which was closed to adsorbed dye capacity experiment ($Qe_{experiment}$), was also became data support to determind kinetic parameter. PSO kinetic model adsorption was denoted that the electrostatic attraction from adsorbate and adsorbent were happened. In addition, the enhancement of rate constant indicated that the adsorbate molecule became reactive.

3.2.3 Effect of Concentration and Temperature of Graphite Adsorption

The effect of concentration and temperature of graphite adsorption were displayed in Fig 7. The data showed that the enhancement of concentration and temperature can also increased the adsorption capacity of dyes.

The effect of equilibrium concentration was predicted by isotherm model of adsorption. It was determined by Langmuir and Freundlich isotherm model. The Langmuir isotherm model described a monolayer adsorption process at homogeneous surface. This isotherm model also described the intreaction of adsorbate and adsorbent. The isotherm model can be predicted using these equations below :

$$\frac{Ce}{qe} = \frac{1}{qmax}Ce + \frac{1}{qmax}KL \quad (3)$$

$$Lnqe = LnKF + \frac{1}{n}LnCe \quad (4)$$

where qmax and KL are Langmuir constant, KF and n are Freundlich constant. The favorable adsorption process can be invetigated by n value. It also verified the type of adsorption process. Table 3 showed the result of calculated parameter. The result obtained that correlation coefficient of isotherm was closed to Langmuir model and Freundlich model. It denoted that the adsorption process was happened monolayer. The higher capacity of adsrobed CR and DO dyes were in temperature 333K which were 87.719 mg/g and 94.251 mg/g.

The effect of adsorption temperature was described by the thermodynamic parameter study, such as enthalpy (ΔH), entropy (ΔS) and Gibbs energy (ΔG). Most of adsorption condition was depended on temperature. Enthalpy (ΔH) value determined exothermic and endothermic process in adsorption. Gibbs energy (ΔG) was used to determined the spontaneity and feasibility. The thermodynamic parameter was calculated by these equations below:

$$inKL = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \quad (5)$$

$$\Delta G = \Delta H - T\Delta S \quad (6)$$

where T was temperature and R was the gas constant. The thermodynamic parameter was listed in Table 4 and Table 5. The Gibbs energy value on the data was negative

Table 2. Kinetic Parameters of CR And DO Dyes Adsorption onto Graphite

Dye	Initial Concentration (mg/L)	$Qe_{experiment}$ (mg/g)	PFO Qe_{Calc} (mg/g)	R^2	k_1	PSO Qe_{Calc} (mg/g)	R^2	k_2
Congo Red	10	5.227	4.913	0.976	0.039	5.662	0.998	0.013
	50	30.682	42.953	0.972	0.049	34.602	0.993	0.001
	100	38.561	37.222	0.954	0.039	41.842	0.997	0.001
	150	43.636	34.857	0.893	0.033	48.076	0.997	0.001
	200	52.326	44.781	0.975	0.051	53.191	0.998	0.003
Direct Orange	10	6.5	11.833	0.809	0.069	7.204	0.978	0.008
	50	46	81.846	0.919	0.076	48.076	0.997	0.002
	100	55.111	51.784	0.965	0.04	58.479	0.994	0.001
	150	72.444	104.399	0.926	0.077	75.757	0.996	0.002
	200	92.222	145.747	0.819	0.072	98.039	0.993	0.001

Table 3. Isotherms Parameter of Adsorption CR and DO Dyes onto Graphite

Dye	Adsorption Isotherm	Adsorption Constant	T(K) 303	313	323	333
CR	Langmuir	Qmax	81.301	82.645	86.207	87.719
		Kl	0.267	0.367	0.411	0.648
		R^2	0.999	0.999	0.999	0.999
	Freundlich	n	1.918	1.543	1.642	2.232
		Kf	1.914	1.507	1.517	1.532
		R^2	0.941	0.93	0.971	0.967
DO	Langmuir	Qmax	92.593	93.458	94.162	94.251
		Kl	0.094	0.121	0.142	0.182
		R^2	0.985	0.991	0.994	0.996
	Freundlich	n	0.797	0.869	1.042	1.207
		Kf	1.094	1.82	3.714	6.305
		R^2	0.964	0.954	0.959	0.953

which means that the adsorption process was spontaneous. The decreasing of Gibbs energy indicated that the adsorption process was more spontaneous and favored in high temperature. Entropy value described the randomness of the adsorption process due to solid and solution interface. The positively value of enthalpy denoted the endothermic adsorption.

3.3 Desorption and Regeneration of Graphite

Desorption of dyes was studied by using some solvents which have different characteristic. They were water, HCl, NaOH, acetone and ethanol. Figure 8 showed the suitable solvent which can be used to desorped dyes. Based on the data, HCl was the best solvent which can desorped CR and DO dyes onto graphite. HCl solvent has like dissolved like effect to the dyes so that this solvent can soluted the adsorbate onto the adsorben.

The regeneration of graphite was investigated by using HCl three times cycles. The recycling process of graphite adsorption-desorption was illustrated in Figure 9. The data indicated that adsorption ability of graphite was decreased

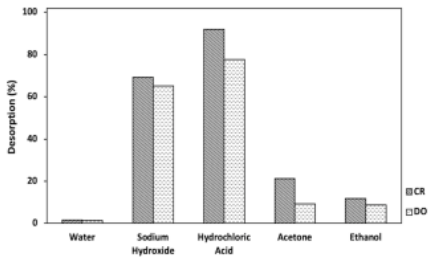

Figure 8. Desorption of CR And DO Dye on Graphite

Table 4. Thermodynamic Parameter of Adsorption Congo Red onto Graphite

Konsentrasi	T (K)	Qe (mg/g)	ΔH (kJ/mol)	ΔS (kJ/mol)	ΔG (kJ/mol)
50 mg/L	303	27.381	41.067	0.137	-0.323
	313	29.921			-1.688
	323	32.619			-3.054
	333	36.349			-4.42
75 mg/L	303	50.429	20.487	0.074	-1.872
	313	53.683			-2.61
	323	56.857			-3.348
	333	60.587			-4.086
100 mg/L	303	71.46	15.16	0.057	-2.194
	313	74.397			-2.767
	323	77.73			-3.34
	333	81.381			-3.912
125 mg/L	303	75.508	9.184	0.034	0.547
	313	78.206			-0.229
	323	81.381			-1.006
	333	84.635			-1.783
150 mg/L	303	77.079	6.696	0.022	-0.032
	313	79.46			-0.254
	323	83.111			-0.477
	333	85.968			-0.699

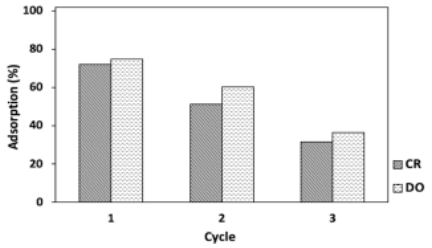

Figure 9. Regeneration of CR And DO Dye on Graphite

from the cycles to next cycles. It can be caused by damaged structure of graphite on the desorption process.

4. CONCLUSIONS

Based on this research, graphite was one of efficient adsorbent which can be used to remove DO then CR in aqueos solution. The XRD characteristic of graphite was indicated that it has an amorphorus structure which is showed by peak 26,405°(002). The surface area of graphite showed that graphite has surface area which is 11.558 m^2/g. The optimum pH of CR and DO dyes adsorptions were at pH 6 and pH 2. The optimum contact time both of dyes were 120 mimutes. The investigation of graphite kinetic parameter showed adsorption mechamism follow PSO where q calculation approached $q_{experiment}$. However, the isotherm adsorption followed Langmuir isotherm model. Desorption process for graphite was good recycled by using HCl as desorbed reagent. In addition, the regeneration process showed that graphite has a weak structure because desorption process caused damaged structure.

5. ACKNOWLEDGEMENT

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Table 5. Thermodynamic Parameter of Adsorption Direct Orange onto Graphite

Konsentrasi	T (K)	Qe (mg/g)	ΔH (kJ/mol)	ΔS (kJ/mol)	ΔG (kJ/mol)
50 mg/L	303	35.385	17.742	0.065	-1.88
	313	37.462			-2.527
	323	39.615			-3.175
	333	41.615			-3.822
75 mg/L	303	54.792	14.355	0.055	-2.374
	313	57.408			-2.926
	323	59.485			-3.478
	333	61.792			-4.03
100 mg/L	303	72.946	9.891	0.04	-2.312
	313	75.792			-2.715
	323	77.638			-3.117
	333	79.869			-3.52
125 mg/L	303	79.946	5.266	0.022	-1.352
	313	81.638			-1.57
	323	83.485			-1.788
	333	85.331			-2.007
150 mg/L	303	81.531	4.041	0.013	0.134
	313	83.531			0.005
	323	85.685			-0.124
	333	87.531			-0.253

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