

Polyurethane Composites for Dye and Heavy Metal Removal: (Adsorption Kinetics and Isotherms Studies)

M.E. Ossman¹ and M. Abdelfattah¹

Abstract—The need to maintain a cleaner environment for the survival of both aquatic and terrestrial lives including human beings is very crucial and is a subject of increasing concern to the environmentalist. Pollution caused by agents such as heavy metals and organic pollutants are amongst the list which rendered the environment unwholesome and posed serious health concern to the populace. The removal of Heavy metals and organic pollutants from wastewater before their discharge in the environmental is very important due to its polluting effect on the underground water. This work dealt with the preparation and characterization of polyurethane foam composite with bentonite and CuO and the usage of these adsorbents in removing Eosin dye (as an organic pollutants) and Cd (heavy metal) from industrial wastewater using a batch operation system. Studying of the operating conditions namely; effects of contact time, dosage of adsorbent, initial concentration as well as effect of pH were carried out. Also kinetics and isotherm study that describe the adsorption process are carried out.

Keywords— Adsorption, Cd, Dye, composite.

I. INTRODUCTION

The need to maintain a cleaner environment for the survival of both aquatic and terrestrial lives including human beings is very crucial and is a subject of increasing concern to the environmentalist. Pollution caused by agents such as heavy metals and organic pollutants are amongst the list which rendered the environment unwholesome and posed serious health concern to the populace [1]. Heavy metals and organic pollutants are toxic, carcinogenic and hazardous for aquatic living organisms [2]. The removal of Heavy metals and organic pollutants from wastewater before their discharge into the environmental is very important due to its polluting effect on the underground water. Many methods have been used to treat the wastewater before discharge into the natural water. Adsorption [6], oxidation [7], microfiltration [8], coagulation [9] and degradation [10] are methods used for the removal of heavy metals and organic pollutants from wastewater. The adsorption methods are the best effective technique which have been successfully applied because of the easy operation, inexpensive, ability to treat waste water, and have the ability to reuse the spent sorbent via regeneration [11]. Several sorbents are applied to remove the heavy metals and organic pollutants from

wastewater e.g. polyurethane [12-16] organo bentonite [17], carbon [18], Zn2Al-NO3 [19] and miswak [20]. Polyurethane foam (PF) is a good sorbent to remove some organic and inorganic pollutants from wastewater [21, 22]. Polyurethane (PU) foams have been used as adsorbents in the treatment of contaminated water and effluent because they are cheap, a high surface area because of their open porous structures and thus can be used as matrices to immobilize various adsorbents and can be used without prior treatment [23]. PU foams are able to retain different classes of substances because of the presence of both polar and nonpolar groups in their structures [14–23]. The high basicity of PF decreases sorption capacity of PF. This problem demands the preparation of low basicity PF with high sorption capacity. In previous studies, some other research developed PF that contains polyhydroxyl functional group which has high sorption capacity [24, 25]. Composite materials composed of polymers and adsorbents show potential for purification applications [26–27]. This work study the possibility of preparing polyurethane foam composite with bentonite and CuO with good stability and high sorption capacity and the applications of using the prepared adsorbents for removing heavy metals and organic pollutants from wastewater.

II. MATERIALS AND METHODS

All chemicals used were of analytical grade.

A. Preparation of Adsorbents

2.1.1. Preparation of CuO: For preparation of CuO; 0.5 gm of copper acetate salt is dissolved in 500 ml of water and Drop of Glacial acetic acid (1 ml) is added. Constant stirring of the solution on a hot plat until temperature reaches 90°C. Then 1.2 g of NaOH is added to above heated solution till pH reaches to 6-7. Leave the mixture at 90°C c for 2 hrs then copper oxide is formed. Centrifuge and wash 3-4 times with distilled water. The obtained precipitate was dried in air for 24 h.

2.1.2. Preparation of Polyurethane (PU): For preparation of Polysurethane; Mixture (1) [350 g castor oil, 100 g glycol, 50 drop silicone oil, 150 drop water and Cap and shake] have been prepared then pour 8.5 ml of mixture (1) in a waxed soft drink cup and stir well .Add 5 ml of Toluene di-isocyanate. The mixture becomes warm and begins to evolve bubbles of carbon dioxide after about 1 min. Collect the foam and grind.

2.1.3. Preparation of CuO Polyurethane Composite (CuO/PU) and Bentonite /PU: Repeat the same procedures that used in the preparation of polyurethane with adding 1 g of

M.E. Ossman, Professor, Petrochemical Engineering Department, Pharos University, Alexandria, Egypt

M. Abdelfattah, Petrochemical Engineering Department, Pharos University, Alexandria, Egypt

CuO/ Bentonite before adding the Toluene di-isocyanate..

III. RESULTS AND DISCUSSIONS

B. Batch experiments

A solution of cadmium sulphate with 1000ppm concentration was prepared and different concentration of Cd solution were prepared (10, 20, 30 & 50 ppm). While A solution of Eosin Yellow dye with 1000ppm concentration was prepared and used to Prepare different concentration of Eosin dye (5, 10, 15, 20 ppm). The determination of the concentration of remaining cadmium ion were taken place by atomic absorption while analyzing the dye solutions were taken place using spectrophotometer at 517 nm. Equilibrium isotherms were obtained by performing batch adsorption studies. Solutions of 250 mL of the required concentration were adjusted to optimum pH values and adsorbents amounts ranging between 1 and 5 g were added to solutions.

The adsorbed cadmium amount/ dye (q_e) per unit adsorbent mass was calculated using the following formula:

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

Where; C_o is the initial heavy metal (Cd)/ Eosin dye concentration, C_e is the concentration of heavy metal/ Eosin dye at equilibrium (mg/L), W is the adsorbent mass (mg) and V is the solution volume (L).

A. Characterization of Adsorbents

In order to know the structure of the adsorbent, scanning electron microscopy (SEM) was employed to visualize sample morphology using a JEOL JSM-5300 Scanning Electron Microscope operated between 15 and 20 KeV. SEM image is presented in Fig. 1 which shows the rectangular morphology with high surface roughness of the particles for CuO and Bentonite (Fig. 1 (a) and (d)). The increase in surface roughness leads to an increase in the surface area of the adsorbent. The particle size distribution for nano particles found to be 33-160 nm for CuO. The morphology of Polyurethane with low surface roughness of the particles Fig. 1(b), the PU/CuO composite Fig.1(c) and Pu/ Bentonite composite Fig. 1(e). The particle size distribution for the particles found to be 220-540 nm for PU, 150-350 nm for PU/ CuO and 200-300 nm for PU/ Bentonite.

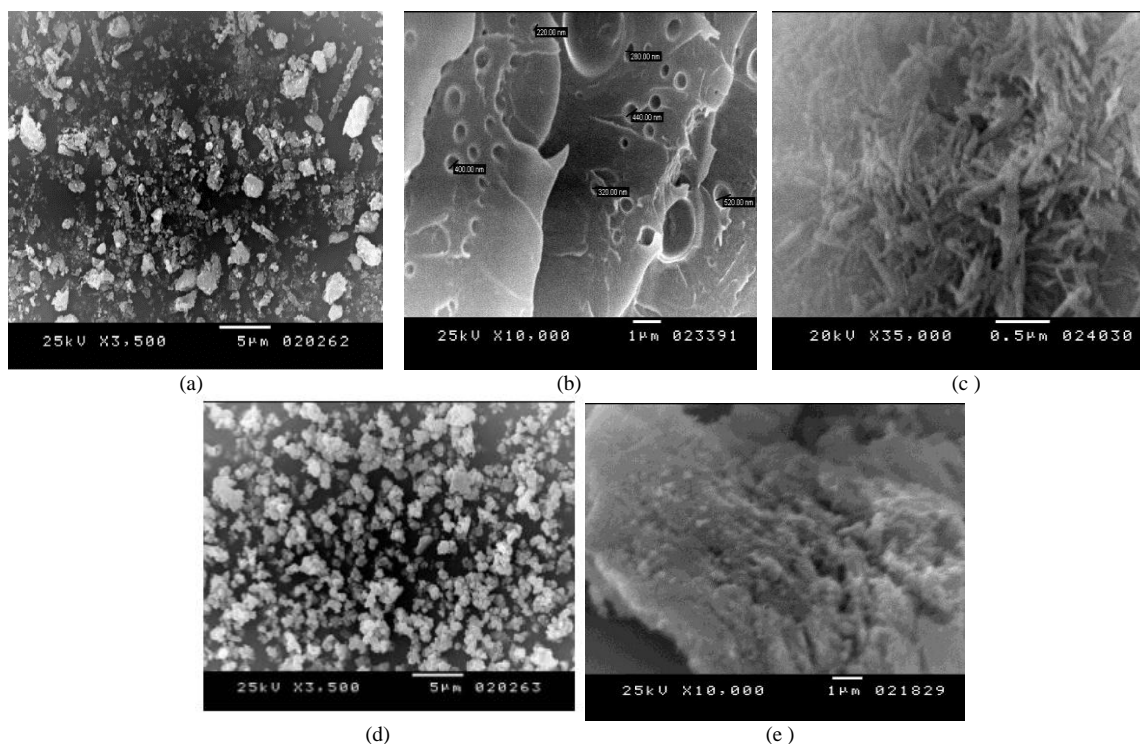


Fig. 1: SEM for (a) CuO nano powder (b) PU (c) CuO/PU composite (d) Bentonite (e) Bentonite/PU composite

B. Effect of contact time

In order to establish equilibration time for maximum uptake and to know the kinetics of adsorption process, the adsorption of cadmium ion and Eosin dye on CuO, CuO/PU, PU and PU/Bentonite as adsorbents were studied as a function of contact time and results are shown in Fig. 2 (a and b). Fig.2 (a) showed that the rate of uptake of the cadmium ion are rapid in the beginning and 79% adsorption is completed using CuO,

73% adsorption is completed using PU/Bentonite, 42% adsorption is completed using Bentonite, 43 % adsorption is completed using PU/CuO and 15% adsorption is completed by using PU for 20 ppm concentration. The time required for equilibrium adsorption is 30 min for 20 ppm cadmium ion concentration. Fig. 2 (b) showed that the rate of uptake of the cadmium ion are rapid in the beginning and 62% adsorption is completed using CuO , 55% adsorption is completed using

PU/Bentonite, 36% adsorption is completed using Bentonite, 13 % adsorption is completed using PU/CuO and 4 % adsorption is completed by using PU for 20 ppm concentration. The time required for equilibrium adsorption is 40 min for 20 ppm Eosin dye concentration. Fig.2(a and b) indicates that the CuO has high adsorption capacity for cadmium ion and Eosin dye followed by PU/Bentonite, followed by Bentonite then PU/CuO while PU has the lowest adsorption capacity and can't be used as adsorbent for neither cadmium ion nor Eosin dye.

C. Effect of pH

The pH of the solution plays an important role in the whole adsorption process and particularly on the adsorption capacity. In this group of experiments, the 20 ppm concentration of Cd ion and Eosin dye, the amount of adsorbents (4 g/l) for Cd removal and (6g/l) for Eosin dye removal and contact time (30

min) were kept constant. The initial pH of solutions varied from 2.0 to 8. Fig. 4 shows the effect of pH on the percentage removal of Eosin dye. The results indicate that the adsorption is higher at acidic conditions rather than the alkaline conditions and reached maximum at pH =4 for CuO, PU, Bentonite, PU/CuO and PU/Bentonite. Fig. 3 shows the effect of pH on the percentage removal of Cd ions. The results indicate that the adsorption is lower at acidic conditions rather than the alkaline conditions. In acidic conditions the surface of the adsorbent is positively charged due to the high concentration of H^+ and the Cd ion also has positive charge so decrease the electrostatic .higher adsorption of Cd ion under alkaline condition, is probably due to the presence of OH^- ions on the surface of adsorbents attracted with the adsorbate (Cd^{+2} ion) for adsorption sites. Since Cd ion can be precipitated under alkaline conditions.

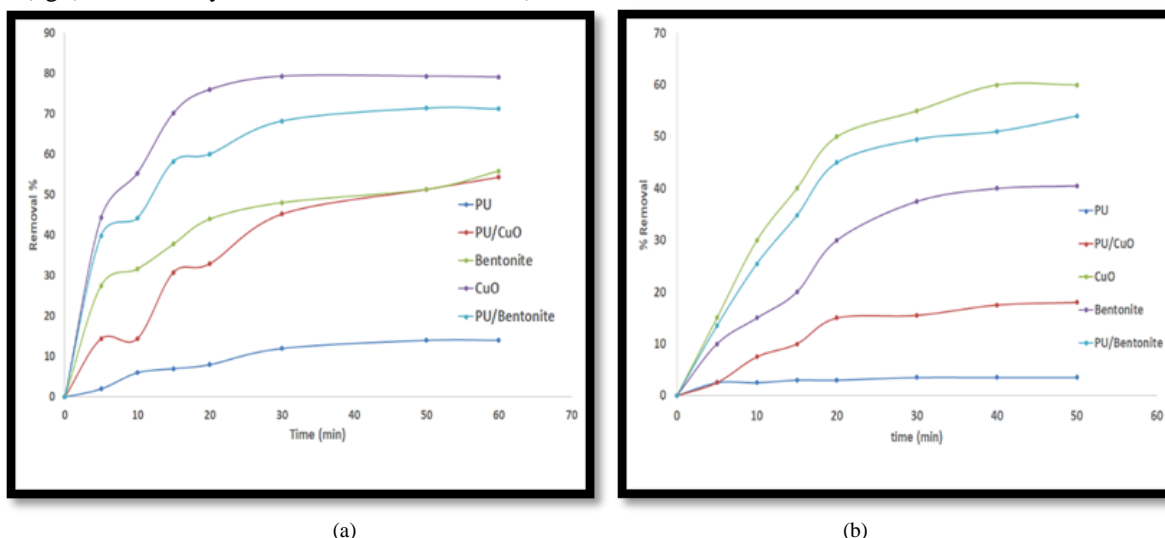


Fig.2: Comparison between the % removal of different type of adsorbents for (a) Cd at pH is 6, adsorbent dosage is 4 g/l, concentration 20 ppm and Temperature is 25 OC. (b) Eosin Dye at pH is 4, adsorbent dosage is 6 g/l, concentration 20 ppm and Temperature is 25 OC.

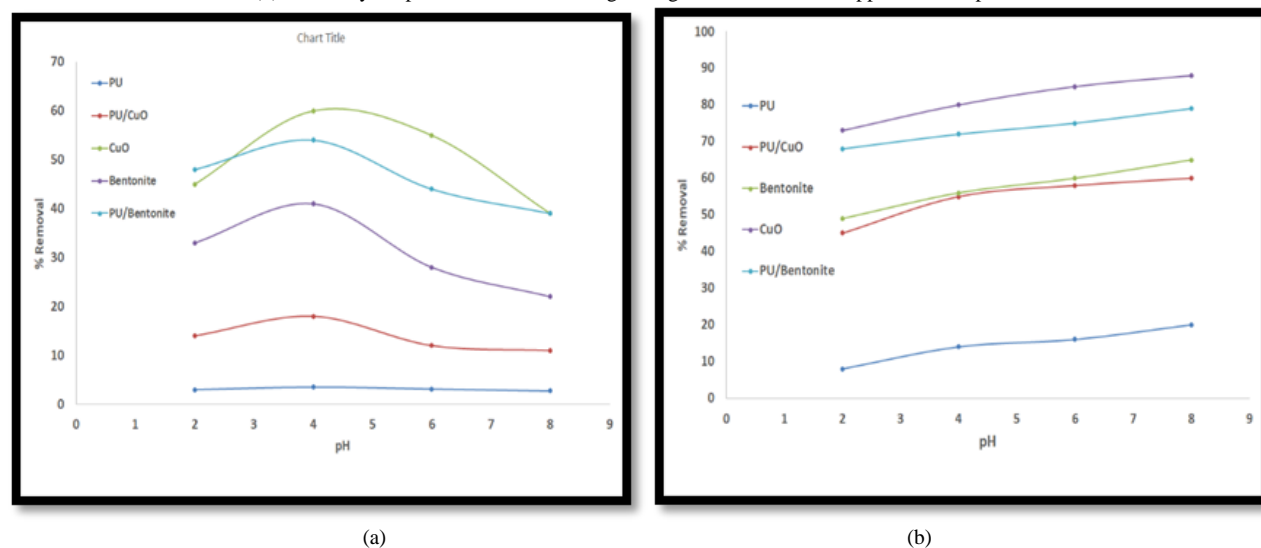


Fig. 3: The effect of pH on % removal of (a) Dye by different adsorbents at initial concentration of 20 ppm and adsorbent dosage is 6 g/l and Temperature is 25 OC. (b) % removal of Cd ions by different adsorbents at initial concentration of 20 ppm and adsorbent dosage is 6 g/l and Temperature is 25 OC.

D. Effect of Adsorbent dosage

The results of the experiments with varying adsorbent dosage are presented in Fig. 4. It was found that with an increase in the

adsorbent dosage from 2 to 6 g/L, the removal of Cd ion and Eosin dye increase by increasing the adsorbent dosage for CuO, PU, Bentonite, PU/CuO and PU/ Bentonite. This is probably due to the greater availability of the exchangeable sites and

surface area.

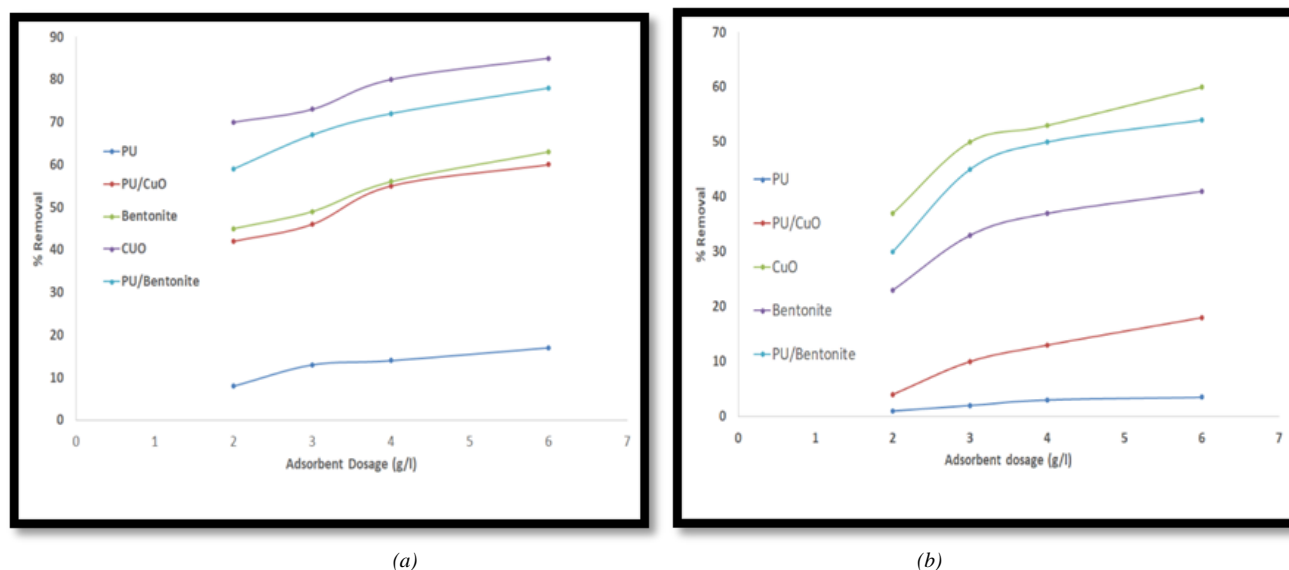


Fig. 4: The effect of Adsorption dosage on % removal of (a) Cd by different adsorbent at initial concentration of 20 ppm, pH 6 and Temperature is 25 OC. (b) Dye by different adsorbent at initial concentration of 20 ppm, pH 4 and Temperature is 25 OC.

E. Equilibrium of Adsorption

Adsorption isotherms used to determine the capacity and optimizing the use of the adsorbent at equilibrium. Therefore, the correlation of equilibrium data by either theoretical or empirical equations is essential to the practical design and operation of adsorption systems.

3.5.1. Langmuir isotherm

The linear form of Langmuir isotherm equation is represented by the following equation (Langmuir, 1918)

$$\frac{C_e}{q_e} = \frac{1}{Q^0 b} + \frac{1}{Q^0} C_e \quad (2)$$

Where Q^0 is the maximum metal ions uptake per unit mass of adsorbent (mg/g) related to adsorption capacity and b is Langmuir constant (L/mol) related to energy of sorption. Therefore, a plot of C_e/q_e versus C_e , gives a straight line of slope $1/Q^0$ and intercept $1/(Q^0 b)$.

3.5.2. The Freundlich isotherm

This empirical isotherm is expressed by the following equation (Freundlich, 1907)

$$q_e = K_F C_e^{1/n} \quad (3)$$

The equation is conveniently used in the linear form by taking the logarithm of both sides as

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (4)$$

Freundlich constants, K_F and $1/n$, are related to adsorption capacity and intensity of adsorption, respectively. The applicability of the Freundlich sorption isotherm and Langmuir isotherm were analyzed, using the same set of experimental data. The data are represented in Table 1. The correlation coefficients reported in Table 1 showed strong positive evidence that the adsorption of Cd ions onto Bentonite and CuO follow the Langmuir isotherm while for PU, PU/Bentonite and CuO/PU; the adsorption of Cd ions showed strong positive evidence that the adsorption follow the Freundlich isotherm. While for Eosin dye the adsorption onto Bentonite, CuO, PU, PU/Bentonite and CuO/PU showed strong positive evidence

that the adsorption follow the Freundlich isotherm, with $1/n$ less than 1.

Kinetic modeling:

Kinetics of adsorption is one of the most important characteristics to be responsible for the efficiency of adsorption. In order to investigate the mechanism of process and potential rate controlling steps, the kinetics of Cd ions and Eosin dye adsorption onto CuO, CuO/PU, PU, Bentonite and PU/Bentonite were analyzed using pseudo-first-order and pseudo-second-order kinetic models. The conformity between experimental data and the model predicted values was expressed by the correlation coefficients (R^2 , values close or equal to 1).

3.6.1 Pseudo-first-order equation

The linear form equation is generally expresses a follows:

$$\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} t \quad (8)$$

Table 1: Isotherm models for the removal of Cd ion and Eosin Dye onto CuO, CuO/PU, PU, Bentonite and PU/Bentonite

3.6.2 Pseudo-second-order equation

The linear equation is generally given as follows:

$$\frac{t}{q_e} = \frac{1}{K_2} \frac{1}{q_e^2} + \frac{1}{q_e} t \quad (9)$$

The linear plot of $\log(q_e - q_t)$ versus t show good agreement between experimental and calculated q_e values (Table 2). The correlation Coefficients for the pseudo first-order kinetic model are greater than 0.98, which led to believe that the pseudo 1st order kinetic model provided good correlation for the adsorption of Cd ions onto CuO/PU and Bentonite, while pseudo 2nd order kinetic model provided good correlation for the adsorption of Cd ions onto CuO, PU and PU/ bentonite. While for the Adsorption od Eosin dye the pseudo 1st order kinetic model provided good correlation for the adsorption onto CuO/PU and Bentonite, while pseudo 2nd order kinetic model provided good correlation for the adsorption of Eosin dye onto CuO, PU/Bentonite and PU.

TABLE I: ISOTHERM MODELS FOR THE REMOVAL OF Cd ION AND EOSIN DYE ONTO CuO, CuO/PU, PU, BENTONITE AND PU/BENTONITE

Isotherm model	CuO		CuO/PU		Bentonite		PU/Bentonite		PU	
	Cd	Dye	Cd	Dye	Cd	Dye	Cd	Dye	Cd	Dye
Langmuir										
Q ⁰ (mg g ⁻¹)	78.74	63.98	12.85	8.67	68.493	49.254	72.523	42.317	8.53	5.65
K _a (L mg ⁻¹)	0.224	0.189	0.125	0.007	0.1081	0.009	0.149	0.108	0.014	0.119
No. of parameter	2	2	2	2	2	2	2	2	2	2
Data point	4	4	4	4	4	4	4	4	4	4
R ²	0.973	0.956	0.2957	0.562	0.938	0.876	0.903	0.887	0.822	0.809
Freundlich										
1/n	0.3352	0.6314	0.695	0.777	0.499	0.246	0.187	0.319	0.798	0.502
K _F (mg g ⁻¹)	22.448	12.111	2.232	1.200	10.367	2.134	18.30	9.034	3.809	0.564
No. of parameter	2	2	2	2	2	2	2	2	2	2
Data point R ²	4	4	4	4	4	4	4	4	4	4
	0.791	0.991	0.787	0.81	0.903	0.895	0.962	0.895	0.863	0.851

TABLE II: KINETICS MODELS FOR THE REMOVAL OF Cd ION AND EOSIN DYE ONTO CuO, CuO/PU, PU, BENTONITE AND PU/BENTONITE

Kinetics models	CuO		CuO/PU		Bentonite		PU/ Bentonite		PU	
	Cd	Dye	Cd	Dye	Cd	Dye	Cd	Dye	Cd	Dye
Pseudo 1st order kinetics										
K ₁ (min)	0.0949	0.0043	0.0622	0.0368	0.0017	0.0002	0.0295	0.0006	0.0105	0.1112
q ₁ (mg/g)	63.453	50.238	11.79	7.231	50.76	54.32	60.142	42.999	4.352	1.352
R ₁ ²	0.794	0.587	0.984	0.874	0.988	0.991	0.794	0.698	0.883	0.785
Pseudo 2nd order kinetics										
K ₂ (g/mg min)	0.0214	0.0157	0.002	0.345	0.048	0.140	0.0129	0.0053	0.1002	0.237
q ₂ (mg/g)	63.694	52.196	16.181	6.231	38.45	43.263	65.583	59.784	2.503	3.006
R ₂ ²	0.999	0.756	0.865	0.762	0.9354	0.897	0.998	0.897	0.956	0.941

IV. CONCLUSIONS

Five different adsorbents are used for removal of Cd ions and Eosin dye from industrial waste water. The results indicated that the CuO has high adsorption capacity for Cd ions and Eosin dye followed by PU/Bentonite and Bentonite then PU/CuO while PU has the lowest adsorption capacity and can't be used as adsorbent for neither Cd ions nor Eosin dye. By studying the effect of pH on the adsorption of both Cd ions and Eosin dye, the results indicated that the adsorption of Cd ions is lower at acidic conditions rather than the alkaline conditions, while the adsorption of Eosin dye is higher at acidic conditions rather than the alkaline conditions and reached maximum at pH =4. The results showed strong positive evidence that the adsorption of Cd ions onto Bentonite and CuO are followed the Langmuir isotherm while for PU, PU/Bentonite and CuO/PU; the adsorption of Cd ions showed strong positive evidence that the adsorption follow the Freundlich isotherm. For Eosin dye the adsorption onto Bentonite, CuO, PU, PU/Bentonite and CuO/PU showed strong positive evidence that the adsorption follow the Freundlich isotherm, with 1/n less than 1. For kinetics study, the results showed that the pseudo 1st order kinetic model provided good correlation for the adsorption of Cd ions onto CuO/PU and Bentonite, while pseudo 2nd order kinetic model provided good correlation for the adsorption of Cd ions onto CuO, PU and PU/ bentonite. While for the Adsorption od

Eosin dye the pseudo 1st order kinetic model provided good correlation.

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