



## Polycyclic Aromatic Hydrocarbon Plucking Out from Industrial Wastewater Using Nano Composite Polymer;(Kinetics and Isotherm Mediation Studies)



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### Abstract

Basic wastewater treatment facilities reduce organic and suspended solids to limit pollution to the environment. The evolution of treatment techniques to remove dissolved materials and harmful compounds has been forced by advances in needs and technology. Currently, advances in scientific understanding and moral awareness have resulted in a decrease in discharges through pollution control and recycling, with the lofty goal of zero pollution discharge. The major purpose of this research was to see how well Polyaniline adsorbents that have been functionalized with Bentonite performed. To synthesize polyaniline/bentonite nano composite (PAN/B), the process relies on induced bentonite during aniline polymerization. SEM, BET, FTIR, and EDAX investigations were employed to recognize the adsorbent, which was then exploited as a possible naphthalene adsorbent for effluent treatment. pH, adsorbent dose, contact time, and starting pollutant concentration were investigated as experimental factors impacting naphthalene sorption onto the PAN/B nanocomposite. Naphthalene was rapidly removed by (PAN/B) that reached 80% removal at 30 min. Also, 81.4% removal of naphthalene using 5g/l of (PAN/B). The pseudo-second-order model best expressed the naphthalene adsorption when the kinetic data were assessed. The equilibrium data was fit to isotherm models, with the Langmuir model delivering the greatest fit to the naphthalene removal experimental data.

*Key words:* Polyaniline; naphthalene elimination; wastewater treatment; kinetics and isotherm

### 1. Introduction

The wastewater contains many pathogenic bacteria, microorganisms, suspended solids, nutrients, minerals, toxic metals etc. For several years the primary goal of wastewater treatment was to reduce the number of suspended solids, oxygen-demanding materials, harmful bacteria, and dissolved inorganic compounds. However, in recent years more stress has been placed on improving the municipal treatment processes for the disposal of solid waste. In traditional wastewater treatment physical, chemical and biological processes are employed to remove organic matter, nutrients and solids from wastewater [1]. In a wastewater treatment plant, the procedure is carried out. Depending on what it was used for, wastewater frequently contains a variety of contaminants. Source categorizes it into two key groups; Sewage from a home or a sanitary facility [2]. Organic pollutants, such as endocrine disrupting chemicals (EDCs), emerging contaminants, persistent organic pollutants (POPs), medicines, and others, can create massive

environmental damage, pose serious ecotoxicological hazards, and have negative environmental consequences [3]. Polycyclic aromatic hydrocarbons (PAHs) were classified as the ninth most dangerous chemical to human health which are organic molecules that include solely carbon and hydrogen and are made up of numerous aromatic rings (electrons that are delocalized)[4]. PAHs have fused aromatic rings, which means they have one or more sides that are connected. PAHs are a type of organic contaminant that is quite abundant that Several compounds in this category were shown to be carcinogenic and mutagenic. PAHs are mostly produced in the petroleum and petrochemical industries. PAHs contamination occurs when oil seeps into ground water or from above-ground holding tanks [5-7]. Some PAHs are classified as known, potentially, or probably carcinogenic to humans by the International Agency for Research on Cancer (IARC). Among these are Benzo[a]pyrene (Group 1), Naphthalene, chrysene, Benz[a]anthracene, Benzo[k] fluoranthene and

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benzo[b]fluoranthene (Group 2B). Inhalation exposure to PAHs is expected to increase the risk of lung cancer the utmost [8]. Naphthalene is the most basic type of PAH (C<sub>10</sub>H<sub>8</sub>). It's a white crystalline solid with a distinct order that may be detected at extremely low concentrations. The polycyclic aromatic hydrocarbon naphthalene is made up of a fused pair of benzene rings [9]. Hemolytic anaemia is one of the most prevalent diseases caused by exposure to high levels of naphthalene, especially in youngsters who often consume naphthalene-containing mothballs or deodorant blocks [10]. Naphthalene can injure or kill red blood cells in the body, resulting in severe anaemia. The National Institute for Occupational Safety and Health has set a limit of 10 mg/L for an 8-hour time weighted average and 15 mg/L for a short-term exposure, respectively [10]. As a result, naphthalene should not be dumped of in waste water. Physical, biological, and chemical techniques are all used in remediation technologies. Residual compounds retrieved or generated throughout treatment operations must be saved a lot and reused or disposed of. Biological and physical-chemical methods have been explored to effectively remove PAHs. The activated sludge process, sequencing batch reactors, and membrane bioreactors are often used in Municipal Wastewater Treatment Plants (MWWTPs) to accomplish aerobic and anaerobic biodegradation of organic pollutants [11-12]. Concerning physical-chemical treatment methods, the most commonly applied to remove PAHs are Membrane filtration (as well as microfiltration, ultrafiltration, nanofiltration, and reverse osmosis) [13-14], adsorption [15-18], advanced oxidation processes (such as ozonation, electrochemical oxidation, Fenton reagents, and UV) [19-20] and chemical precipitation [21].

The goal of this study was to determine the feasibility of conventional waste water treatment techniques in the simultaneous removal of naphthalene as PAHs by adsorption employing polymer nano-composites in order to achieve waste water treatment sustainability that is prepared by addition of bentonite during aniline polymerization process to increase the activity of the adsorbent. Scanning electron microscopy (SEM), powder X-ray diffraction (XRD), Brunauer–Emmett–Teller (BET), Fourier transform infrared (FTIR) spectroscopy, and Elemental analysis (EDAX) were used to evaluate the morphology and structure nanocomposite polymer (PAN/B).

## 2. Materials and methods

### 2.1. Synthesize polyaniline/bentonite nano composite (PAN/B)

Polyaniline was obtained by dissolving 5.0 g aniline in 250 ml HCl(1N) and chilling the mixture with just an ice bath at around 5°C. For 30 minutes, 250 ml of pre-cooled (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub> (0.3M) in HCl

solution (1N) was gently added to the monomer (aniline) solution using vigorous stirring.

5.0 g natural bentonite was combined with 50 mL polyaniline in formic acid in a beaker (100 cm<sup>3</sup>) and swirled for two hours at room temperature before being left for another two hours without stirring to achieve polyaniline/bentonite nano composite (PAN/B). All chemicals used were of analytical grade exported from International Company for Scientific and Medical Supplies Figure (2.1).



Figure (2.1): Adsorbent PAN/B prepared

### 2.2. Characterization

The morphology and structure of polyaniline/bentonite nano composite (PAN/B) was characterized by SEM (JEOL JSM 6360LA) and the crystalline pattern of samples was investigated by XRD (Shimadzu XRD-7000.). The BET surface area was determined by N<sub>2</sub> adsorption/desorption on an ASAP2020 surface area analyzer using the adsorption date branch in the relative P/P<sup>0</sup> pressure range going from 0.0 to 1.0. In addition, the FTIR spectroscopy was used to determine the functional groups of polyaniline/bentonite nano composite (PAN/B) from 400–4,000cm<sup>-1</sup> FTIR- 8,400 S Shimadzu. The elemental composition of the bentonite was determined by EDAX

### 2.3. Batch experiments

Fresh stock solution of naphthalene was prepared every run. Determine the concentration of remaining naphthalene by spectrophotometer at wave length 270 nm.

The mixture in 250-mL Erlenmeyer flasks was kept on a constant agitation shaker (200 rpm at 25°C) to enhance the interaction of the adsorbent and pollutants solution. samples were taken from the solution in predetermined intervals during the stirring process to determine the concentration of the pollutant remaining in the medium. Before the analysis, the samples were filtered through a 0.45 membrane syringe filter and analyzed for remaining supernatant fluid pollutants amount. It was observed that the absorbed and adsorbent reached to equilibrium after 40 min. However, the experiment was kept for 120 min to ensure that the absorption process reached to complete equilibrium. At the end of the experiment, the adsorbents were removed from suspension by centrifuging at 4000 rpm for 20 min and the residual

pollutants concentration in the solutions was measured using a UV–visible spectrophotometer (Hack 3900 model). All experiments were repeated twice, and average values were used for future calculations).

Equilibrium isotherms were obtained by performing batch adsorption studies. The % removal of naphthalene was calculated as follows:

$$\% \text{ Removal} = \frac{(C_0 - C_e)}{C_0} * 100 \quad (1)$$

where  $C_0$  is the initial concentration,  $C_e$  is the concentration at equilibrium (mg/L).

### 3. Results and discussions

#### 3.1. Characterization of polyaniline/bentonite nanocomposite (PAN/B)

##### 3.1.1. Characterization of bentonite used in composite preparation:

###### 3.1.1.1. EDAX for Bentonite

Bentonite used in the study was procured from a commercial dealer. The elemental composition of the bentonite was determined by EDAX and shown in figure 3.1.a. The results below show that the weight % of Si is 36.18% and oxygen is 45.33% and some metals which is the composition of bentonite.

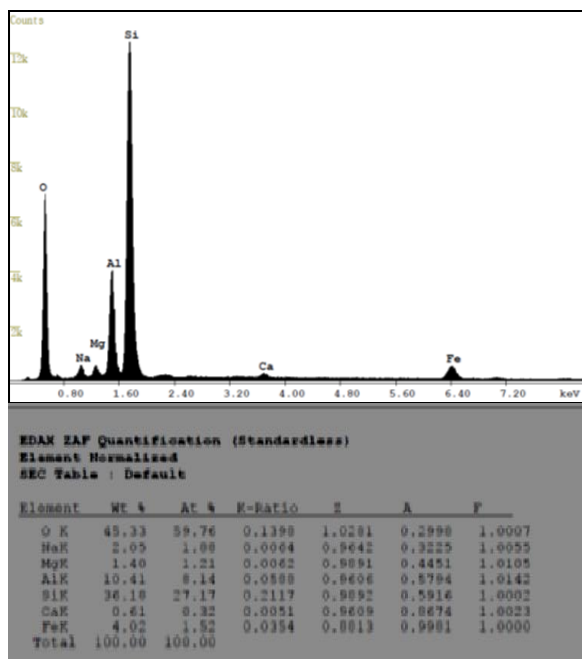


Figure (3.1a) EDAX for bentonite

###### 3.3.1.2. FTIR for bentonite

The FTIR analysis permits spectrophotometric observation of bentonite in the range 400–4,000 $\text{cm}^{-1}$  and serves as a direct means for the identification of the functional groups on the surface. Figure (3.1b) shows the Fourier transformed spectrum of bentonite at room temperature. In the IR studies of clay, the Si–O stretching vibrations were observed at 794.79  $\text{cm}^{-1}$ , 615.89  $\text{cm}^{-1}$  and 517.35  $\text{cm}^{-1}$  showing the presence of quartz ( $\text{SiO}_2$ ).

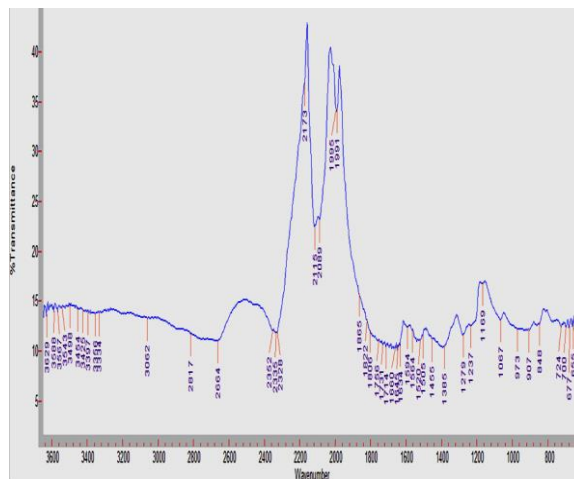


Figure (3.1b) FTIR for bentonite

#### 3.2. Characterization of polyaniline/bentonite prepared nanocomposite (PAN/B)

##### 3.2.1 Surface morphology

SEM technique was used to morphological analyses and characterization of the size and shape. SEM images with different levels of magnifications factor for the prepared poly aniline-bentonite composite (PAN/B) as shown in Figure (3.2a&b). These results show that the poly aniline-bentonite composite is spherical in shape with smooth surface like agglomerate which is composed of small spherical nanoparticles.

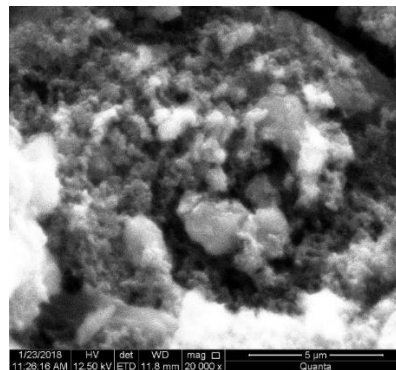


Figure (3.2a) SEM images for poly aniline-bentonite nanocomposite (PAN/B)

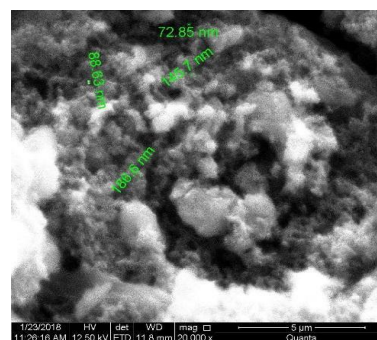


Figure (3.2b) SEM images for poly aniline-bentonite nanocomposite (PAN/B)

### 3.2.2. FTIR for the polyaniline-bentonite composite

The FTIR analysis permits spectrophotometric observation of polyaniline- bentonite nanocomposite (PAN/B) in the range 400–4,000  $\text{cm}^{-1}$  and serves as a direct means for the identification of the functional groups on the surface. Figure (3.3) shows the Fourier transformed spectrum of poly aniline-bentonite composite nano powder at room temperature. In the IR studies of clay, the Si–O stretching vibrations were observed at 794.79  $\text{cm}^{-1}$ , 615.89  $\text{cm}^{-1}$  and 517.35  $\text{cm}^{-1}$  showing the presence of quartz ( $\text{SiO}_2$ ). On polymerization, the sharp absorption peaks at 3407.66  $\text{cm}^{-1}$  and 3629.15  $\text{cm}^{-1}$  for aromatic asymmetric and symmetric NOH stretching of the primary O-NH<sub>2</sub> group, respectively. The intensity of the peak at 1635.44  $\text{cm}^{-1}$ , attributed to N-H in-plane bending of the primary NH<sub>2</sub> group, decreased to a considerable extent on polymerization as the intensity of the peak for N-H in-plane bending of the secondary aromatic amine was very weak. However, a broad band at 3407.64  $\text{cm}^{-1}$  in the spectrum suggest the possibility of water of hydration in the adsorbent. The inter layer hydrogen bonding in clay is assigned by a characteristics band at 3629.15  $\text{cm}^{-1}$ .

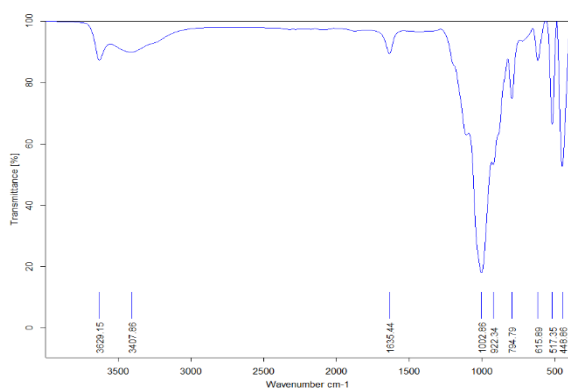


Figure (3.3) FTIR for the polyaniline-bentonite nanocomposite (PAN/B)

#### 3.1.2.3 Surface area BET

The results show that the polyaniline-bentonite nanocomposite (PAN/B) prepared has 21.7  $\text{m}^2/\text{g}$  and pore volume  $6.81 \times 10^{-2} \text{ cm}^3/\text{g}$  as shown in the table 1.

Table 1: BET for PAN/B

Sample weight	0.1613 [g]	Date of measurement	17/12/21
Saturated vapor pressure	102.97 [kPa]	Time of measurement	5:45:01
$V_m$	4.8926 [ $\text{cm}^3(\text{STP}) \text{g}^{-1}$ ]	$a_{s,BET}$	2.1730E+01 [ $\text{m}^2 \text{g}^{-1}$ ]
C	31.653	Total pore volume( $p/p_0=0.990$ )	6.8153E-02 [ $\text{cm}^3 \text{g}^{-1}$ ]
Mean pore diameter	12.545 [nm]		

### 3.3. Effect of Contact time on % removal of naphthalene

In order to establish the equilibration time for maximum adsorption and to determine the kinetics of the adsorption process, naphthalene adsorption on (PAN/B) was investigated as a function of contact time and the results are shown in Figure (3.4). As shown in (Fig. 3.4), naphthalene was rapidly removed by (PAN/B) (less than 20 min) that reached 80% removal at 30 min. for 30 & 50 ppm due to more vacant sites in adsorbent active surface until reach equilibrium state. These results are qualitatively in a good agreement with those found in the literature [16].

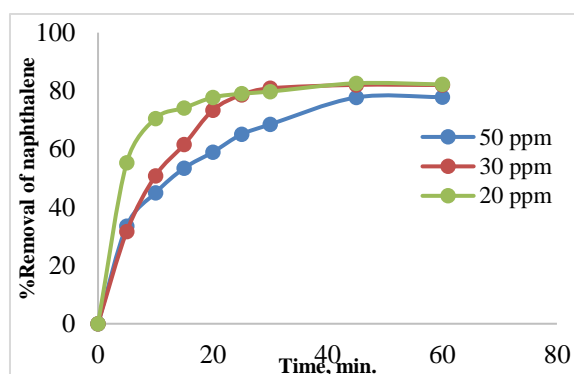


Figure (3.4) Effect of contact time on % removal of naphthalene at different concentration, pH=6, 25 °C, 50 rpm, PAN/B 4g/l

### 3.3. Effect of PAN/B dosage on %removal of naphthalene

The results of the experiments with varying adsorbent dosage are presented in Figure (3.5). It was found that with an increase in the adsorbent dosage from 2 to 5 g/L, the removal of naphthalene increases from 33.4 % to 81.4% for 45 min. This can attribute to the availability of vacant sites can connect with the small concentrations of pollutants which more dosage meaning more vacant sites so more removal of pollutants. These results are qualitatively in a good agreement with those found in the literature [16,22-23]. Also, increasing the surface area provide more adsorption rate which reflect to increase the pollutant removal as shown in table1.

### 3.4. Effect of pH on % removal of naphthalene

The acidity of solution (pH) is one of the most important parameters controlling adsorption of polycyclic aromatic hydrocarbons from aqueous solutions. Figure (3.6) describes the effects of pH on adsorption of naphthalene, the initial pH of solutions varied from 2.0 to 8 that justified by using NaOH solution (10%) and HCl.

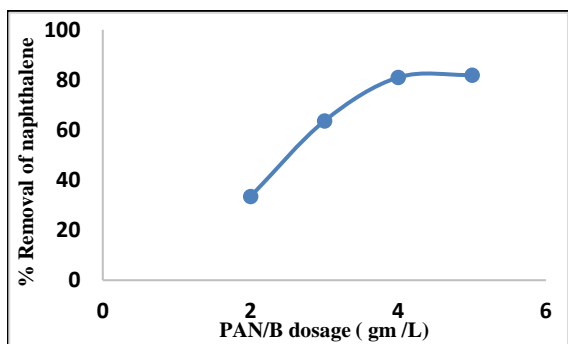


Figure (3.5) Effect of PAN/B dosage on % removal of naphthalene at 30 ppm, pH=6, 25 °C, 50 rpm for 45 min.

Studies beyond pH 8 were not attempted because precipitation of the ions as hydroxides would be likely. At lower values, the naphthalene adsorption was 43% at pH = 2 limited in this acidic medium, this can be attributed to the presence of H<sup>+</sup> ions which compete with the naphthalene for the adsorption sites.

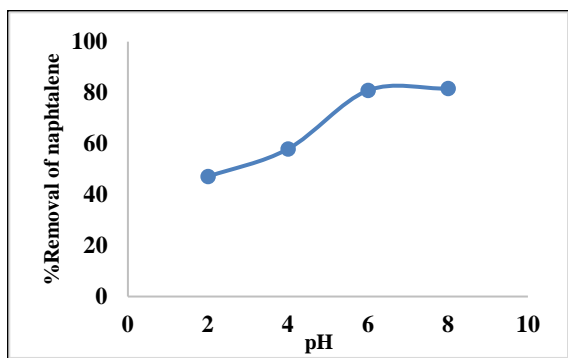


Figure (3.6) Effect of pH on % removal of naphthalene at 30 ppm, 25 °C, PAN/B 4g/l, 50 rpm for 45 min

### 3.5. 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. Modelling of adsorption isotherm

The analysis of the isotherm data is important to develop an equation which accurately represents the results and could be used for design purposes. In order to investigate the adsorption isotherm, the adsorption data can be interpreted using several relationships which describe the distribution of heavy metal molecules between the aqueous and solid phases. In order to investigate the adsorption isotherm, the experimental data of equilibrium isotherms were interpreted using two equilibrium models; the

Freundlich and the Langmuir. This modelling permits us to determine the maximal capacity of removal.

##### 3.5.1.1. Langmuir isotherm

Langmuir's isotherm model suggests that the uptake occurs on homogeneous surface by monolayer sorption without interaction between adsorbed ions. The linear form of Langmuir isotherm equation is represented by the following equation

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

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,  $C_e$ : equilibrium concentration, mg/l and  $q_e$ . Equilibrium adsorption capacity, mg/g. Therefore, a plot of  $C_e/q_e$  versus  $C_e$ , gives a straight line of slope  $1/Q^0$  and intercept  $1/(Q^0 b)$ .

The results obtained from the Langmuir model for the removal naphthalene shown in table 2 and figure 3.7.

##### 3.5.1.2. The Freundlich isotherm

Freundlich isotherm describes the adsorption equation for non-ideal adsorption that involves heterogeneous adsorption. This empirical isotherm is expressed by the following equation:

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

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 values of  $n$  and  $K_F$  can be calculated from the slope and intercept of the plot of  $\log q_e$  versus  $\log C_e$  derived from Eq. (4). The magnitude of the exponent  $1/n$  gives an indication of the favourability of adsorption as shown in table 2 and figure 3.8.

Table 2: Langmuir and Freundlich models for the removal Naphthalene on PAN/B.

Isotherm model	Naphthalene
<b>Langmuir: figure 3.7</b>	
$Q^0$ (mg g <sup>-1</sup> )	61.75
$K_a$ (L mg <sup>-1</sup> )	0.023
$R^2$	<b>0.9733</b>
<b>Freundlich: figure 3.8</b>	
$1/n$	0.6992
$R^2$	0.9514

From the previous table. Langmuir isotherm model provided good correlation for the adsorption of naphthalene by PAN/B.

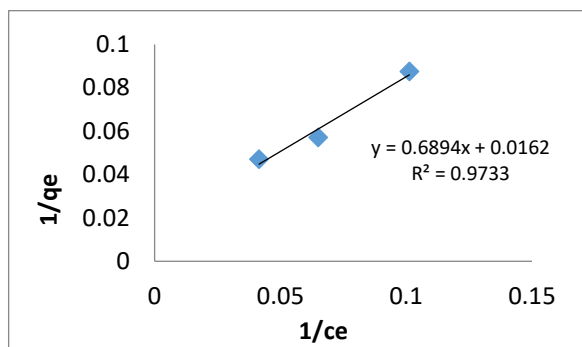


Figure (3.7): Langmuir isotherm

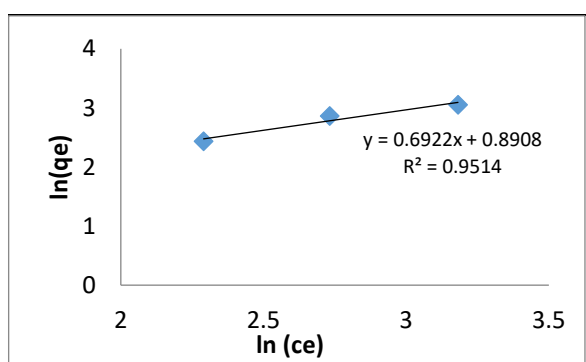


Figure (3.8): Freundlich isotherm

### 3.6. Kinetic modeling

Kinetics of adsorption is one of the most important characteristics to be responsible for the efficiency of adsorption. The adsorbate can be transferred from the solution phase to the surface of the adsorbent in several steps and one or any combination of which can be the rate-controlling mechanism: (i) mass transfer across the external boundary layer film of liquid surrounding the outside of the particle; (ii) diffusion of the adsorbate molecules to an adsorption site either by a pore diffusion process through the liquid filled pores or by a solid surface diffusion mechanism; and (iii) adsorption (physical or chemical) at a site on the surface (internal or external) and this step is often assumed to be extremely rapid.

The overall adsorption can occur through one or more steps. In order to investigate the mechanism of process and potential rate controlling steps, the kinetics of Ni and Pb adsorption onto activated carbon were analysed using pseudo-first-order [24] 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 adsorption kinetic data were described by the Lagergren pseudo-first-order model [20], which is the earliest known equation describing the adsorption rate based on the adsorption capacity. The linear form equation is generally expressing a follow:

$$\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} t \quad (5)$$

In order to obtain the rate constants, the values of  $\log(q_e - q_t)$  were linearly correlated with  $t$  by plot of  $\log(q_e - q_t)$  versus  $t$  to give a linear relationship from which  $K_1$  and predicted  $q_e$  can be determined from the slope and intercept of the plot, respectively. The variation in rate should be proportional to the first power of concentration for strict surface adsorption. However, the relationship between initial solute concentration and rate of adsorption will not be linear when pore diffusion limits the adsorption process.

#### 3.6.2. Pseudo-second-order equation

The adsorption kinetic may be described by the pseudo-second order model. The linear equation is generally given as follows:

$$\frac{t}{q_t} = \frac{1}{K_2} \frac{1}{q_e^2} + \frac{1}{q_e} t \quad (6)$$

If the second-order kinetics is applicable, then the plot of  $t/q_t$  versus  $t$  should show a linear relationship. Values of  $K_2$  and equilibrium adsorption capacity  $q_e$  were calculated from the intercept and slope of the plots of  $t/q_t$  versus  $t$ . The linear plots of  $t/q_t$  versus  $t$  show good agreement between experimental and calculated  $q_e$  values (Table 3). The correlation coefficients for the pseudo second-order kinetic model are greater than 0.99, which led to believe that the pseudo second order kinetic model provided good correlation for the adsorption of naphthalene by PAN/B.

Table 3: Kinetic parameters for the adsorption Naphthalene

Kinetics models	Naphthalene
Pseudo first order kinetics	
$K_1(\text{min})$	0.0614
$q_1(\text{mg/g})$	7.058
$R_1^2$	0.8935
Pseudo second order kinetics	
$K_2(\text{g/mg min})$	0.004
$q_2(\text{mg/g})$	24.68
$R_2^2$	<b>0.9968</b>

#### 4. Conclusions

In this research, the adsorption performance of naphthalene on the PAN/B nano composite was explored. The adsorption equilibrium of naphthalene on the PAN/B nano composite can be explained using Langmuir and Freundlich isotherms, with naphthalene fulfilling the Langmuir isotherm model. The adsorption of naphthalene on the PAN/B nano composite was effectively predicted using a simple kinetic model based on the pseudo second-order equation.

The naphthalene removal conditions were 80% at 30 minutes for 20 ppm at pH 6 and 4 g/l PAN/B nano composite. As a result, removing contaminants from industrial waste water allows it to be reused, so accomplishing the role of sustainability.

#### Conflicts of interest

“There are no conflicts to declare”.

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