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King Saud University

## Arabian Journal of Chemistry

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# Kinetics, equilibrium, and isotherm of the adsorption of cyanide by MDFSD



## Hisham J. El-Aila \*, Khaled M. Elsousy, Khaled A. Hartany

Department of Chemistry, Al-Aqsa University, P.N.A., Gaza Strip, Gaza, Occupied Palestinian Territory

Received 2 January 2011; accepted 3 March 2011 Available online 9 March 2011

#### **KEYWORDS**

Adsorption; MDFSD; Isotherms; Cyanide; Kinetic: Thermodynamic parameters Abstract Adsorption of cyanide out of aqueous media by moderate density fiberboard saw dust (MDFSD) was investigated using a batch system under controlled conditions. The kinetic data were followed more closely to the pseudo-first-order model. The adsorption rate and the related parameters, such as the MDFSD relative quantity, cyanide initial concentration, pH, temperature, and ionic strength were studied. The saturation equilibrium was also studied as a function of the related parameters (MDFSD quantity, initial concentration, pH and temperature). The adsorption isotherm was established and compared to other isotherms of activated carbon, TDRP and polyurethane foam. Linearized Langmuir, Freundlich and Frumkin models were applied to estimate the adsorption constants.

It was estimated through this study that the adsorption rate is positively affected by increasing MDFSD's relative quantity, adsorbent concentration, acidity and temperature. The adsorption equilibrium was shifted toward higher adsorption capacity by the substrate concentration, temperature and acidity. The adsorption data fit to Freundlich, Langmuir and Frumkin models, but attained a better correlation with the order of models as Langmuir > Frumkin > Freundlich. Activation thermodynamic parameters of adsorption, the adsorption thermodynamic parameters, and Freundlich, Langmuir and Frumkin constants were obtained.

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#### 1. Introduction

Corresponding author. Address: Department of Chemistry, Al-Aqsa University, P.O. Box 4051, P.N.A., Gaza, Occupied Palestinian Territory. Tel.: +972 08 2453524; fax: +972 08 2845488. E-mail address: heshamaila@yahoo.com (H.J. El-Aila).

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Cyanide is present in environmental matrices and waste streams as simple and complex cyanide, cyanates, and nitriles (Ebbs, 2004). Examples of the undesirable forms of cyanide are HCN and CN<sup>-</sup> (Tsoung, 1992). Cyanide compounds are extensively used in industry and their influents are the major source of these toxic pollutants in our environment (Desai and Ramakrishna, 1998). The release of cyanide from industries worldwide has been estimated to be more than 14 million kg/year (Ebbs, 2004). The major industries that used cyanide are electroplating and mixing {extraction of gold, silver, etc.}

#### http://dx.doi.org/10.1016/j.arabjc.2011.03.002

1878-5352 © 2011 Production and hosting by Elsevier B.V. on behalf of King Saud University. This is an open access article under the CC BY-NC-ND license (http://creativecommons.org/licenses/by-nc-nd/3.0/). (Desai and Ramakrishna, 1998; Akcil, 2003; Patil and Paknikar, 2002).

Adsorption and biodegradation are two significant methods for the treatment of waste water bearing cyanide compounds, either operated separately or simultaneously (Roshan et al., 2006). The selection of the treatment methods is based on the concentration of waste and the cost of treatment. The process of adsorption is a well established and powerful technique for treating domestic and industrial effluents (Naeem and Zafar, 2009). Activated carbon which has been the most used adsorbent, however, is relatively expensive, other specific examples of materials which are used as adsorbents include both the naturally occurring materials, such as the minerals, zeolites of different types, clays, and synthetic materials which include Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> (Tsoung, 1992; Naeem and Zafar, 2009). Iron-cyanide adsorption onto gamma Al<sub>2</sub>O<sub>3</sub> (Hang and Cheng, 1997) as well as adsorption of cyanide from aqueous solutions at pyrophyllite surface (Sona et al., 2001) has also been reported by different workers. Likewise batch and column tests were performed with various soils yielded significant removal of free cyanide at near neutral pH values (David, 2006).

Moderate density fiberboard saw dust (MDFSD) is a wood substitute which is made from fine wood fibers in a resin which is bonded under heat and pressure. It is manufactured by a dry process at a lower temperature than other fiberboard e.g., hardboard. The natural glues and resins contained within the wood are not effective. MDFSD, therefore, uses artificial bonding agents/resins. The resin used is usually urea formaldehyde, but some fiberboard including exterior or marine quality board will use stronger glues such as phenol formaldehyde. MDFSD may be used instead of plywood or chipboard. It is dense, flat, stiff, has no knots and is easily machined. It is made up of fine particles and, therefore, does not have an easily recognizable surface grain (Ayrilmis, 2007; Bowyer and Smith, 1998). The physical and mechanical properties of MDFSD are mainly dependent upon the properties of the raw materials (wood, binders, and other additives) and manufacturing parameters (Akbulut et al., 2004; Ayrilmis, 2008).

TDRP (tire derived rubber particles) is an economical, available material; reclaimed tire rubber has been used as an asphalt additive (Tortum and Çelik, 2005), as a molded products additive (Kim and Lee, 2002) and also as a component in landscaping mulch (Reid, 2005). Also, it is used as an adsorption material for gaseous phases as well as liquid media (Reid, 2005).

Polyurethane foam is one of the most versatile materials in the world today. They are known for being a perfect material for footwear, machinery industry, coatings and paints, rigid insulation, elastic fiber, soft flexible foam, medical devices (Randall and Lee, 2002; Szycher, 1999). Unique properties of polyurethanes foam deserve the popularity as polyurethanes offer the elasticity of rubber combined with the toughness and durability of metal, it is resistant to oils, solvents, fats. Products coated with polyurethane foam last longer, polyurethane adhesives provide strong bonding advantages, polyurethane elastomers can be molded into any shape, they are lighter than metal and offer good resistance to environmental factors (Randall and Lee, 2002; Szycher, 1999).

The objective of this work is to investigate the applicability of MDFSD, an inexpensive environment friendly material, as an adsorbent for the removal of cyanide from aqueous solution. The pertinent parameters that influence adsorption, such as adsorbent quantity, initial cyanide ( $CN^-$ ) concentration, pH, ionic strength and temperature were investigated. Adsorption isotherms at three different initial concentrations of cyanide on different adsorbent (MDFSD, charcoal, TDRP, polyurethanes foam) were studied. The adsorption data were interpreted using Freundlich, Langmuir and Frumkin isotherms. Various thermodynamic parameters including the mean energy of adsorption were calculated.

#### 2. Experimental

#### 2.1. Materials and methods

All reagents were used of analytical grade and purchased from E. MERCK. MDFSD was used as the waste saw dust and was obtained locally.

MDFSD was taken, washed with distilled water, then immersed in boiling water for 15 min to extract any possible water soluble components, filtered to discard the water soluble substances. The filtrate was dried at 95 °C for 48 h, sieved between 0.01 and 0.1 mm diameter. The produced powder was kept dry and was used with no further treatment. TDRP, trademarked by Envirotech Systems, Inc. of Lawton, IA (USA), is available and was used in this work. Polyurethane foam was taken out of refrigerator waste, powdered, sieved between 0.01 and 0.1 mm diameter and used without further treatment. Activated carbon from Nuchar Peltized West Vacco of Covington, VA (USA) was used.

Stock solution of cyanide (2000 mgL<sup>-1</sup>) was prepared by dissolving sodium cyanide in distilled water. The concentration range of cyanide prepared from stock solution varied between 10 and 200 mg L<sup>-1</sup>.

#### 2.2. Instrumentation

The pH values of the solutions were adjusted using a microprocessor combined pH-meter and thermometer, model pH 211 by Hanna instruments.

A thermostated automatic shaker associated with circulation model YCW, USA was used for the batch experiments. The centrifugation was done with Wirowka Type WE-1 centrifuge machine at 4500 rpm. Absorbance of (cyano-picrate) complex in the sample was measured directly using a Shimadzu-1601 UV–VIS spectrophotometer at 530 nm using alkaline picrate mixture as the coloring agent (indirect method).

#### 2.3. Adsorption studies

The adsorption kinetics of  $CN^-$  on MDFSD was studied using batch technique (Sumra et al., 2009; Sumra and Uzma, 2009). A known weight (i.e., 1.0 g of MDFSD) was equilibrated with 100 mL of varying concentration of aqueous cyanide solution (10–200 ppm) in a well-sealed 200 mL bottle, at a fixed temperature in a thermo-stated shaker water bath for a known period of time. A relatively large amount of MDFSD was used (1.0 g) to establish a pseudo-first order adsorption condition to simplify the calculations. The uptake was calculated from the initial and first one hour concentrations. The initial adsorption rate was taken as an observed rate constant (s<sup>-1</sup>). After equilibrium the suspension was centrifuged in a stoppered tube for 5 min at 4500 rpm, then filtered through Whatman 41 filter paper. All absorbance readings were taken at pH 10.0. For kinetic rate study, 2 mL samples of the previous solution were taken in different time intervals (5 min to 1 h) and were isolated for analysis. The effect of pH was investigated in the range of (1.6–12.0) and it was adjusted by using NaOH and H<sub>2</sub>SO<sub>4</sub>.

Ionic strength effect was studied by using different concentrations of  $K_2SO_4$  (0.01–0.1 M).

#### 2.4. Sample preparation (Cardoso et al., 2005)

2 mL of picric acid (2.56 g in 100 mL of distilled water) and 4 mL of sodium carbonate (5% w/v) were mixed in a glass tube to have alkaline picrate just on time for analysis. 2 mL of the unknown cyanide solution in addition to 2 mL of 0.01 M  $H_2SO_4$  were taken in a second glass tube. Both tubes were mixed and incubated for 15 min in water bath at 37 °C.

Adsorption capacity of cyanide has been evaluated from the Freundlich, Langmuir and Frumkin adsorption isotherms that were studied at 22 °C. The cyanide concentration studied was in the range of 10–200 ppm. All experiments were carried out in triplicates with respect to each condition and mean values were used for further calculations, standard deviation was with accuracy of  $\pm 2\%$ . Identical blanks (composed of all the constituents of a sample except the absorbent) were used for every condition to account for any unexpected inner deviation like bottle surface adsorption or loss by evaporation. The final concentration of the blank was taken as  $C_{\circ}$ .

#### 3. Results and discussion

Adsorption kinetics, saturation equilibrium, and isotherm of adsorption of cyanide by MDFSD out of aqueous solution were tested by changing the following parameters: contact time, the amount of each adsorbent and adsorbate, ionic strength, pH and temperature.

#### 3.1. Adsorption kinetics

The elimination rate of cyanide by adsorption on MDFSD was followed with respect to contact time under the experimental conditions. As shown in Fig. 1, the adsorption follows a pseudo-first order model; where the natural logarithm of  $(C_{\circ}/C)$  has a straight line relation with time. It can be estimated also that a 24 h period is adequate time to achieve practical equilibrium.

Dose of MDFSD influences on the adsorption rate was shown in Fig. 2. It shows that the rate increases with increase in the amount of MDFSD up to 1.0 g and reach to a constant plateau (1.0-2.0 g) in the studied range. This indicates that the proper weight of adsorbent is 1.0 g for the present conditions.

The study of the initial adsorbate concentration influence shows that the rate of adsorption (mg dm<sup>-3</sup> s<sup>-1</sup>) is directly proportional to the adsorbate concentration in the studied range (10–200 ppm) as shown in Fig. 2.

The effect of variation of pH on adsorption rate was found to show a trend of decreasing rate versus increasing pH as shown in Fig. 2. According to Le Chatliere principle, this influence is thought to be a direct result of increasing the HCN



**Figure 1** The relation between  $\ln C_{\circ}/C$  vs. time to verify pseudo first order kinetics. [MDFSD] = 1.0 g, [cyanide] = 100 ppm, T = 22 °C.



**Figure 2** Variation of the initial rate of adsorption vs. either mass of MDFSD [0.1–1.6 g]; cyanide concentration [25–160 ppm; pH [1–12] at 22 °C.

species by acidity according to the Eqs. (1) and (2) where the reaction is shifted to the right by increasing  $H^+$  concentration. Then, molecular HCN is thought to be adsorbed in the unionized form and vice versa in the case of increasing pH. So, as pH is increased,  $OH^-$  is increased and lowers the concentration of HCN present, which limits the adsorption potential.

$$NaCN \to Na^+ + CN^- \tag{1}$$

$$H^+ + CN^- \leftrightarrow HCN$$
 (2)

The influence of temperature enhances the adsorption rate (285–333 K). The relation was found to fit an Arrhenius equation (Eq. (3)) by showing a straight line when  $(\ln k_{obs})$  is plotted against (1/T) at (MDFSD = 1.0 g, [cyanide] = 100 ppm, T = 285-333 K). The activation energy  $E_a^*$  of the process was calculated from Arrhenius plot which is shown in Fig. 3.

$$\ln k_{\rm obs} = \ln A - \frac{E_a^*}{RT} \tag{3}$$



**Figure 3** Relationship between  $\ln k_{obs}$  vs. 1/T to verify Arrhenius equation. [MDFSD] = 1.0 g, [cyanide] = 100 ppm, T = 22 °C.

Where A is the Arrhenius factor, R is the universal gas constant =  $8.303 \text{ J} \text{ mol}^{-1} \text{ K}^{-1}$  and T is the absolute temperature, K.

According to Aliola and Oforka (2002) and Damaskin (1971) a value of activation energy less than 80 kJ/mol represents physisorption. Hence, in the present observation, a considerably lower value of activation energy (29.5 kJ mol<sup>-1</sup>) indicates that the transfer of adsorbate from the aqueous phase to the solid surface passes through a physical adsorption with low activation energy. Also, the negative value of  $\Delta S^*$  (-212.3 J mol<sup>-1</sup> K<sup>-1</sup>) and the positive value of  $\Delta G^*$  (95.1 kJ mol<sup>-1</sup>) indicate the spontaneous formation of transition state.

From the study it was found that ionic strength of different concentrations of  $K_2SO_4$  (0.01–0.1 M) has no influence on the adsorption rate constant, which ensures that the adsorption process does not include an ionic reaction (House, 2007).

#### 3.2. Adsorption equilibrium

Adsorption equilibrium was studied under conditions of relatively low amount of MDFSD (1.0 g), higher cyanide concentration (100.0 ppm) and suitable time (24 h) to reach a saturation equilibrium according to kinetic testing parameters.

The adsorption equilibrium constant or adsorption distribution coefficient ( $K^{\circ}$ ) (Tahir and Rauf, 2003) was obtained from Eq. (4):

$$K^{\circ} = \frac{C_{\text{solid}}}{C_{\text{liquid}}} \tag{4}$$

where,  $C_{\text{solid}}$  is the solid phase concentration at equilibrium (mg/g);  $C_{\text{liquid}}$  is the liquid phase concentration at equilibrium (mg/L).

The equilibrium constant  $(K^{\circ})$  increases with increasing mass of MDFSD up to 1 g and reach nearly a constant plateau in the studied range (0.1-2.0 g) as shown in Fig. 4.

But the influence of the initial cyanide bulk concentration on the adsorption rate shows that the equilibrium is shifted toward higher adsorption capacity by increasing initial concentrations as shown by the slightly lower equilibrium constant  $(K^{\circ})$  in Fig. 4.



**Figure 4** Variation of equilibrium constant ( $K^{\circ}$ ) vs. variation either mass of MDFSD [0.1–1.6 g]; initial concentration of cyanide [10–160 ppm]; pH [1–12] and temperature [12–60 °C].

The effect of pH on  $K^{\circ}$  was shown in Fig. 4, where a trend of decreasing of  $K^{\circ}$  versus increasing pH was indicated. So, the pH of aqueous media has a strong influence on the adsorption equilibrium constant. The hydrogen ion concentration [H<sup>+</sup>] has no direct influence on the adsorption process specifically, but it is referred to the increasing of the adsorbate species concentration (HCN) which is increased by acidity, according to Le Chatliere principle when applied on Eq. (2).

Ionic strength has no observed influence on the adsorption equilibrium as all other parameters are kept constant. The absence of any effect of ionic strength shows that the adsorption process does not include an ionic reaction. This may support the expectation that the actual adsorbed species is HCN based on a physisorption process and not  $CN^-$  chemisorption; where the second probability is mainly affected by ionic strength (House, 2007).

The influence of temperature on the adsorption equilibrium revealed that the equilibrium is shifted sharply toward higher adsorption capacity by increasing the temperature. The relation between equilibrium constant  $K^{\circ}$  and temperature is plotted in Fig. 4. This relation within the studied range of temperature (285–333 K) shows that the adsorption supports an endothermic process. Thermodynamic equilibrium parameters such as change in free energy ( $\Delta G^{\circ}$ , kJ/mol), enthalpy ( $\Delta H^{\circ}$ , kJ/mol) and entropy ( $\Delta S^{\circ}$ , J/K mol) were determined using the following Eqs. (5) and (6):

$$\Delta G^{\circ} = -RT\ln K^{\circ} \tag{5}$$

$$\ln K^{\circ} = \frac{\Delta S^{\circ}}{R} - \frac{\Delta H^{\circ}}{RT} \tag{6}$$

The  $\Delta H^{\circ}$  and  $(\Delta S^{\circ}$  values were obtained from the slope and intercept of Van't Hoff plots of ln  $K^{\circ}$  vs. 1/T (Tahir and Rauf, 2003) as shown in Fig. 5.

The positive and lower value of  $\Delta H^{\circ}$  (7.7 kJ mol<sup>-1</sup>) shows the endothermic nature of adsorption and the possibility of physical adsorption. (Gong and Sun, 2005; Vadivelan and Vasanthkumar, 2005) The negative values of  $\Delta H^{\circ}$  (-2.1 kJ mol<sup>-1</sup>) shows that the adsorption is favorable and spontaneous. The positive values of  $\Delta S^{\circ}$  (32.7 Jmol<sup>-1</sup> K<sup>-1</sup>) show the increased disorder at the solid solution interface components. The higher 1.3

12

1.1

0.9

0.7

ריים <sup>0.0</sup>





**Figure 5** Van't Hoff plot of  $\ln K^{\circ}$  vs. 1/T mass of MDFSD = 1.0 g; [cyanide] = 100 ppm.

adsorption capacity of MDFSD at higher temperatures was attributed to the enlargement of the pore size and activation of the adsorbent surface.

#### 3.3. Adsorption isotherm

The adsorption isotherms were plotted after performing all experimental conditions to be constant, using relatively wide range of adsorbate concentrations [10–200 ppm cyanide]. The adsorption isotherms of different adsorbents, MDFSD, activated carbon, polyurethane foam and TDRP were constructed for comparison purpose under the same experimental conditions. Fig. 6 shows the isotherms of each of them; it is obvious that the overall (neglecting the differences in porosity, surface specific area and particle average size) adsorption capacity of adsorbents is arranged as following:

Activated carbon > MDFSD > poly urethane foam > TDRP

The adsorption capacity of activated carbon is clearly higher as the adsorbent is expressed in grams.

Several adsorption models have been published in the literature to verify the experimental data in which the most common models are the Langmuir, Freundlich and Frumkin's isotherms (Arivoli and Kalpana, 2007; Casey, 1997; Damaskin, 1971). In order to facilitate the estimation of the adsorption capacities at various concentrations, Langmuir, Freundlich and Frumkin's adsorption isotherms, typical models for monolayer adsorption, were applied. The linearized Langmuir model can be written as (Arivoli and Kalpana, 2007; Casey, 1997; Damaskin, 1971):

$$\frac{1}{Q_{\rm e}} = \frac{1}{K_{\rm L}} + \frac{1}{bK_{\rm L}} \cdot \frac{1}{C_{\rm e}} \tag{7}$$

Where  $C_e$  is the concentration of adsorbate equilibrium (mg/L),  $Q_e$  is the amount of HCN adsorbed at equilibrium (mg/g),  $K_L$  (g/mg) and b (dm<sup>3</sup>/mg) are the Langmuir constants, representing the maximum adsorption capacity for the solid phase loading and the energy constant related to the heat of





**Figure 6** Adsorption isotherms of different adsorbents (MDFSD, activated carbon, TDRP and polyurethane foam). Mass of adsorbent = 1 g; [cyanide] 100 ppm, T = 22 °C.

adsorption. The constants b and  $K_{\rm L}$  can be evaluated from the intercept and slope of the linear plot of the experimental data of  $1/Q_{\rm e}$  versus  $1/C_{\rm e}$ , respectively. The linearized Langmuir isotherm was developed with slope, intercept and correlation factor ( $R^2$ ) equal to 4.8337, 0.0104 and 0.991, respectively. The Langmuir constants were calculated from the slope and intercept ( $K_{\rm L} = 96.15$  g/mg, b = 0.002 dm<sup>3</sup>/mg).

The Freundlich isotherm has been widely used to characterize the adsorption capacity of organic pollutants using different adsorbents by fitting the adsorption data. The Freundlich isotherm in its linearized form can be written as (Naeem and Zafar, 2009; Casey, 1997; Damaskin, 1971; Arab and Al-Turkustani, 2006):

$$\ln Q_{\rm e} = \ln K_{\rm f} + \frac{1}{n} \ln C_{\rm e} \tag{8}$$

where,  $K_{\rm f}$  is the Freundlich constant related to the adsorption capacity (mg/g), and 1/n is the intensity of adsorption. The values of  $K_{\rm f}$  and 1/n can be determined from the intercept and slope, respectively, of linear plot of ln  $Q_{\rm e}$  versus ln  $C_{\rm e}$ . The linearized Freundlich adsorption isotherm was developed with slope, intercept and correlation factor equal 0.7719, intercept -0.771 and 0.911, respectively. The Freundlich constants were calculated from the intercept and slope ( $K_{\rm f} = 0.46$  mg/g, n = 1.3).

The adsorption on a homogeneous surface with an interaction in the adsorption layer obeys Frumkin's isotherm (Naeem and Zafar, 2009; Arab and Al-Turkustani, 2006). The equation of Frumkin isotherm is (Naeem and Zafar, 2009; Arab and Al-Turkustani, 2006),

$$\left(\frac{Q_{\rm e}}{1-Q_{\rm e}}\right)e^{2aQ_{\rm e}} = KC_{\rm e} \tag{9}$$

Taking log on both sides, it becomes,

$$\log\left(\frac{Q_{\rm e}}{1-Q_{\rm e}}\right) \cdot \frac{1}{C_{\rm e}} = \log K + 2aQ_{\rm e} \tag{10}$$

where *a* is the interaction parameter which can be positive or negative. A positive value indicates that the adsorption energy is increased by the lateral attraction between the adsorbed molecules and the negative values suggest that there is a presence of the lateral force of repulsion between the molecules in the adsorbed layer. The plot of Log  $\{Q_e/(1 - Q_e)C_e\}$  vs.  $Q_e$ , shows a linear relationship with slope, intercept and correlation factor equal to 0.013, 1.4726 and 0.926, respectively. The values of *K* and *a* are calculated from the intercept and the slope, respectively. The positive value of *a* (0.0062 mg/g) indicates that the adsorbed molecules. The value of *K* is equal to 0.036 dm<sup>3</sup>/mg.

The values of free energy of adsorption ( $\Delta G_{ads}$ ) are calculated by using the following equation (Naeem and Zafar, 2009; Arab and Al-Turkustani, 2006):

$$\Delta G_{\rm ads} = -RT \ln 55.5 \, K \tag{11}$$

The negative value of  $\Delta G_{ads}$  (-12.9 kJ mol<sup>-1</sup>) indicates the spontaneity of the adsorption process and the stability of the adsorbed layer on MDFSD surface. The low  $\Delta G_{ads}$  value in the present case further confirms physical adsorption.

The high regression values show that the equilibrium data for HCN fitted generally either Langmuir, Freundlich or Frumkin isotherms in the studied concentration ranges, although the volatility of adsorbate caused some roughness of the observed data. Based on the correlation factors, the equilibrium data better fitted the models in the order of:

Langmuir > Frumkin > Freundlich

#### 4. Conclusion

It was concluded in this study that MDFSD can be successfully used as a purification adsorbent. The adsorption process can be applied in a continuous reactor system to purify industrial waste water of electroplating factories and in extraction of gold, silver, etc. The application can be economic since the MDFSD is a waste material which is priceless.

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