Membrane Water Treatment, *Vol. 5, No. 4 (2014) 295-304*
DOI: http://dx.doi.org/10.12989/mwt.2014.5.4.295

Adsorptive separation of adipic acid from aqueous solutions by perlite or its composites by manganese or copper

Hasan Uslu *[1], Göksel Demir [2], Cuma Bayat [3],
Kailas L. Wasewar [4] and Hisham S. Bamufleh [5]

[1] *Beykent University, Engineering and Architecture Faculty, Chemical Engineering Department, Ayazağa, İstanbul, Turkey*
[2] *Bahçeşehir University, Engineering Faculty, Environmental Engineering Department, Beşiktaş, İstanbul, Turkey*
[3] *İstanbul Esenyurt University, Engineering and Architecture Faculty, Industrial Engineering Department, Esenyurt, Istanbul, Turkey*
[4] *Advance Separation and Analytical Laboratory (ASPAL), Department of Chemical Engineering, Visvesvaraya National Institute of Technology, Nagpur-440010 (M.S) India*
[5] *Department of Chemical & Materials Engineering, Faculty of Engineering, King Abdulaziz University, P.O. Box 80204, Jeddah 21589, Saudi Arabia*

(*Received May 16, 2014, Revised September 23, 2014, Accepted October 30, 2014*)

Abstract. Adipic acid (hexane-1,6-dioic acid) is one of the most used chemical in industrial applications. This must be separated from any environmental contaminant. In this study, adipic acid separation from wastewater by adsorption method onto Perlite or Perlite + Mn or Perlite + Cu composites was investigated. Adsorption of Adipic acid was investigated in terms of equilibrium, and thermodynamic conditions. For thermodynamic investigations the experiments carried out at three different temperatures (298 K, 318 K, 328 K). In the equilibrium studies, 2 g of perlite and its composites were determined as the optimal adsorbent amount. Freundlich and Langmuir isotherms were applied to the experimental data. Freundlich isotherms for all temperatures used in this work gave some deviations with R square values under 0.98 where as Langmuir isotherm gave good results with R square values upper 0.99 at different temperatures. As a result of thermodynamic studies, adsorption enthalpy (ΔH), adsorption entropy (ΔS), and adsorption free energy (ΔG) have been calculated for each adsorbents.

Keywords: adsorption; adipic acid; perlite; manganese; copper

1. Introduction

Adipic acid is one of the most important carboxylic acids. It is used as a precursor of the “nylon” production. The annual adipic acid production is nearly 2.5 million tons. Adipic acid is used as flavorant in foods. And it is used in medicine, too. The recovery of the adipic acid is important. Adsorption method can be used for adipic acid recovery.

There are many separation processes for removing organic pollutant from wastewater stream for example extraction (Uslu *et al.* (2008), Tuyun and Uslu (2011), adsorption (Uslu *et al.* (2010),

*Corresponding author, Professor, E-mail: hasanuslu@gmail.com

http://www.techno-press.org/?journal=mwt&subpage=7 ISSN: 2005-8624 (Print), 2092-7037 (Online)

Uslu and Demir (2010), membrane membrane (Terry *et al*. 1982, Khoiruddin *et al*. 2014, Melita *et al*. 2014), distillation (Norwitz *et al*. (1987) etc. Adsorption enables separation of selected compounds from dilute solutions. When comparing with alternative technologies, adsorption is attractive for its relative simplicity of design, operation and scale up, high capacity and favorable rate, insensitivity to toxic substances, ease of regeneration and low cost. Additionally, it avoids using toxic solvents and minimizes degradation.

In this study, perlite and its composites are used for adipic acid adsorption from wastewater. Perlite is an abundant, versatile and cheap material. Perlite consists of SiO_2, Al_2O_3, Na_2O, K_2O, Fe_2O_3, MgO and CaO. Several studies have been proposed in literatures for use of perlite as adsorbent. Akkaya (2013) investigated adsorption of radio nuclides on perlite modified with polyhydroxy ethyl methacrylate and ph effect, concentration effect and temperature effect was studied. Sarı *et al*. (2012) studied antimony (III) adsorption by using raw and Mn modified perlite. Silber *et al*. (2011) used the perlite for adsorption of zinc. The conditions pH, ionic strength and temperature were investigated. Zvezdelina *et al*. (2012) reported of mechanism of nitrophenols adsorption on perlite. Equilibrium and kinetic models were presented. Thanh *et al*. (2011) explained preparation of perlite composites. Fe_2O_3 and MnO_2 nanomaterials were used in composites. Torab-Mostaedi *et al*. (2011) examined adsorption of strontium and barium from aqueous media by using expanded perlite. Ghassabzadeh *et al*. (2010) presented characterizations of adsorption process of Co (II) and Pb (II) from aqueous solution.

This study focused on adsorption of adipic acid by perlite and its composites with copper(II) and manganese(II) and their efficiencies. The thermodynamic parameters, ΔH^0_{ads}, ΔS^0_{ads} and ΔG^0_{ads}, were calculated.

2. Material and methods

2.1 Material

Adipic acid is a white crystalline solid. Its melting point is 425.2 K, and its boiling point is 612.5 K. The water solubility at 288 K is 15 $g.L^{-1}$. The flash point is 469 K. The adipic acid was purchased from Sigma-Aldrich with purity > 99 % in mass. MnO_2 and CuO were purchased from Sigma-Aldrich with purity > 99 % in mass.

Perlite has been purchased by a commercial company in Turkey. Chemical composition of typical perlite contains approximately 72-75 % SiO_2, 11-14 % Al_2O_3, 0.-0.8 % CaO, 0.1-0.25 % MgO, 0.5-0.9 % Fe_2O_3, 4.8-5.7 % K_2O, 2.8-4.3 % Na_2O 3, 3.2-4.3 % H_2O in mass. The Specific surface area and total pore diameter are 3.6 $m^2.g^{-1}$ and 58.3 A, respectively.

2.2 Preparation of composites

In this study, according to Thanh *et al*. (2011)'s explanations the surface properties of perlite have been modified by using manganese and copper metals. The adipic acid adsorption abilities were compared with pure perlite. For the synthesis of perlite/MnO_2 or CuO composites, 0.5 g of MnO_2 or CuO particles and 1 g of ball-milled expanded perlite powder have been putted in 30 mL of a water: ethanol (1:1, v/v) solution and stirred for 6 h for making uniform. After that the sample has been kept overnight in a glass bottle at room temperature to let the sample to sediment. The highest layer has been discarded and the lowest layer (sediment) has been vacuum dried at 353 K for 12 h. The color change of the powder from white (Perlite) to brown (perlite/ CuO) and black

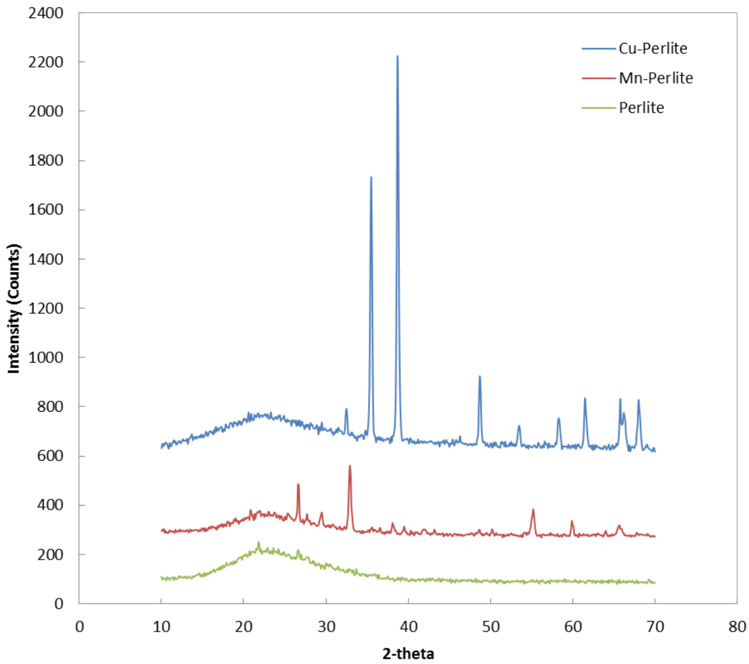

Fig. 1 X-Ray Diffraction pattern results for composites of perlite with Cu (II) and Mn (II)

(perlite /MnO_2), indicates the presence of the respective metal oxides particles. Fig. 1 shows the characterization of perlite composites with copper (II) and manganese (II). As seen from Fig. 1 the manganese (II) and copper (II) peaks are clearly observe the broad peak band between 25° and 35° for manganese (II) and between 35° and 40° for copper (II). Detailed information about structure of these composites was presented in literature (Thanh *et al*. (2011)).

2.3 Equilibrium methods

Three different concentrations of adipic acid were prepared for the equilibrium studies. These concentrations were 5 $g.L^{-1}$, 10 $g.L^{-1}$, 15 $g.L^{-1}$. The concentration 15 $g.L^{-1}$ is the maximum solubility of adipic acid in water at 288 K.

The 20 ml of adipic acid solution and different amount of adsorbent was putted to series of Erlenmeyer flasks 100 ml capacity and equilibration studies were carried out in a temperature controlled shaker. Equilibrium time was determined by preliminary tests as 120 min. The samples were shaken for 120 min., and the optimum amount of adsorbent was determined as 2 g for adsorbents used in this study. After this, an aqueous phase sample was titrated to determine the amount of adipic acid by NaOH (0.1 N) with indicator phenolphthalein (Uslu and Demir 2010). In most cases the deviation between the amount of acid analyzed and the amount of acid known by preparing the solutions by weighing did not exceed 3%.

The amounts adsorbed by adsorbent particles at equilibrium, q_e (mg/g), was calculated by

$$q_e = \frac{V\left(C_0 - C_e\right)}{W} \tag{1}$$

where C_0 and C_e are the liquid concentrations at the start and at equilibrium, respectively, V is the volume of aqueous solution and W is the mass of adsorbent.

Adipic acid removal (%) from aqueous phase onto adsorbent was calculated using the following equation

$$\text{Phenol removal}(\%) = \frac{C_0 - C_e}{C_0} \times 100 \tag{2}$$

3. Results and discussion

3.1 Equilibrium studies

5 g.L^{-1}, 10 g.L^{-1}, and 15 g.L^{-1} of initial adipic acid concentrations were prepared to investigate effect of initial adipic acid concentration on the adsorption by perlite and it composite. The results were presented in Tables 1 to 3 for all adsorbents. It can be seen Table 1, increase of initial acid concentration from 5.00 g.L^{-1} to 15.00 g.L^{-1} the adsorbed adipic acid concentration decreased at the all temperatures. The removal efficiency, which is defined as the amount of adipic acid adsorbed from aqueous solution on to the adsorbents, decreased from 97.80 % to 16.46 % with increasing initial concentration of adipic acid at 298 K. Similar results have been observed in other temperatures. This situation can be explained the saturation of accessible exchangeable sites on the adsorbents.

Table 1 Experimental results of the adsorption of adipic acid onto Perlite at different temperatures (298 K, 318 K, 328 K)

Initial Conc. of AA C_0 (g. L^{-1})	Amount of Perlite (g)	Equilibrium Aq. Conc. 298 K C (g.L^{-1})	Equilibrium Aq. Conc. 318 K C (g.L^{-1})	Equilibrium Aq. Conc. 328 K C (g.L^{-1})	Removal of AA 298 K (%)	Removal of AA 318 K (%)	Removal of AA 328 K (%)
	0.5	4.03	4.22	4.31	19.4	15.6	13.8
	1	3.41	3.57	3.67	31.8	28.6	26.6
5	1.5	2.75	2.91	3.02	45	41.8	39.6
	2	2.13	2.31	2.41	57.4	53.8	51.8
	2.5	1.66	1.87	1.95	66.8	62.6	61
	0.5	8.14	8.53	8.73	18.6	14.7	12.7
	1	7.38	7.72	7.96	26.2	22.8	20.4
	1.5	6.16	6.45	6.73	38.4	35.5	32.7
10	2	5.42	5.67	5.83	45.8	43.3	41.7
	2.5	4.54	4.73	4.96	54.6	52.7	50.4
	0.5	12.53	12.86	13.21	16.46	14.26	11.93
	1	11.31	11.72	12.09	24.6	21.86	19.4
15	1.5	10.46	10.89	11.11	30.26	27.4	25.93
	2	9.05	9.56	9.79	39.66	36.26	34.73
	2.5	8.46	8.95	9.25	43.6	40.33	38.33

Table 2 Experimental results of the adsorption of adipic acid onto Perlite + Cu + 2 at different temperatures (298 K, 318 K, 328 K)

Initial Conc. of AA C_0 (g. L^{-1})	Amount of Perlite + Cu^{+2}) (g)	Equilibrium Conc. 298 K C (g.L^{-1})	Equilibrium Conc. 318 K C (g.L^{-1})	Equilibrium Conc. 328 K C (g.L^{-1})	Removal of AA 298 K (%)	Removal of AA 318 K (%)	Removal of AA 328 K (%)
5	0.5	3.26	3.41	3.54	34.8	31.8	29.2
	1	2.54	2.76	2.95	49.2	44.8	41
	1.5	1.96	2.28	2.49	60.8	54.4	50.2
	2	1.49	1.74	1.95	70.2	65.2	61
	2.5	1.14	1.47	1.74	77.2	70.6	65.2
	0.5	6.96	7.2	7.46	30.4	28	25.4
10	1	6.16	6.45	6.73	38.4	35.5	32.7
	1.5	5.42	5.67	5.83	45.8	43.3	41.7
	2	4.54	4.73	4.96	54.6	52.7	50.4
	2.5	3.6	3.86	4.08	64	61.4	59.2
	0.5	11.26	11.47	11.61	24.93	23.53	22.6
15	1	10.05	10.27	10.44	33	31.53	30.4
	1.5	8.84	8.98	9.21	41.06	40.13	38.6
	2	7.72	7.95	8.16	48.53	47	45.6
	2.5	6.59	6.79	6.95	56.06	54.73	53.66

Table 3 Experimental results of the adsorption of Adipic acid onto Perlite + Mn^{+2} at different temperatures (298 K, 318 K, 328 K)

Initial Conc. of AA C_0 (g. L^{-1})	Amount of Perlite + Mn^{+2}) (g)	Equilibrium Conc. 298 K C (g.L^{-1})	Equilibrium Conc. 318 K C (g.L^{-1})	Equilibrium Conc. 328 K C (g.L^{-1})	Removal of AA 298 K (%)	Removal of AA 318 K (%)	Removal of AA 328 K (%)
5	0.5	2.67	2.79	2.9	46.6	44.2	42
	1	1.72	1.84	1.95	65.6	63.2	61
	1.5	1.15	1.24	1.35	77	75.2	73
	2	0.75	0.85	0.95	85	83	81
	2.5	0.11	0.18	0.26	97.8	96.4	94.8
	0.5	6.12	6.24	6.35	38.8	37.6	36.5
10	1	5.05	5.17	5.29	49.5	48.3	47.1
	1.5	3.97	4.09	4.21	60.3	59.1	57.9
	2	2.86	3	3.12	71.4	70	68.8
	2.5	1.84	1.95	2.07	81.6	80.5	79.3
	0.5	10.15	10.27	10.41	32.33	31.53	30.6
15	1	9.06	9.19	9.33	39.6	38.73	37.8
	1.5	7.94	8.07	8.22	47.06	46.2	45.2
	2	6.88	7	7.21	54.13	53.33	51.93
	2.5	5.75	5.89	6.05	61.66	60.73	59.66

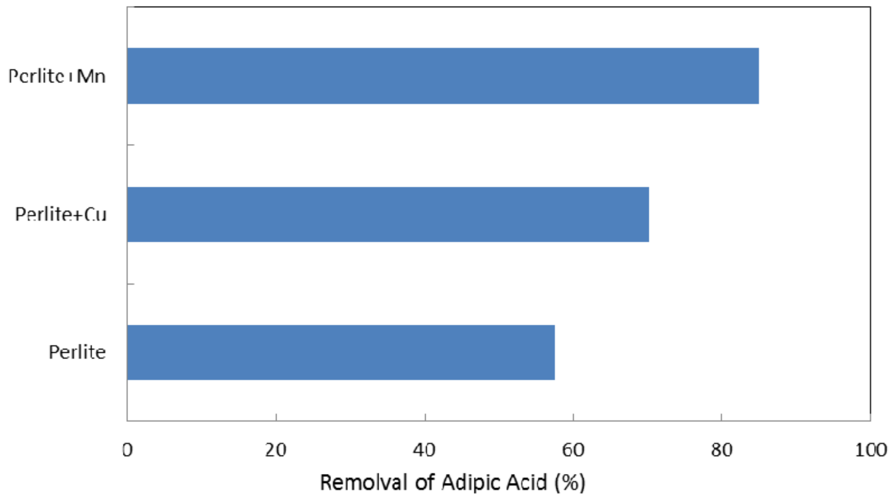

Fig. 2 Comparison of Efficiency of adsorbents at 298 K for optimum amount of adsorbent 2 g

Amount of adsorbent on the effect of adipic acid adsorption were also performed from 5 $g.L^{-1}$ to 15 $g.L^{-1}$ of adipic acid solution with the following each adsorbent masses (perlite, perlite + Cu^{+2} and perlite + Mn^{+2}) 0.5, 1.0, 1.5, 2, 2.5 g at different temperatures (298 K, 308 K, 318 K). The results in Tables 1 to 3 show that adsorption capacity of adipic acid decreased rapidly with increase of each adsorbent dose. However, conversion of adsorption capacity to adipic acid gave an increase in percentage adsorbed acid. An increase in adipic acid uptake from 16.46 to 66.8 % for perlite, 24.93 to 77.2 % for perlite + Cu^{+2}, 32.33 to 97.8 % for perlite + Mn^{+2} as the dose concentration was observed at 298 K. Similar results have been observed at the other temperatures. Nevertheless, the efficiency of adsorption exhibited a reverse trend to the removal percentage adsorptions. The highest adsorption capacity was reached in 2.5 g of adsorbent, but after 2 g of adsorbent, the capacity of adsorption increased slowly. Hence, 2 g of adsorbent is the optimum amount for this adsorption study. Fig. 2 shows the comparison of adsorption efficiency of perlite, perlite + Cu^{+2} and perlite + Mn^{+2}. It can be seen from Fig. 2 adsorption capacity of perlite has been incresing up to 57%. Whereas, It was reached 70% and 85% for perlite composite with manganese (II) and copper (II), respectively.

3.2 Adsorption isotherms

Adsorption isotherms

There are many isotherm to explain characterizetion of adsorption mechanism in the literature. In this study, we applied langmuir isotherm, and Freundlich isotherm to experimental data.

The Langmuir Eq. (3) (Langmuir 1916, Rajoriya *et al*. 2007)

$$q_e = \frac{K_L.Q_f.C_e}{1+K_L.C_e} \tag{3}$$

q_e indicates the adsorbent-phase concentrations of adipic acid, and Q_f is a particle restricting

Table 4 Freundlich and Langmuir isotherm parameters of adipic acid onto perlite, perlite + Cu^{+2} and perlite + Mn^{+2}

		Freundlich parameters				Langmuir parameters			
	Temperature (K)	Log K_f	K_f (mg·mg^{-1})	n	R^2	$1/Q_0$	Q_0	K_L (mg.L^{-1})	R^2
Perlite	298	1.66	49.32	1.47	0.993	0.034	28.97	0.0847	0.990
	318	1.39	25.44	1.35	0.987	0.044	22.54	0.0521	0.995
	328	1.23	16.30	1.24	0.984	0.059	16.72	0.0415	0.994
Perlite + Cu	298	1.73	53.82	1.58	0.985	0.029	33.56	0.0632	0.991
	318	1.44	29.60	1.42	0.977	0.039	25.50	0.0427	0.990
	328	1.28	19.22	1.31	0.972	0.046	21.63	0.0325	0.992
Perlite + Mn	298	1.86	56.75	1.69	0.987	0.025	38.82	0.0557	0.995
	318	1.53	37.52	1.55	0.975	0.033	29.66	0.0358	0.992
	328	1.33	24.43	1.40	0.974	0.041	24.37	0.0286	0.993

adsorption capacity as the surface is wholly covered with phenol molecules that aid for comparison of adsorption performance. K_L is the Langmuir constant connected to the closeness of binding sites (L.mg^{-1}). The K_L and Q_f are developed by linearizing the Eq. (3)

$$\frac{C_e}{q_e} = \frac{1}{K_L Q_f} + \frac{C_e}{Q_f} \tag{4}$$

As seen from Eq. (4), K_L and Q_f can be found by intercept and slope of the straight line. The parameters of Langmuir equation for perlite, perlite + Cu and perlite + Mn are presented in Table 4.

Freundlich isotherm (Freundlich 1906) is the most widely used isotherm equation in the literature for adsorption mechanism. The general Eq. (5) is

$$q_e = K_f . C_e^{1/n} \tag{5}$$

This equation can be linearized by a logarithmic formed Eq. (5).

$$\ln q_e = \ln K_f + (1/n) \ln C_e \tag{6}$$

Eq. (6) enables to determine constant n and K_f. Slope and intercept gives n and K_f values, respectively. All results related Freundlich have been presented in Table 4.

The obtained linear plot with a good correlation coefficient confirms that the Langmuir isotherm is a suitable isotherm for adsorption of adipic acid onto perlite, perlite + Cu^{+2} and perlite + Mn^{+2}. The R square value is about 0.99 for each studied temperature in this study. However, the Freundlich isotherm does not obey the results of adsorption at each temperature. Especially, some deviations have been observed with the Freundlich isotherm at 318 K and 328 K.

Table 5 Thermodynamic parameters for adsorption of adipic acid onto perlite, perlite + Cu^{+2} and perlite + Mn + 2 at different temperatures (298 K, 318 K, 328 K)

	Temperature (K)	ΔG^0_{ads} (kJ.mol^{-1})	K_L (mol.L^{-1})	ΔS^0_{ads} (j.mol^{-1}.K^{-1})	ΔH^0_{ads} (kj.mol^{-1})
	298	6.11	8.47×10^{-2}		
Perlite	318	7.81	5.21×10^{-2}	-84.22	-17.95
	328	8.67	4.15×10^{-2}		
	298	6.84	6.32×10^{-2}		
Perlite + Cu^{+2}	318	8.33	4.27×10^{-2}	-81.88	-17.59
	328	9.34	3.25×10^{-2}		
	298	7.15	5.57×10^{-2}		
Perlite + Mn^{+2}	318	8.80	3.58×10^{-2}	-85.26	-19.29
	328	9.69	2.86×10^{-2}		

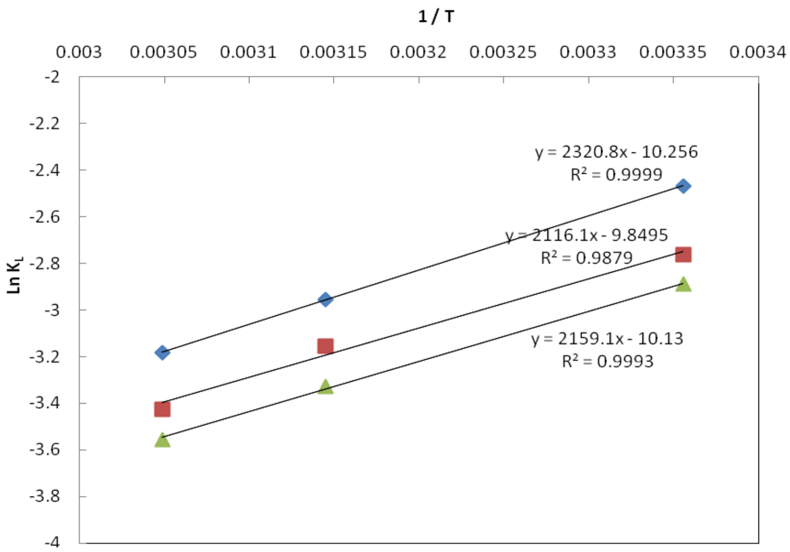

Fig. 3 Plot of ln K_L against $1/T$, ▲ perlite; ■ perlite + Cu^{+2}; ◆ Perlite + Mn^{+2}

3.4 Effect of temperature

The adsorption of adipic acid from aqueous solution has been investigated at different temperature to learn the effect of temperature in this adsorption system. The removal efficiencies have been presented in Tables 1 to 3 at three different temperatures (298 K, 318K, 328K) for all adsorbents. It can be seen from the experimental results the adsorption capacity of the perlite, perlite + Cu and perlite + Mn is decreasing with increasing temperature.

As related to the temperature effect, the thermodynamic parameters have been calculated for this adsorption system. The free energy change of adsorption ΔG^0_{ads} was calculated by using the Eq. (7)

$$\Delta G^0_{ads} = -RT \ln K_L \tag{7}$$

where R is the universal gas constant and T is the Kelvin temperature. K_L is the thermodynamic equilibrium constant for the adsorption process. It was determined by plotting ln (C_e/q_A) versus C_e and extrapolating to zero C_e as suggested by Khan and Singh (1987).

The other thermodynamic parameters, like the enthalpy change ΔH^0_{ads} and the entropy change ΔS^0_{ads} were calculated from the slope and intercept of the plots of ln K_L against $1/T$ according to following Eq. (8)

$$\ln K_L = \frac{\Delta S^0_{ads}}{R} - \frac{\Delta H^0_{ads}}{R.T} \tag{8}$$

ΔH^0_{ads} was obtained from the slope of the straight line and ΔS^0_{ads} was determined from the intercept of the graph (Demirbaş *et al.* (2006)). In order to evaluate the thermodynamic equilibrium constant K_L, the C_e/q_A values were plotted versus C_e values at 298 K, 318 K and 328 K. Linear graphs were obtained for all temperatures. The obtained K_L parameters were used to calculate the ΔG^0_{ads} function. The calculated thermodynamic parameters (ΔH^0_{ads}, ΔS^0_{ads} and ΔG^0_{ads}) at different temperatures are given in Table 5. ΔH^0_{ads} and ΔS^0_{ads} were obtained from plots of ln K_L versus $1/T$. This plot is shown in Fig. 3.

4. Conclusions

Efficiency of adsorption adipic acid by perlite, perlite + Cu^{+2} and perlite + Mn^{+2} has been investigated. Especially, perlite + Mn composites are more efficient than raw perlite and perlite + Cu^{+2}. The removal of adipic acid from aqueous media strongly depended on adsorbent amount, initial adipic acid concentration. The maximum adsorption efficiency have been obtained with 2.5 g perlite + Mn^{+2} adsorbent mass at all temperature used in this study. The maximum adsorption enthalpy was found as -19.29 kj.mol^{-1} for perlite + Mn^{+2}. Experimental data were fitted to Langmuir isotherm with high R squares at all temperatures.

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