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REVIEW ARTICLE

ANALYTICAL AND KINETIC STUDY OF SCHIFF BASE (4-HYDROXY-6-METHYL - 2H-PYRANE -2-ONE) - ETHYLIDIIMINE LIGAND ON A SURFACE OF INITIATED CALCINED IRAQI MONTMORILLONITE USING COLUMNAR METHOD

^{1,*}Mohammed H. Abdul Latif, ²Mohammed A. K. Alsouz, ³Taghreed M. Musa and ⁴Kawther A. Sadiq

^{1,4}Department of Chemistry, Ibn Al Haitham College of Education, University of Baghdad, Adhamiya, Al- Dilal Square, Baghdad, Iraq

^{2,3}Chemistry department, College of science, Al- Mustansiriya University, Al- Mustansiriya st., Baghdad, Iraq

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ABSTRACT

Removal enhancement of Schiff base (4-hydroxy-6-methyl -2H-pyrane -2-one)-ethylidiimine (ligand) on the surface of initiated calcined Iraqi montmorillonite has been investigated from an aqueous solution via columnar method. The Schiff base removal found to be dependent on initial conc. of the Schiff base, adsorbent dosage, and contact time. All columnar experiments performed at three different pH values (5.5, 7 and 8) using buffer solutions at flow rate of (3 drops/ min.), at ambient temperature (25±2) °C. The experimental isotherm data were analyzed using Langmuir, Freundlich and Temkin equations. The monolayer adsorption capacity is 3.0349 mg Schiff base ligand (L) per 1g calcined montmorillonite. This study revealed that highest removal rate 86.89% for Schiff base ligand at pH 5.5. The kinetic data for the adsorption process obeyed pseudo-second-order rate equations.

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INTRODUCTION

Schiff bases are important class of ligands due to their stability and of their easy synthesis (Iqbal *et al.*, 2007). This class of ligands containing the azomethine linkage exhibit various antibacterial, antifungal, herbicidal, biological and analytical activities (Jarrahpour *et al.*, 2004). A large number of Schiff's bases containing dehydro acetic acid (DHA) moiety already found to possess good antibacterial and antifungal activities (Saini *et al.*, 2013; Hafeez Ullah, 2012; Gupta *et al.*, 2014 and Seema *et al.*, 2012). Studies have shown that dehydro acetic acid (DHA) has both antibiotic and antifungal effects (Iqbal *et al.*, 2007). Moreover the derivatives of DHA are well known to possess potential anti-fungal and anti-microbial properties (Jednacak *et al.*, 2011). Natural mineral clays possess specific surface chemical properties, e.g., cation exchange capacity, and adsorptive affinity for some organic and inorganic compounds (Lee *et al.*, 2003). Bentonite clay deposits are mostly composed of clay minerals, a subtype of phyllosilicate minerals, which imparts plasticity and harden when fired or dried (Patterson *et al.*, 1983).

West Iraqi (Traifawi) Bentonite consists mostly of calcium – montmorillonite (60 – 65 %). Montmorillonite possess adsorption properties mainly because of their colloidal nature which stems from their very small particle size. In present work the analytical and kinetic studies of the Symmetrical Schiff Base Ligand (derived from condensation of dehydro acetic acid and diethylene triamine), from aqueous solutions, on the surface of burned initiated Iraqi Montmorillonite clay packed in glass columns have been investigated. We are focusing in this research paper and in the previous works (Mohammed *et al.*, 2014; Mohammed *et al.*, 2014) on the implementing of this class of compounds in the sewage water treatment.

MATERIALS AND METHODS

Materials

All Chemicals used were of analytical reagent grad unless otherwise are mentioned, Dehydro-acetic acid and di-ethylene tri-amine (dien) obtained from Merck, was used as supplied, Distilled and deionized water with conductivity value of $1.5 \times 10^{-5} \text{ S cm}^{-1}$ was used in all experiments.

*Corresponding author: Mohammed H. Abdul Latif

Department of Chemistry, Ibn Al Haitham College of Education,
University of Baghdad, Adhamiya, Al- Dilal Square, Baghdad, Iraq

Bentonite mineral clay obtained from the General Company for Geological Survey and Mining in Baghdad, Iraq.

Preparation of the Schiff base bis ((4-hydroxy-6-methyl - 2H-pyrane -2-one)-ethyldiimine) ligand

Ligand (L) was prepared by mixing on ethanolic solution (100 ml) (3.26 g, 0.02 mole) of dehydro acetic acid and (3g, 0.01mole) of diethylene triamine (dien) in 50 ml of ethanol, and stirring with reflux on water bath for about 9 h. Ligand (L) was separated out, filtered, and washed with cold ethanol, and diethyl ether. It was then dried in vacuum over analysis calcium chloride, the mixture to room temperature overnight. m.p. 44 - 46 °C . The yield was Ca 60%, (Scheme 1). Exact mass 403.17 g/mol, of $C_{20}H_{25}N_3O_6$ formula with good solubility in Me-OH (warming) Et-OH (warming), DMF and DMSO, to give clear yellow, micro elemental analysis % calc. (found) %C 59.54 (58.69), %H 6.25 (5.81), %N 10.42(11.61). As well as data for H^1 NMR: ppm(13.5 ppm, singlet (2H), 5.6 ppm (S,NH), 2-3.3 ppm, t, 4H), (3.95,6H), 3 ppm (5,6 H) for C^{13} NMR 185 C=N, 178 CH_3 -C=N, (165, 162 C=O, CH=CH), 20 ppm CH_2 - CH_2 , 80, 110,118, CH_2 -N, CH_3 - pyridine ring.

Characterization of ligand (L) and its metal complexes

Electronic spectra of the new ligand is recorded in the region 800-200nm on Shimadzu 670 spectrophotometer. IR-spectra were recorded on PC. Shim adz FT-IR spectrophotometric – Japan model as KBr and CsI-disc in the range 400-4000 cm^{-1} . H^1 & C^{13} NMR spectra were recorded in DMSO (d6) Bruker ACF (300) spectrometer at 300MHZ reference to TMS (0ppm).

Preparation of buffer and stock solutions

Three phthalate and phosphate buffer solutions of pH values (5.5, 7, and 8) were prepared respectively (Robinson *et al.*, 1968), also a three stock solutions of the prepared ligand (L) of 2000 mg /L concentration were prepared in 100 ml volumetric flask using the above buffer solutions.

Procedure for synthesis of granulated Iraqi Burned Initiated Na – montmorillonite

In order to convert calcium - montmorillonite to sodium – montmorillonite, the samples of impure bentonite have been treated according to our own method (Mohammed *et al.*, 2012) to produce Na- Montmorillonite (percent). Then the resulting bentonite burned at 650 °C to make an ion – exchange column ready to be used. The surface area (123 m^2 /g) was estimated using methylene blue (MB) adsorption method (Potgieter, 1991).

Columnar adsorption procedure

Three standard solutions (30 mg /l concentration) of ligand (L) were prepared from stock solutions of ligand (L) in different pH values (5.5, 7and 8) using buffer solutions. UV-Visible scanning spectrum has been recorded and wavelength value corresponding to the maximum absorption found to be at (312 nm for pH = 5.5), (234 nm for pH = 7), and (230 nm for pH = 8), as shown in (Figure 1), (Figure 2), and (Figure 3) respectively, these values chosen for all measurements. Seven standard solutions in the range of (5 - 35 ppm or mg /L concentration) of ligand (L) were prepared from stock solutions of ligand (L) at different pH values (5.5, 7and 8) using buffer solutions. The absorbance of each solution was measured at max. (312 nm for pH = 5.5), (234 nm for pH = 7), and (230 nm for pH = 8) respectively against blank (buffer solution Corresponding to pH value). The calibration curves were shown in (Figure 4), (Figure 5), and (Figure 4) respectively.

A six glass columns (50 cm X 10 mm i.d.) filled with known mass (1gm of surface area 23.904 cm^2 physically calculated) of (Initiated Burned Na - montmorillonite) corresponding to bed heights of (17 mm) have been prepared in order to run(10 ml) of each of (50 – 90 mg/L) ligand (L) solutions adjusted to different pH values (5.5, 7and 8) using buffer solutions at flow rate of (3 drops/ min.), at ambient temperature (25±2) °C.

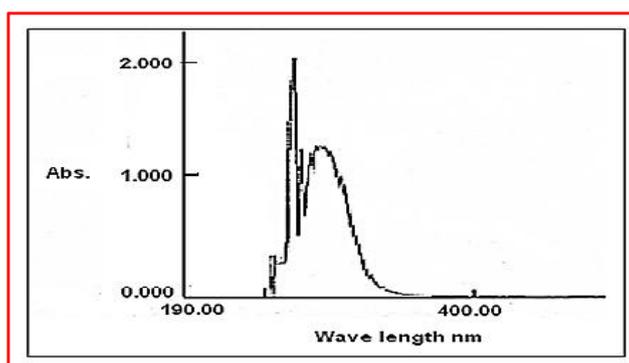
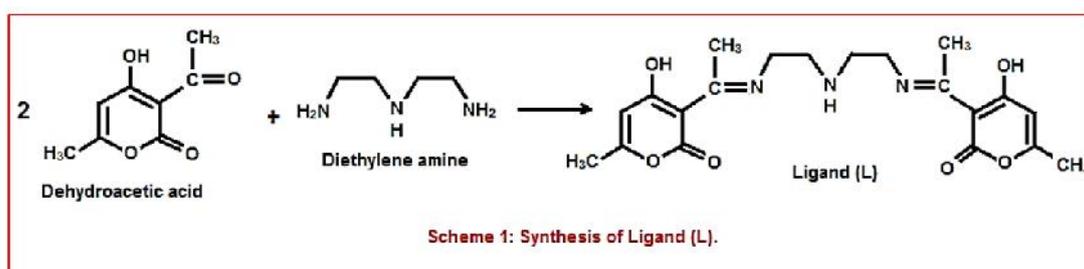


Figure 1. Scanning graph of Ligand (L), Buffer soln. pH = 5.5, Conc.= 30 ppm., at max. = 312 nm

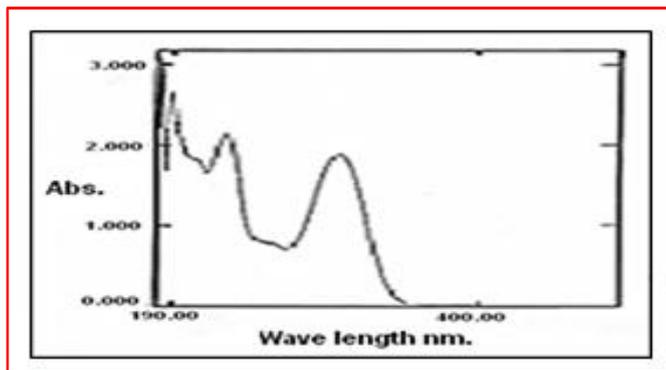


Figure 2. Scanning graph of Ligand (L), Buffer soln. pH =7, Conc.= 30 ppm., at max. = 234 nm

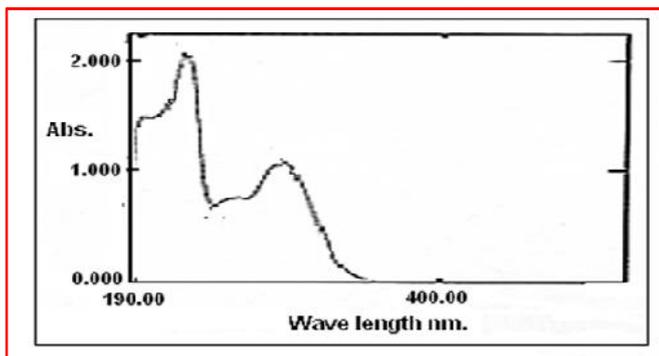


Figure 3. Scanning graph of Ligand (L), Buffer soln. pH = 8, Conc. = 30 ppm. , at max. = 230 nm

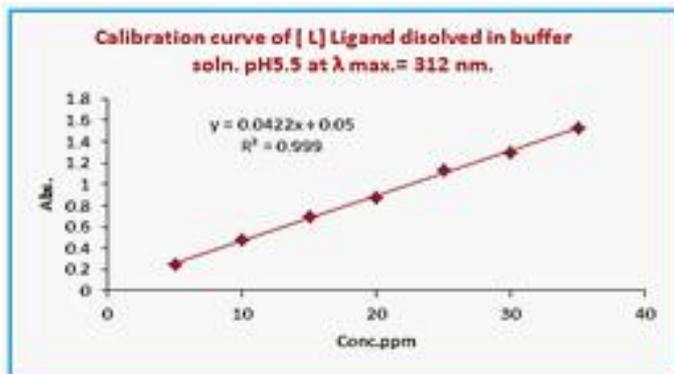


Figure 4. Calibration curve of Ligand (L) dissolved in buffer soln. pH = 5.5 at max. = 312 nm

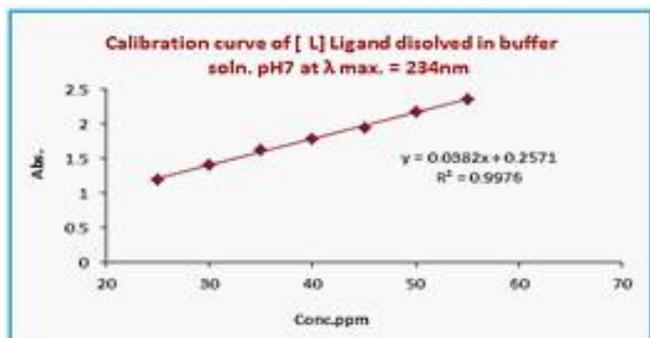


Figure 5. Calibration curve of Ligand (L) dissolved in buffer soln. pH = 7 at max. = 234 nm

Then the absorbance of each eluate has been measured at a given max. using (UV-Vis – 1800 Shimadzu Spectrophotometer). The equilibrium adsorption uptake and removal percent of ligand (L) from the aqueous solution q_e (mg/g) was calculated using the relationship given at the bottom of the (Table 1).

RESULTS AND DISCUSSION

Calibration graphs

Three linear calibration curves for ligand (L) were obtained at different pH values (5.5, 7 and 8) (Figs.4, 5, and 6), which show that Beer's law was obeyed in the concentration range of (5 – 35) mg L^{-1} . The values of Coefficient of determination R^2 , Limit of detection (sensitivity) LOD, in addition to other parameters given in (Table 2) reveal that the pH 5.5 is a better value to be selected for this work.

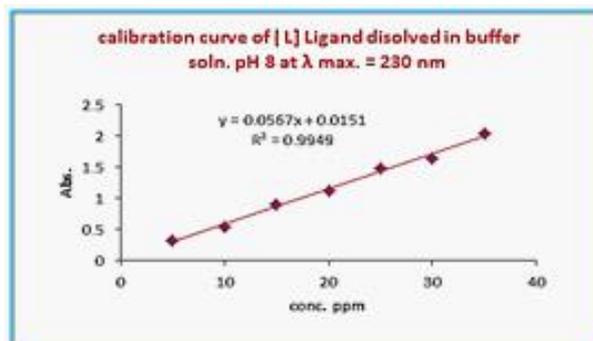


Figure 6. Calibration curve of Ligand (L) dissolved in buffer soln. pH = 8 at max. = 230 nm

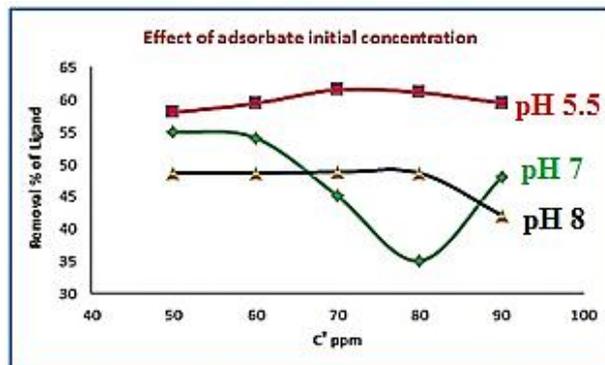


Figure 7. Adsorbate concentration effect of Schiff base ligand (L) at different pH values

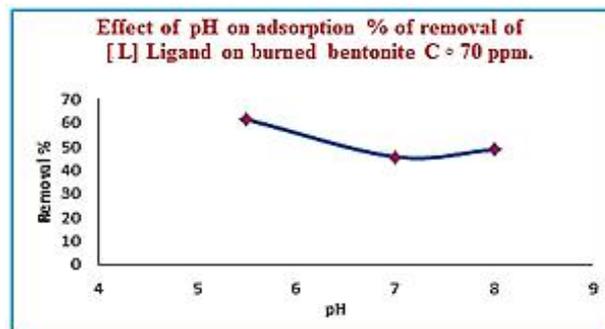


Figure 8. Effect of pH on adsorption % of removal of Ligand (L) on burned bentonite, C = 70 ppm.

Table 1. Adsorption parameters of ligand (L) on initiated burned Iraqi bentonite clay mineral

Sorbent ligand (L) concentration C ₀	pH 5.5			pH 7			pH 8		
	C _e	Removal %	q _e	C _e	Removal %	q _e	C _e	Removal %	q _e
50	21.03	57.94	0.289	22.15	55.70	0.278	25.70	48.59	0.243
60	24.41	59.31	0.355	27.47	54.21	0.325	30.84	48.54	0.291
70	26.98	61.46	0.430	38.13	45.53	0.318	35.90	48.70	0.341
80	31.19	61.01	0.488	51.55	35.56	0.284	41.15	48.56	0.388
90	36.55	59.38	0.534	46.89	47.90	0.431	46.21	42.04	0.437

Amount adsorbed $q_e = (C_0 - C_e)V/W$ (mg of adsorbate / g of adsorbent), removal % = $100(C_0 - C_e) / C_0$, Where C_0 is the initial sorbate concentration (mg/L), C_e the equilibrium adsorbate concentration (mg/L), V is the volume of solution in L and w is the mass of the adsorbent in g (Zlem and Demet, 2000).

Table 2. Spectral characterization and statistical data of the regression equation for Ligand (L) adsorption on burned Iraqi bentonite

Parameter	Ligand (L)		
	pH 5.5	pH 7	pH 8
Linearity range (ppm)	5 -35	25 -55	5 -25
Regression equation			
Intercept	0.050000	0.257143	0.015143
Standard deviation of intercept	0.013339	0.034381	0.040514
Slope	0.042243	0.038221	0.056736
Standard deviation of slope	0.000597	0.000834	0.001812
Coefficient of determination R ²	0.999004	0.997626	0.994927
Standard deviation of the regression	0.015783	0.022062	0.047937
Standard error of Y & X axis STEYX	0.015783	0.022062	0.047937
Limit of detection (sensitivity) LOD	1.22	1.91	2.77
Limit of quantitation LOQ	3.7	5.8	8.4

Table 3. Experimental data of Adsorption Isotherms

Langmuir		Freundlich		Temkin	
1 / C _e	1 / q _e	Log q _e	Log C _e	q _e	Ln C _e
0.047	3.36	1.322	-0.539	3.045	0.289
0.04	2.816	1.387	-0.449	3.194	0.355
0.037	2.465	1.431	-0.386	3.295	0.43
0.032	2.049	1.494	-0.311	3.44	0.488
0.027	1.872	1.562	-0.272	3.598	0.534

Table 4. Isotherm Models Constants and the coefficient of determination (R²) of ligand (L) from Aqueous Solution

Adsorbent	Langmuir isotherm			Freundlich isotherm			Temkin		
	q _m (mg/g)	K _L (L/mg)	R ²	K _f	N	R ²	A (L/g)	B	R ²
Initiated burned Iraqi bentonite clay	3.0349	0.00424	0.978	0.0094	0.880	0.9747	0.09108	0.45626	0.9963

Where q_m(mg/g) and K_L (L/mg) are the Langmuir constants, K_f and n are the Freundlich constants, Temkin constants A and B

Table 5. Effect of contact time on the adsorption of Ligand (L) (70 mg / mL) aqueous solution

Initial Conc. (mg/L)	Flow rate drop / min.	Flow rate ml / min.	Time (minute)	Equilibrium Conc. (C _i) (mg/L)	Removal %	q _t (mg/g)	Ln (q _e - q _t)	t/q _t	Equilibrium Time (minute)
70.000	2	0.1	100.00	9.170	86.89	0.3370		296.735	66.666
	3	0.15	66.666	9.170	86.89	0.3370		197.822	
	4	0.2	50.000	13.457	77.37	0.2999	- 3.2941	166.722	
	6	0.3	33.333	20.569	66.17	0.2565	- 2.5195	129.953	
	12	0.6	16.666	23.461	55.15	0.2137	- 2.0939	77.988	

Effect of adsorbate initial concentration (C) of ligand (L)

In order to study the adsorbate concentration on a fixed 1g clay bed at different pH values (5.5, 7 and 8) using buffer solutions, and at ambient temperature (25±2) °C, the C₀ values from (Table 1) have been plotted against removal % of ligand (L) (Figure 7). We notice that the highest adsorption of ligand occurs at pH = 5.5 and the increase in the initial concentration of ligand increases the amount of ligand removed to the extent that maximum adsorption capacity of the clay surface has been reached.

Effect of pH

The pH impact on the adsorption of ligand (L) On the surface of burned initiated bentonite has been studied by passing down 10 ml of (70 ppm of ligand solution) adjusted to different pH values (5.5, 7 and 8) using buffer solutions a three columns of 1g clay bed with constant flow rate (3 drops min⁻¹) at ambient temperature (25±2) °C. plotting removal % against the pH values (Figure 8) reveal that the best pH value of adsorption of ligand (L) was (pH = 5.5).

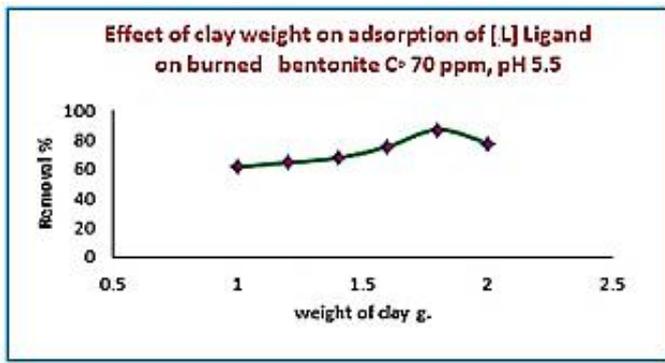


Figure 9. Effect of clay weight on adsorption removal % of Ligand (L) on burned bentonite, C = 70 ppm

This is perhaps due to the enhancement of protonation of NH_2 of the ligand which increase the attraction of the ligand to the clay surface.

Effect of clay weight

The weight effect of clay on the removal percent of the ligand has been examined by passing down 10 ml of 70 ppm ligand

Table 6. The pseudo- first and second- kinetic order parameters for the adsorption of 70 ppm.of Ligand (L) at $(25\pm 2)^\circ\text{C}$ under optimum conditions

q_e Experimental (mg/g)	The pseudo-first-order kinetic models			The pseudo-second-order kinetic models		
0.337	q_e calculated (mg/g)	K_1 (min^{-1})	R^2	q_e calculated (mg/g)	K_2 (g/mg/min)	R^2
	0.2157	-0.43567	0.9726	0.3926	0.00405	0.9935

solution at pH 5.5 a five packed column (1-1.8 g) with constant flow rate of (3 drops/ min.) at room temperature $(25\pm 2)^\circ\text{C}$. the plot of removal percent vs clay weight (Figure 9) illustrate that highest ligand (L) adsorption occurs on clay bed of 1.8 g weight. This means increasing absorption with increasing weight of clay to the upper limit of clay capacity, and adsorption surface area

Effect of ionic strength

Ionic strength factor was examined by adding different concentrations of sodium chloride (0.1- 0.7 M) to (10) ml of 70 ppm ligand (L) solutions run through four adsorption columns of clay bed weight of 1.8 calculation of (q_e), and plotting (q_e) against the molar concentration of NaCl (Figure 10) shows that the adsorption decreases with increasing of Na^+ and Cl^- ions concentration on the clay bed surface. It seems that ions may prevent the ligand species to reach the adsorption active site of the clay.

Adsorption Isotherms

Adsorption isotherms are mathematical models that describe the distribution of the adsorbate species among liquid and adsorbent, based on a set of assumptions that are mainly related to the heterogeneity/homogeneity of adsorbents, the type of coverage and possibility of interaction between the adsorbate species. Adsorption data (Table 3) are usually described by adsorption isotherms, such as Langmuir, Freundlich, and Temkin isotherms (Senthil Kumar and Kirthika, 2009).

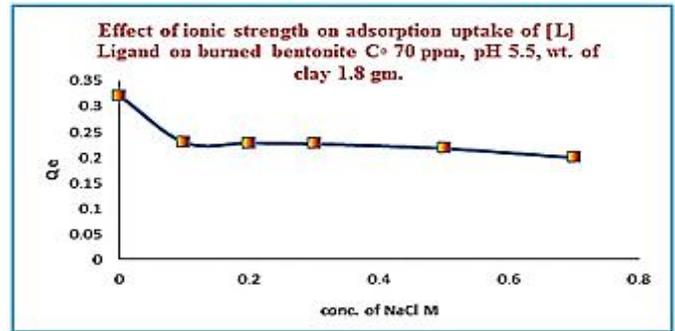


Figure 10. Effect of ionic strength on adsorption removal % of Ligand (L) on burned bentonite, C = 70 ppm

The set of experimental results as presented in (Figure 11) at room temperature (25 ± 2) were fitted with the Langmuir, Freundlich, and Temkin models. Adsorption isotherms were obtained and the adsorptive capacity interpreted using both models. The Langmuir, Freundlich, and Temkin adsorption constants calculated from the corresponding isotherms with the coefficient of determination (R^2) are presented in (Table 4).

Effect of flow rate (contact time)

Adsorption of ligand (L) was measured at five different flow rates for a solution of ligand (L) (70) ppm. concentration. From (Figure 12), the plot reveals that removal percent of ligand (L) is higher at low flow rates.

This is probably due to higher contact time at these low flow rates. (Table 5) show the calculated parameters that illustrate the importance of flow rate and contact time.

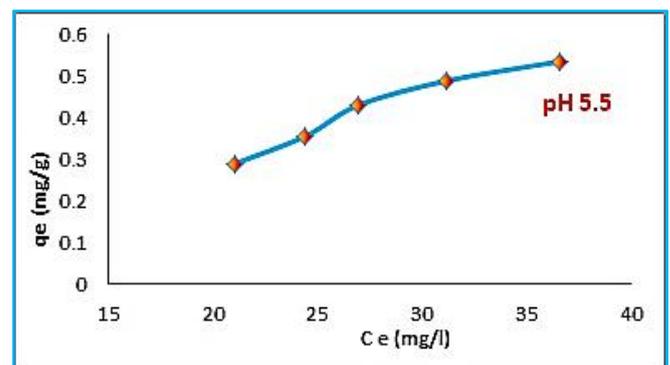


Figure 11. Adsorption isotherm of Ligand (L) at pH = 5.5

Adsorption kinetics

Kinetic models are used to examine the rate of the adsorption process in the present work; the kinetic data obtained from the studies have been analyzed by using pseudo-first-order and pseudo-second-order models.

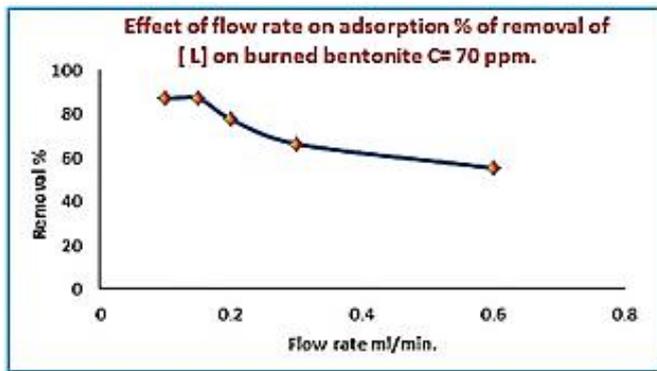


Figure 12. Effect of flow rate on adsorption removal % of Ligand (L) on burned bentonite, C = 70 ppm

The first order equation of Lagergren is generally expressed as follows.

$$dq/dt = k_1(q_e - q_t)$$

Where q_e is the amount of Ligand (L) adsorbed at equilibrium (mg/g), q_t is the amount of Ligand (L) adsorbed at time t (min^{-1}), and k_1 is the rate constant of pseudo-first-order adsorption. If it supposed that $q=0$ at $t=0$, then:

$$\ln(q_e - q_t) = \ln q_e - k_1 t$$

The pseudo-second-order kinetic rate equation is expressed as follows.

$$dq/dt = k_2(q_e - q_t)^2$$

Where k_2 is the rate constant of pseudo-second-order sorption (g/mg/min). The integrated form of Equation when ($t=0$ and $q_t=0$) the following expression is obtained:

$$t/q_t = 1/k_2 q_e^2 + t/q_e$$

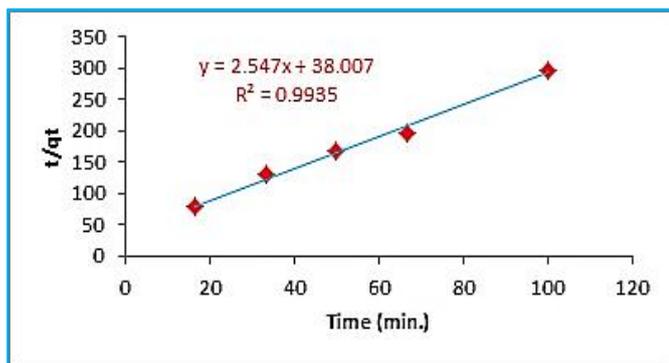


Figure 13. The pseudo-second-order kinetic models for the adsorption of 70 ppm. of Ligand (L) at (25±2) °C under optimum conditions

The rate constant k_1 , k_2 and q_e calculated from the slopes and intercepts of the linear plot of $\ln(q_e - q_t)$ or (t/q_t) against t respectively (Figures 13 and 14). It is seen that Ligand (L) adsorption is well described by the pseudo second order reaction kinetic. Moreover, the correlation coefficient (R^2), of second order reaction kinetic (0.9935) is higher than that of the

first order reaction kinetic (0.9726) and greater value of rate constant for the adsorption data. While the value of q_e experimental is approximately equal q_e calculated for the second order reaction kinetic, but it is highly different for the first order reaction kinetic. (Table 6) shows the rate constants, q_e (experimental and calculated) and correlation coefficient (R^2) for pseudo first and second order reaction kinetic.

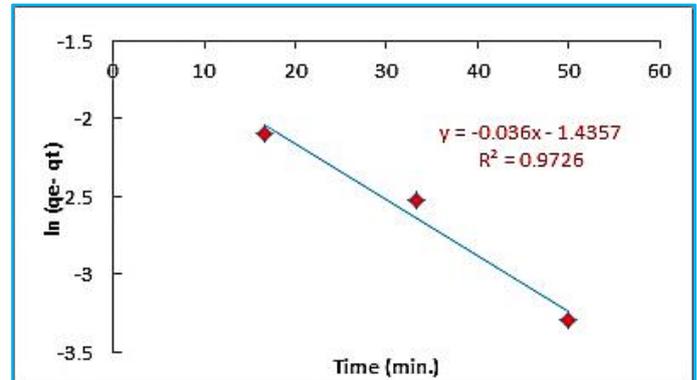


Figure 14: The pseudo-first-order kinetic models for the adsorption of 70 ppm. of Ligand (L) at (25±2) °C under optimum conditions

Conclusion

The present work reveals that Iraqi initiated burned montmorillonite has an effective adsorption aptitude for the removal of Schiff base ligand (L) from aqueous solutions. Highest removal achieved at pH 5.5. The ligand (L) uptake is very rapid in the initial stage and decreases while approaching equilibrium. The removal percent of Schiff base ligand (L) increases with the increase in adsorbent dosage and decreases with increase in initial ligand (L) concentration. The equilibrium time increases with initial ligand (L) concentration. Experimental data are in good coincidence with Langmuir, Freundlich and Temkin adsorption isotherm models, and have shown a good fitting to the experimental data. Adsorption of ligand (L) obeys pseudo-second order equation with good correlation.

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