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Degradation Study of Phenazin Neutral Red from Aqueous Suspension by Paper Sludge

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Abstract

The potential ability of paper sludge to remove neutral red (NR) from aqueous solution was investigated using UV-visible and FTIR spectroscophotometers. The stability of the optical properties of NR was assessed in terms of evolution of the main absorption bands of NR in aqueous solution and in NR/incinerated sludge suspension as a function of pH and exposure time. The results showed that, the adsorption kinetic of NR onto incinerated paper sludge was fast as a result of 60.09% removal efficiency obtained within 80 minutes at pH 5. The adsorption reaction was perfectly described by pseudo- second-order kinetic model. The profile of isotherm adsorption of NR onto incinerated sludge was S-shape type. Incinerated sludge exhibited excellent performance for adsorption of NR with a maximum of 374. 98 mg/g. The equilibrium adsorption data were well fitted with Freundlich model. FTIR analysis revealed the strong interaction forces operating on heterogeneous surface of the incinerated sludge between the dissociation of the oxygenated groupings, which are in general acid functional, and dimethylamine goup [$-N^+(CH_3)_2$] of the NR dye which could be used to explain the high adsorption capacity of cationic dye onto incinerated sludge. These findings can support the design of remediation processes and also assist in predict their fate in the environment.

Keywords: Incinerated sludge; Kinetic; Isotherm; Neutral red; UV visible; FTIR

Introduction

The industry of paper rejects significant amounts of the waste whose setting in discharge was the means the elimination of simplest and cheapest. One distinguishes several dies from assessment of sludge including: the setting as a cover for discharges, the sludge incineration at a prohibitory cost and present a risk related to the pollutant gas impact on the environment such as that of the dioxin (ADEME, 1999 /CE) [1], energy valorization (production of biogas like source of heat and of electricity), biological or agricultural valorization (production; manure and of compost) and valorization in the sector of building. The choice of a die must be dependent on the cost of installation, origin of sludge, added-value of the product which results from it and the impact which the die retained on the environment could have. The setting discharges some (also called storage) proves to be a technique little developing and is legally prohibited in many countries (directive 1999/31/CE) [2].

Dyes effluents released (approximately 7. 10^5 tons) into the environment by technological activities pose a serious threat to the environment. Their presence in water, even at very low concentrations, may significantly affect photosynthetic activity in aquatic life due to reduced light penetration.

Due to their synthetic origin and complex aromatic molecular structures, which make them more stable, non-biodegrade, conventional methods of treatment of the aqueous solutions containing dye (the oxido-reduction and the exchanging resins of ions [3], coagulation/ flocculation [4], membrane separation [5], the biological methods [6] and more recently the advanced processes of oxidation [7], do not allow obtaining threshold of pollution lower or equal to the maximum permissible concentration (MPC) imposed by the environmental recommendations. Adsorption is one of the highly efficient methods to remove colored textile contaminants from wastewaters. The adsorption appeared a very effective method for the reduction of the color in aqueous mediums. Many low-cost adsorbents for dye removal from mineral waste [8], agricultural wastes, microbial biomass [9], higher plant biomass [10], tree fern [11], orange peel [12], date pits [13], palm kernel fiber, sawdust [14], peanut hull [15], neem leaf [16], de-oiled soya [17], moss Rhytidiadelphus squarrosus [18], activated carbon [19] rice husk-based porous carbon [20] magnetic particles [21] and paper sludge [22]. The aim of the present study was to assess the potential ability of locally available and highly efficient paper sludge for removal of neutral red as dye model from the aqueous solutions. The stability of the absorption band maxima of NR in aqueous solution and in NR/ incinerated sludge suspension were assessed in terms of evolution the main absorption bands and adsorption efficiency as a function of pH solution using UV-visible spectroscopy monitoring. The equilibrium and kinetics of adsorption of Neutral red from aqueous solution to incinerated sludge were investigated. Adsorption kinetic reaction was determined quantitatively by the pseudo-first- and second-order models. The equilibrium adsorption isotherm data were analyzed with Langmuir and Freundlich. The incinerated sludge untreated and treated with NR were characterized by FTIR spectroscopy.

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Materials and Methods

The sludge of purification plant of industrial water "workshop 22" of production unit GIPEC (Saida, Algeria) was used in this study. The sludge sample of paper mill was washed, dried with the air during several days and then incinerated at 250°C during 2 hours in a muffle furnace. The material resulting from the incineration of sludge was filtered. A phenazin dye, Neutral red, NR (CI 50040, MW=319.50 $g.mol^{-1}$, λ max=520 nm, ε =25000 cm^{-1} $mole^{-1}dm^{-3}$) and a thiazin group cation, Methylene blue, MB (MW= 319,5 $g.mol^{-1}$, λ_{max}=665 nm, ε =95000cm^{-1} $mole^{-1}dm^{3}$) from Across product for microbiological analysis and used without any further purification. Molecular structures of basic dyes: (a) Methylene blue (MB) and of Neutral red (NR) are shown in Figure 1.

Aqueous dye solution stock was prepared by dissolving accurately weighed neat dye in distilled water to the concentration of 0.1g/L. Experimental solutions were obtained by successive dilutions.

The initial pH of the dye solutions was adjusted by buffer solutions of NaOH /HCl (0.1M) using (WTN: WISSENSCHAFLLICH TECHNISECHE WERKSTÄTTEN; weilehein Allemagne pH-330) digital pH-meter. The incineration of the paper sludge sample was carried out in a muffle furnace (Nabertherm, ZAH 2002). X-ray fluorescence was performed on a spectrometer of mark (Oxford). The sample was prepared (pearl borated) and was subjected to a source of X-radiation of fluorescence characteristic of its chemical composition. The separation of adsorbent /adsorbate suspension was performed by the centrifuge (EBA-Hetlich) at 4000 rpm for 10 min. Vis-absorption spectra of the dye in aqueous solution and adsorbed on incinerated sludge were obtained by (Model: UV – 2401 (PC) SHIMADZU – corporation spectrometer) in the range 350-800 nm, using 1cm optical pathway cells at maximum wavelength of dye. Fourier transformed infrared (FTIR) spectra were measured in dispersed incinerated paper sludge in KBr pellets (1/200 w/w) with Perkin-Elmer spectrometer in the range 4000- 400 cm^{-1}, with resolution of 4°.

The effect of initial pH was performed on incinerated paper sludge suspension in 50 mg/L of dye, (solid /liquid ratio of 1g/L) and over a range of pH values from 2 to 12. The initial pH values of solutions were adjusted with 0.1M HCl / NaOH solutions. The suspensions were stirred for 80 mn, and then separated by centrifugation. Dye concentrations in the supernatant solution were estimated by UV-visible spectrophotometer at maximum wavelength of NR dye.

Batch kinetic adsorption experiment was carried out in flasks containing 1L of NR solutions with defined concentrations. pH value of solutions was adjusted at optimum pH solution (pH=5). An amount of 1g/L of incinerated paper sludge was then added and flask was agitated at 298 K. Samples were taken from flasks at the predeterminated time intervals and adsorbent was separated from the NR/incinerated sludge suspension by centrifugation. Dye concentration in the supernatant was estimated by UV-visible spectrophotometer.

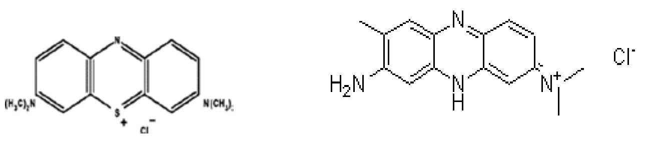

Figure 1: Molecular structures of basic dyes: (a) Methylene blue (MB); (b) Neutral red (NR).

SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	SO_3	SiO_2/Al_2O_3	LF
14.12	9.84	2.34	26.5	1.76	0.42	0	3.9	1.43	41.93
pH	CEC (mg/g)			CEC (meq/100g)			TSS (m^2/g)		
8.5	75.61			23.66			122.53		

LF: Loss of Fire CEC: Cation Exchange Capacity TSS: Total Specific Surface

Table1: Chemical composition and main properties of the incinerated paper sludge.

The removal efficiency η (%) and the amount of dye adsorbed per unit weight of incinerated paper sludge at equilibrium Q (mg/g) were calculated based on following equations Equation (1) and Equation (2):

$$\eta(\%) = \frac{(C_0 - C_e)}{C_e}100 \quad (1)$$

$$Q\left(\frac{mg}{g}\right) = \frac{(C_0 - C_e)}{m}\upsilon \quad (2)$$

where C_o and C_e are the initial dye concentrations and the equilibrium dye concentration (mg/L); V is volume of the solutions (L), and m is the weight of the incinerated paper sludge (g).

Results and Discussion

X-radiation of fluorescence analysis

Table 1 displays the results of chemical composition in the incinerated sludge. The major elements of the incinerated paper sludge sample, expressed in terms of oxide, are primarily of silica, alumina, lime and a strong water content with the moderate presence of the elements such as sodium, magnesium, potassium and iron. The loss of fire raised about 41.93%, sign of the important presence of the organic matter and calcite. The value of pH of the incinerated sludge is estimated at 8.5. This alkalization of the suspension can be attributed to the progressive dissolution of the carbonates (calcite) basically present in incinerated sludge.

Cation exchange capacity and total specific surface

In order to quantify the Cation Exchange Capacity (CEC) and Total Specific Surface (TSS) of MB dye on incinerated paper sludge, the equilibrium adsorption isotherm was carried out using Methylene blue methods [23]. As elucidated in Figure 8, the profile of the MB isotherm is L-3 type, is obtained when the polymoléculaires layers appear only when surface is almost entirely covered with a monomolecular layer [24]. The CEC and SST values were of 75.61 meq /100gr of incinerated sludge and 122.53 m^2/g respectively.

Fourier transformed infrared analysis

As shown in Figure 8, the spectrum of the pure incinerated sludge allowed identifying the characteristic absorption bands of the acid functional groupings of the incinerated sludge such as cellulose (3409.9, 2929.7, 2500, 1427 and 1103.2 cm^{-1}), kaolinite (3693.4 cm^{-1} (OH^-), 912 cm^{-1} (Al-OH), 700 cm^{-1}, 468.7cm^{-1} (Si-O) and 538.1cm^{-1} (Si-O-AL)), calcite (2515, 1797, 1427.2, 875.6 and 710 cm^{-1}) and talk (675 cm^{-1}). The band at 1630 cm^{-1} is also attributed to physical adsorbed water.

Effect of initial pH solution of NR dye

Qualitative study: The evolution of absorption band maxima of NR in aqueous solution and in NR/ incinerated paper sludge suspension at various pH.

Table 2 illustrates the position and the evolution of the main absorption bands of NR in aqueous solution and upon adsorption on incinerated paper sludge suspension at various pH.

The visible spectra of NR dye, in aqueous solution at various pH values, exhibits two forms: NR^+H and NR, which are in equilibrium with each other. NR, which has absorption bands in the range of 451-453.5 nm under neutral and alkali conditions (pH> 6), whereas NR^+H, is found to lie between 519.5 and 521.5 nm under acidic media (pH ≤6).

As seen in Figure 2, the interactions of NR (NR^+H and conjugate base form, NR) with incinerated sludge differ in certain aspects from those in aqueous solutions. The adsorption process of NR^+H in NR/ incinerated sludge suspension under acidic media (pH= 2.41-6.04) was accompanied by a blue shift of the main absorption bands to shorter wavelengths. The highest shift was observed in pH range of 4.26-6.04. However, under alkali conditions (pH=8.15-12.1) the adsorption profile of NR has a nearly constant absorption band maxima. The shift did not exceed 3nm over the pH range of 8.15-12.1, but is higher for NR^+H over pH ranged between 4.26 and 6.04, being 17, 18.5 and 18.5 nm at pH 4.26, 5.24 and 6.04 respectively. Besides, the results showed a slight increase in pH in NR/incinerated sludge suspension under acid conditions, while these of NR/incinerated sludge were decreased under alkali conditions.

Initial pH	NR /Aqueous solution		NR /Incinerated sludge suspension		Equilibrium pH
	Species (nm)		Species (nm)		
	NR^+H	NR	NR^+H	NR	
2.41	519.5	-	518.5	-	2.62
4.26	520	-	503	-	5.82
5.24	519.5	-	501	-	6.34
6.04	521.5	-	503	-	6.55
8.15	-	452.5	-	453	7.02
10.14	-	451	-	454	7.74
12.01	-	453.5	-	450.5	11.45

NR: Conjugate base form; NR^+H: protonated cation of NR dye

Table 2: The position of the absorption band maxima of NR in aqueous solution and upon adsorption on incinerated sludge as a function of pH for [NR] =50mg/L, [Incinerated sludge]=1g/L, T=298K and contact time of 80 min.

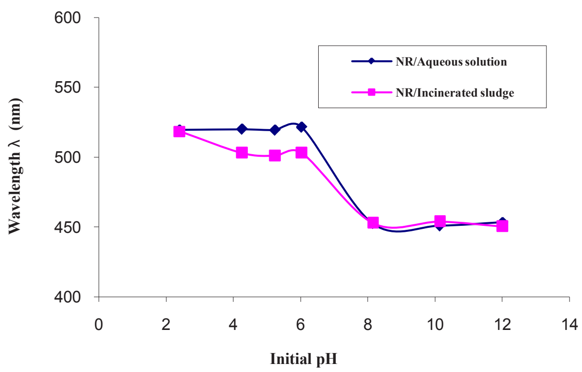

Figure 2: Effect of the pH on the absorption band maxima of NR in aqueous solution and upon adsorption on incinerated sludge for [NR] =50 mg/L, [incinerated sludge]=1g/L, T=298K and contact time of 80 min.

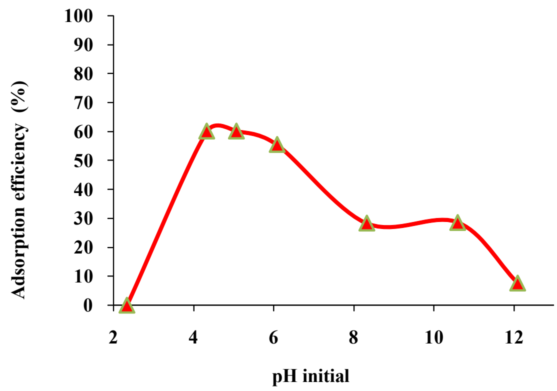

Figure 3: Effect of pH on the adsorption efficiency of NR onto incinerated sludge for [NR] =50 mg/l, m/v=1g/L, T=298K and contact time of 80 min.

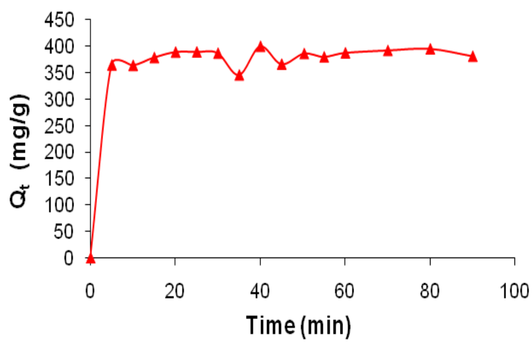

Figure 4: Adsorption capacity of NR adsorbed onto incinerated sludge as a function of contact time for [NR] =50 mg/l, m/v=1g/L, pH=5 and T=298K.

These observations are consistent with chemical reaction between NR^+H monomer cations and dissociated acid functional groupings of incinerated sludge surface and the change in the environmental polarity/acidity of negatively-charged incinerated sludge surface with respect to water surrounding under alkali conditions respectively.

Quantitative study: The evolution of adsorption efficiency of NR in NR/ incinerated paper sludge suspension at various pH: It is commonly accepted that in adsorbate/adsorbent systems, the potential of the surface charge is determined by the activity of ions (e.g. H^+ or pH). The effect of pH solution on the adsorption efficiency of NR onto incinerated sludge was studied over a range of pH values of 2 to12. The pH significantly affected the adsorption rate of NR dye. As shown in Figure 3, the dye adsorption efficiency increased as the initial pH was increased from 2 to 5, and then decreased significantly to reach a percentage of 7. 65% to pH = 12. However no adsorption is observed with pH =2. The maximum dye adsorption of 60.09% of NR was achieved with an initial pH of 5. For this reason, pH 5 is selected for subsequent experiments. The high adsorption percentage observed at pH=5 is due to the presence of dissociated acid functional groupings,

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which are in general oxygenated functions, on the incinerated sludge surface which boost the attractive interactions between dissociated groupings and NR cations, consequently improving the masse transfer and adsorption rate [25].

Adsorption kinetic

The adsorption kinetics of NR on incinerated paper sludge was measured in the range of 0 to 80 min, by varying the equilibrium time between adsorbate/adsorbent. The adsorption capacity of NR adsorbed on incinerated sludge as a function of contact time was illustrated in Figure 4. The results indicated that the process is found to be very rapid initially, and the equilibrium is achieved within 5 min. The maximum efficiency of 66.78 % was obtained within 80 min at a pH=5. On the other hand, results shown in Figure 5 and 6 indicated that the temperature and pH of system increased as the initial dye concentration was increased within contact time, which can be explained by an endothermic and chemical adsorption as a consequence of an exchange phenomenon between organic cations and incinerated paper sludge respectively. The adsorption data were treated with a pseudo-first order and pseudo-second order kinetic reaction models.

The pseudo-first order equation of Lagergren [26] is generally expressed as follows Equation (3):

$$\frac{dQ_t}{dt} = K_1(Q_e - Q_t) \tag{3}$$

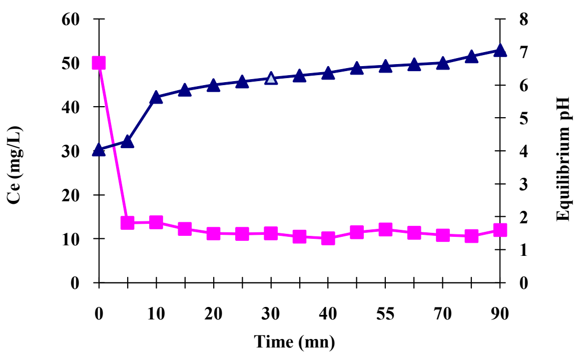

Figure 5: Evolution of initial dye concentration and pH as a function of contact time during the adsorption of NR onto incinerated sludge for [NR] =50 mg/L, pH=5, m/v=1g/L and T=298K.

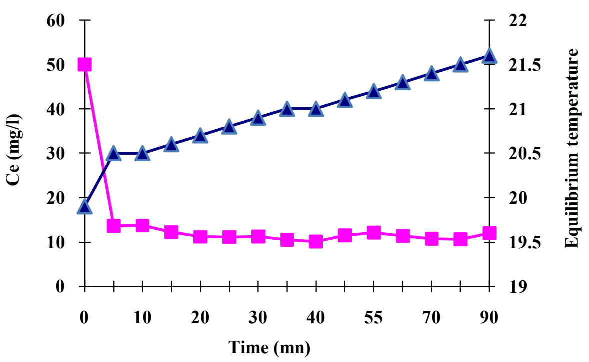

Figure 6: Evolution of initial dye concentration and temperature as a function of contact time during the adsorption of NR onto incinerated sludge for [NR] = 50 mg/L, pH=5, m/v=1g/L and T=298K.

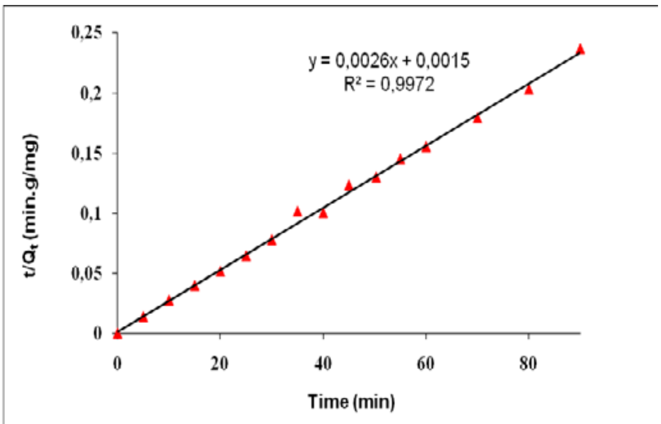

Figure 7: Pseudo-second order kinetic model of NR adsorbed onto incinerated sludge as a function of contact time for [NR] =50 mg/l, m/v=1g/L, pH=5 and T=298K.

System	Experimental result	Pseudo-second order model			Pseudo-first order model		
NR / Incinerated paper sludge suspension	Q_{exp} (mg/g)	Q_{max} (mg/g)	K_2 (g/mg,mn)	R^2 (%)	Q_e (mg/g)	K_1 (min^{-1})	R^2 (%)
	401. 936	384.615	0.0169	0.999	67.050	0.0700	0.899

R^2: Regression coefficient

Table 3: Kinetic parameters of NR dye adsorbed onto incinerated paper sludge for [NR] =50mg/L, [Incinerated sludge] =1g/L, pH =5, T=298K and contact time =80min.

where Q_e and Q_t (both in mg/g) are the amount of dye adsorbed per unit weight of adsorbent at equilibrium and at any time *t*, respectively and K_1 (L/min) is the rate constant for adsorption of dye. At given boundary conditions for t=0, Q_t=0, the equation Equation (3) can be integrated to give Equation (4):

$$\log_{10}(Q_e - Q_t) = \log_{10}(Q_e) - \frac{K_1}{2.303}t \tag{4}$$

The values of k_1 were calculated from the slopes of the respective linear plots of log (Q_e – Q_t) versus t. The regression coefficients, R^2, (given in Table 3) for the pseudo-first–order model did not exceed the values of 0.90. The calculated Q_e value obtained from pseudo-first-order kinetic model was much different compared with experimental Q_{exp} values. These results suggest that the process does not follow the pseudo-first-order adsorption rate equation of Lagergren.

The sorption kinetic following pseudo-second order model given by Ho [27] is represented in the form Equation (5):

$$\frac{dQ_t}{dt} K_2(Q_e - Q)^2 \tag{5}$$

where Q and Q_e represent the amount of dye adsorbed (mg/g) at any time t; K_2 is the rate of sorption (g/mg.min) and Q_e the amount of dye adsorbed onto incinerated sludge at equilibrium (mg/g). Separating Equation (6), gives:

$$\frac{dQ}{d(Q_e - Q)^2} K_2 dt \tag{6}$$

Integrating Equation (5) with respect to the boundary conditions Q = 0 at *t* = 0 and Q = Q at *t* = *t*, the linearised form of pseudo second

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order expression can be obtained as Equation (7):

$$\frac{1}{(Q_e - Q)} = \frac{1}{Q_e} + K_2 t \tag{7}$$

Equation (7) can be further linearised to Equation (8):

$$\frac{t}{Q_t} = \frac{1}{K_2 Qe^2} + \frac{t}{K_2} \tag{8}$$

The linearity of the plots of t/Q_t versus t for adsorption of NR on incinerated sludge (Figure 7 and Table 3) suggests that all the adsorption data were satisfactorily described by pseudo-second order model (R^2=0.999), based on the assumption that the rate–limiting step may be chemisorptions involving valency forces through sharing or exchange of electrons between the hydrophilic edge sites of incinerated sludge and polar dye ions [28]. The calculated Q_e value obtained from pseudo-second-order kinetic model was close to the experimental Q_{exp} value.

Adsorption isotherm

Figure 8 shows the adsorption isotherms of the dyes using incinerated sludge. The shape of the NR isotherm does not show the characteristic plateau of a monolayer in the range of the concentration used. This isotherm can be classified as an (S-shape) isotherm according

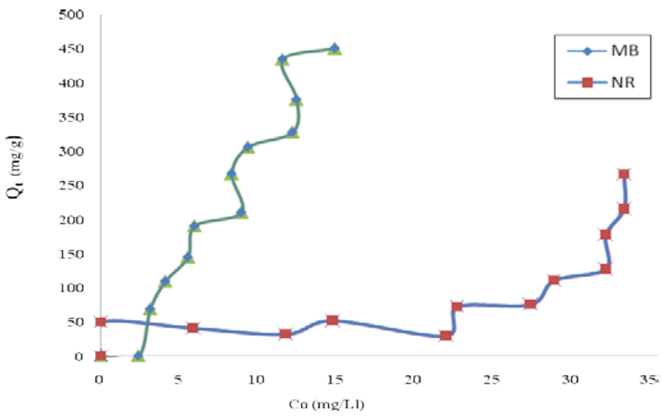

Figure 8: Adsorption isotherms of NR and MB dyes onto incinerated sludge for [NR] =5-60 mg/L, pH=5 (NR) and 7 (MB), m/v=1g/L, T=298K and contact time 80 min.

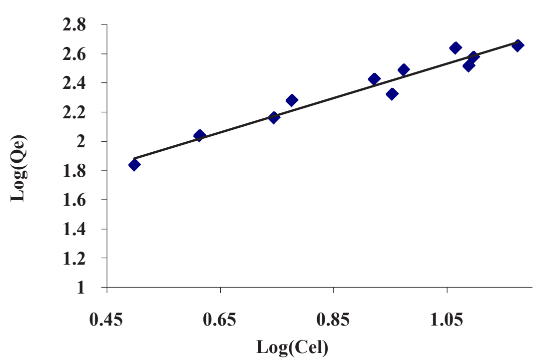

Figure 9: Freundlich isotherm of NR onto incinerated sludge for [NR] =5-60 mg/L, pH=5, m/v=1g/L, T=298K and contact time 80 min.

System	Experimental Results	Langmuir model $C_e/Q_e = 1/(K_L Q_{max}) + C_e/Q_{max}$			Freundlich model $lnQ_e = lnK_F + (1/n) lnC_e$		
	Q_{exp}	Q_{max}	K_L	R^2	K_F	1/n	R^2
NR / Incinerated paper sludge suspension	(mg/g)	(mg/g)	(L/g)	(%)	(L/g)		(%)
	374.982	-344.827	-0.0451	35.74	23,4693	1.3746	95.17

R^2: Regression coefficient

Table 4: Adsorption isotherm parameters of NR dye adsorbed onto incinerated sludge for [NR] =5-60mg/L, [Incinerated sludge]=1g/L, pH =5, T=298K and contact time =80min.

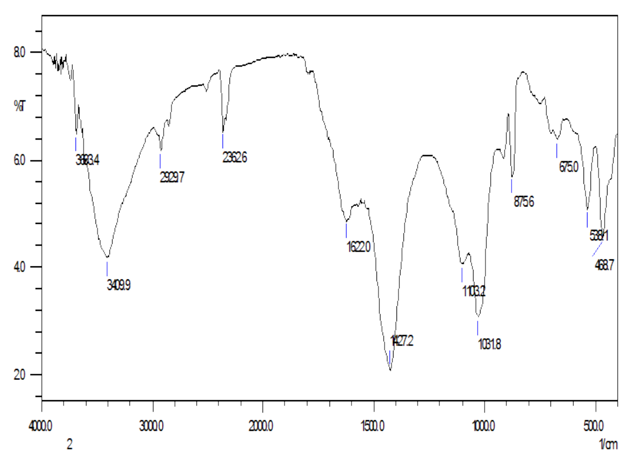

Figure 10: FTRI spectra of samples: (a) pure incinerated sludge (b) and NR / incinerated sludge.

to Giles classification system [29]. The equilibrium adsorption capacity increased more slowly with the increase in initial dye concentration and the maximum adsorption of 374.98 mg/g was obtained for 50 mg/L of NR.

Langmuir [30] and Freundlich [31] isotherms were applied to assess the performance of the adsorption process of NR onto incinerated paper sludge. The linearized equation Equation (9) of Langmuir is given as follows:

$$\frac{Ce}{Qe} = \frac{1}{K_2 Q_{max}} + \frac{Ce}{Q_{max}} \tag{9}$$

where Q_{max} (mg/g) is the maximum amount of the dye per unit weight of incinerated sludge to form a complete monolayer coverage on the surface bound at high equilibrium dye concentration C_e (mg/L) and K_L (L/g) is the Langmuir constant related to the affinity of binding sites. A plot of C_e/Q_e versus C_e leads to a straight line with the slope of $1/Q_{max}$ and an intercept of $1/Q_{max}K_L$.

The logarithmic form of Freundlich equation Equation (10) is expressed as follows:

$$LnQe = LnK_F + \frac{1}{n} LnCe \tag{10}$$

where Q_e (mg/g) is roughly an indicator of the adsorption capacity and (1/n) of the adsorption intensity. Values n > 1 represent a favorable adsorption condition. 1/n and K_F (L/g) can be determined from the

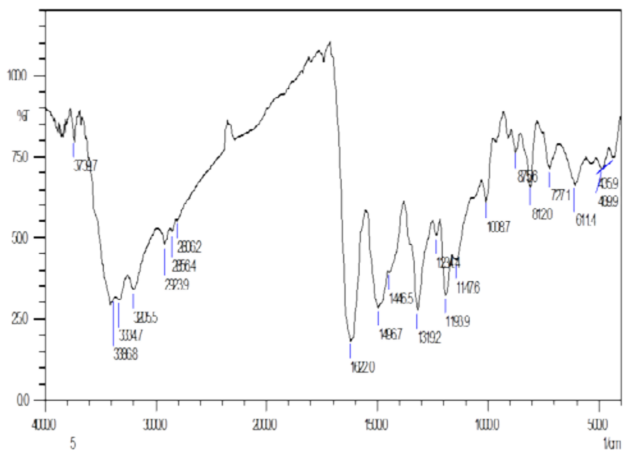

Figure 11: FTIR spectrum of the pure NR dye.

linear plot of $\ln Q_e$ versus $\ln C_e$ (Figure 9). Langmuir and Freundlich isotherms parameters for the adsorption of NR on incinerated sludge were computed in Table 4.

Freundlich model was fitted to the adsorption data with highly significant coefficients of regression ($R^2 > 0.95$). The value of $1/n$ ($1/n > 1$) reveals the nature and strength of adsorptive forces involved indicating the existence of strong adsorption forces operating on heterogeneous surface incinerated sludge.

FTIR analysis

In order to investigate the interactions between NR and incinerated sludge, FTIR analysis was conducted. The FTIR spectra of the incinerated sludge untreated and treated with NR (Figure 10) show a great similarity of some absorption bands with some differences in the intensity. The assignment and interpretation of the bands are the following: a less intense band at 2855 cm^{-1}, assigned to symmetrical aliphatic carbon ($-CH_2$) and a second weak band near 435 cm^{-1} (no identified) corresponding to the NR dye (Figure 11). These results indicated a specific interaction between NR dye molecules and the incinerated sludge surface [32].

Conclusion

Adsorption of neutral red (NR) from aqueous solution using an incinerated paper sludge was investigated using UV visible and FTIR spectrophotometers. Results show that the adsorption efficiency of NR on incinerated sludge was significantly affected by the pH medium. The adsorption process of NR^+H on incinerated sludge under acidic media was accompanied by a blue shift of the main absorption bands. The highest shift was observed in pH range of 4.26-6.04. However, under alkali conditions (pH=8.15-12.1) the adsorption profile of NR has a nearly constant main bands. The uptake increased more slowly with increase in initial dye concentration. The adsorption reaction was perfectly described by pseudo-second-order kinetic model. Incinerated sludge exhibited excellent performance for adsorption of NR with a maximum of 374. 98 mg/g. The adsorption data were well fitted with Freundlich adsorptive model. FTIR analysis revealed the strong interaction forces operating on heterogeneous surface of the incinerated sludge between the dissociation of the oxygenated groupings, which are in general acid functional, and dimethylamine goup [$-N+(CH_3)_2$] of the NR dye which could be used to explain the high adsorption capacity of cationic dye onto incinerated sludge.

Acknowledgments

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