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Adsorption of Reactive Blue 222 onto an industrial solid waste included Al(III) hydroxide: pH, ionic strength, isotherms, and kinetics studies

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ABSTRACT

In this study, an industrial waste sludge composed of metal hydroxides was used as a low-cost adsorbent for removing a reactive textile dye. Removal of the reactive dye Reactive Blue 222 (RB 222) onto an industrial solid waste included Al(III) hydroxides was studied using various parameters such as initial dye concentration, pH, electrolyte concentration, and agitation time. The maximum dye removal efficiency was obtained without pH adjustment in the study. The amount of dye adsorbed increased with increase in agitation time and it achieved to equilibrium at 90 min. The amount of dye adsorbed per unit weight of adsorbent values reduced in the dye solution with NaCl and Na_2SO_4. Adsorption equilibrium studies were employed to determine the adsorption capacity of the adsorbents by using Langmuir and Freundlich isotherm models. Freundlich model yielded better fit than the Langmuir model for the adsorption of RB 222 on waste metal hydroxide. The Langmuir adsorption capacity was found to be 18.38 mg dye/g adsorbent. Adsorption kinetics obeyed a second-order kinetic model.

Keywords: Adsorption; Metal hydroxide; Color removal; Reactive Blue 222

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1. Introduction

Dyes and pigments used by various industries produce colored wastewaters which affects the aquatic flora and fauna. The most sources of colored wastewaters are industries which use dyes in their production processes. Large amounts of highly colored wastewater are discharged from textile, printing, paper, and leather industries which use many kinds of artificial composite dyes [1]. Especially textile industry uses large amounts of water and generates high volumes of colored wastewaters. Textile dyeing effluents are composed of complex mixtures of dyes and auxiliary chemicals, salts, acids, bases, organochlorinated compounds, and heavy metals [2].

Colored wastewaters discharged to receiving water bodies cause esthetic problems and may cause toxic effects to aquatic life [3–5]. Most of the dyes have complex molecular structures and large sizes. Because of this dyes are considered non-oxidizable by conventional treatment methods [6]. Reactive dyes are the major cause for complaint. Reactive dye bath and

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first rinse represent 6 to 30 L/kg of effluent and most of the color and salt. The total water consumption for reactive dyeing ranges from 25 to 100 L/kg fabric, while the water consumption from total textile finishing ranges from 30 to 150 L/kg fabric [7].

Due to dye resistance to biodegradability, light, heat, and oxidizing agents, color removal is one of the main problems in the treatment of such these wastewaters [2]. Most dyes are considered to be non-oxidizable substances by conventional biological and physical treatment because of their complex structure and large molecular size [8]. So, traditional treatments such as biological and coagulation/flocculation methods are generally ineffective for total color removal. Because of these difficulties, there have been several investigations for treatment of colored wastewaters [1,2,9]. Various methods have been studied such as adsorption on organic and inorganic matrices, photo catalysis, chemical oxidation, microbiological, or enzymatic decomposition, etc. [9–15].

Adsorption has several advantages and this technique is one of the most powerful tool for the removal of pollutants from the water [16]. If the adsorbent is inexpensive and readily available adsorption technique will be considered best to removal of toxic substances and more promising that other techniques [17,18]. Adsorption is a simple and cheap technology for removal of dyes. Especially if low-cost waste materials such as bagasse fly ash, hen feather, waste metal hydroxide, coconut, eggshell, rice husk etc. were used adsorption technique may be very advantageous [19–24]. As it is known activated carbon is the most powerful and common adsorbent and has been used successfully. But, the high cost in the preparation of activated carbon restricts its use in the industrial wastewater treatment, especially in the developing countries [1]. Especially the use of agro/industrial waste residues for the adsorption of dyes has also been highlighted as an effective and cheap alternative for dye removal [4,12,16].

In this study, the usage of a waste sludge was evaluated for the adsorption of a reactive dye. This waste sludge was generated by metal precipitation in the effluents of electroplating industries. Most of these media have surface areas over 100 m^2/g and have a continuum of micro- and macro-pores [25]. In the study, the effectiveness of waste metal hydroxide sludge to remove a reactive dye Reactive Blue 222 (RB 222) was investigated. The effect of pH, initial dye concentration, and contact time on the adsorption of RB 222 onto waste metal hydroxide sludge (WMH) were investigated using batch experiments. The study also discusses adsorption isotherms and kinetics of RB 222 adsorption on WMH.

2. Experimental

2.1. Materials

2.1.1. Adsorbent preparation

In the study, the waste metal hydroxide (WMH) was obtained from aluminum electroplating industry in Turkey. WMH was generated by alkaline precipitation of the metal ions present in wastewater. Firstly, the sludge was dried at room temperature, ground, and sieved. Then, the sludge washed with distilled water several times. The powder of particle size smaller than 500 μm was used as the adsorbent for the removal of RB 222 from aqueous solution. The chemical composition of the WMH was measured using ICP spectroscopy. This WMH contained 45.36% Al, 0.31% Mg, 0.26% Na, 0.19% Fe, 0.0032% Cr, 0.002% Cu, 0.0034% Zn, and 0.0004% Mn. In the BET procedure, surface area of the WMH is found as 130 m^2/g (Quantachrome Instruments Nova 4000E).

2.1.2. Aqueous dye solutions preparation

RB 222 is a dye broadly used in the textile industry. The dye was obtained from GOTEK Chemical Company in Turkey. The molecular structure of this dye is shown in Fig. 1. The aqueous solution of RB 222 was prepared by dissolving the desired amount of the dye in distilled water. A stock solution of 300 mg/L was prepared at pH 7. Solutions of the required concentrations were prepared by successive dilution of this stock solution.

2.2. Method

2.2.1. Adsorption studies

The pH where the net total particle charge is zero is called point of zero charge pH (pH_{pzc}). The pH_{pzc} was estimated by the mass titration method [2]. Fifty

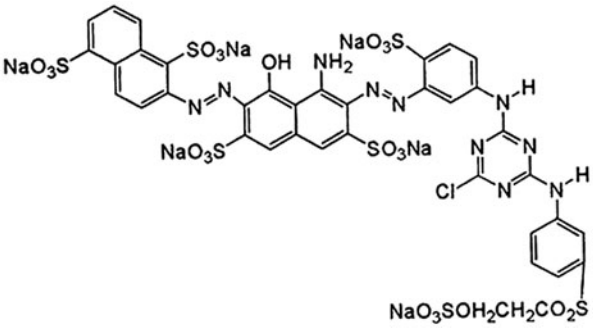

Fig. 1. Molecular structure of RB 222.

milliliter of 0.01 mol/L NaCl aqueous solutions at different initial pH values were stirred with 0.150 g of WMH in closed Erlenmeyer flasks for two days at room temperature. Blank tests were done to take into account the possible effect of CO_2 from air. Equilibrium pH values were then measured and pH_{pzc} determined as the initial pH that equals final pH.

The effects of electrolytes of NaCl and Na_2SO_4 in the dye solution were also studied. The concentrations of both electrolytes were 0.5 M.

The adsorption studies were carried out at different dye concentrations (100, 150, 200, and 300 mg/L) to investigate the adsorptive capacity of the adsorbent (WMH). The experiments were carried out at fixed adsorbent dose of 5 g in 300 mL (16.6 g/L, which has the optimum removal rate in our study) in the aqueous solution at 25˚C temperature. The samples were taken at the end of the desired contact time to determine the effect of shaking time. A control flask with only the adsorbent in 300 mL of deionized water was used simultaneously under the same conditions. The flasks were agitated at room temperature (25˚C) at 250 rpm (SL350 Nüve).

The effect of solution pH was studied by performing the adsorption experiments at four different pH levels; pH: 3 ± 0.1, pH: 5.0 ± 0.1 (natural pH), pH: 7.0 ± 0.1, and pH: 9.0 ± 0.1. The pH of the solution was adjusted with 1 N HCl or NaOH solution by using a WTW340 pH meter. After 90 min contact time, the dye concentration was determined. The effects of electrolytes of NaCl and Na_2SO_4 in the dye solution were also studied. The concentrations of both electrolytes were 0.5 M.

The dye solution was separated from the adsorbent by centrifugation (CN180 Nüve fuge) at 3,500 rpm for 5 min. The dye concentration were measured at a wavelength corresponding to the maximum absorbance (μ_{max} of 612 nm for RB 222) using a thermospectronic AQUAMATE spectrometer.

One of the most important factors in evaluating the efficiency of sorption is analyzing the rate of the sorption. So we conducted experiments to assess the kinetics of RB 222 removal on WMH. In addition, adsorption equilibrium studies were employed to determine the adsorption capacity of the adsorbents by using Langmuir and Freundlich isotherm models.

2.2.2. Desorption studies

The mechanism of adsorption was evaluated by desorption studies. Desorption experiments were carried out between pH 2.5–11.5 conditions with WMH loaded with 16.6 mg/g of RB 222. This WMH was shaken with distilled water (0.20 g/0.05 L) at initial pH of 2.5–11.5. After a contact time of 24 h [2], the percentage of the dye adsorbed from the sludge was determined.

3. Results and discussion

3.1. Effect of pH, contact time, and initial dye concentrations

pH_{pzc} is an important parameter to explain adsorption of anions and cations under different pHs and to find optimum pH for a pollutant. If the pH of a adsorbent is above its point of zero charge the adsorbent surface will have a net negative charge and predominantly exhibit an ability to exchange cations (exchange of one positive ion by another), while the adsorbent will mainly retain anions (electrostatically) if its pH is below its PZC (exchange of one negative ion for another) [2,26]. Fig. 2 shows the results obtained in the determination of pH_{pzc} of the waste sludge. When initial pH value was higher than 4.9 ± 0.1 the final pH decreases and when initial pH value was lower than 4.9 ± 0.1 the final pH increases. This is because of amphoteric behavior of WMH [2]. pH_{pzc} was estimated as 4.9 ± 0.1.

The adsorption process of dyes by different adsorbents was affected by various parameters such as pH, contact time, and initial concentration of dye solution. The adsorption of RB 222 on WMH was conducted at different pH values. Fig. 3 shows the amount of dyes adsorbed on the adsorbent at equilibrium (mg dye /g adsorbent) as a function of pH values. As it is shown from the Fig. 3 that q_e values decreased from 16.60 to 6.16 mg/g when the pH was increased from natural pH to pH 9. It is reported in some studies that metal hydroxides are amphoteric and the lower capacity in adsorption may be due to the solubility of metal hydroxides in strong acid or base [27]. The dissociation of the metal hydroxide complexes cause the surface to become positively charged and the metal hydroxide sludge can be easily applied in a wide range of initial pH values, without pH adjustment [7,27]. As reported in those studies that the maximum dye removal efficiency is obtained without pH adjustment in this study.

The removal efficiency of RB 222 by WMH at different contact times and initial dye concentrations was studied (results not shown). The amount of dye adsorbed increased with increase in contact time and it achieves an equilibrium at 90 min. Generally, dye removal is highly affected by initial dye concentration. Raising the initial RB 222 concentration from 100 to 300 mg/L, the removal efficiency decreased slightly from 99 to 90%. The initial dye concentration provides

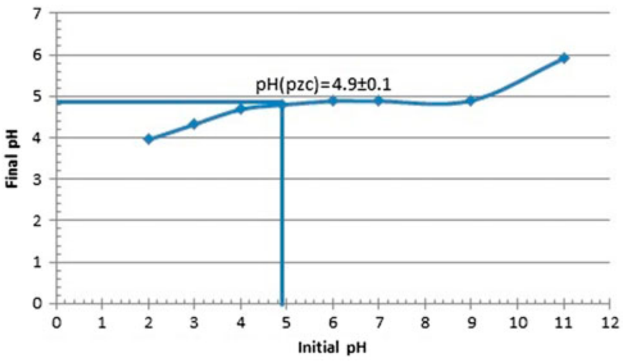

Fig. 2. Experimental determination of $pH_{pzc.}$

the driving force to overcome the resistance to the mass transfer of dye between aqueous and solid phase. At lower initial concentrations, dyes in solution interact with the binding sites and thus higher adsorption is obtained. So, percent color removal decreased with increasing dye concentrations because of nearly complete coverage of the binding sites at high dye concentrations [28,29].

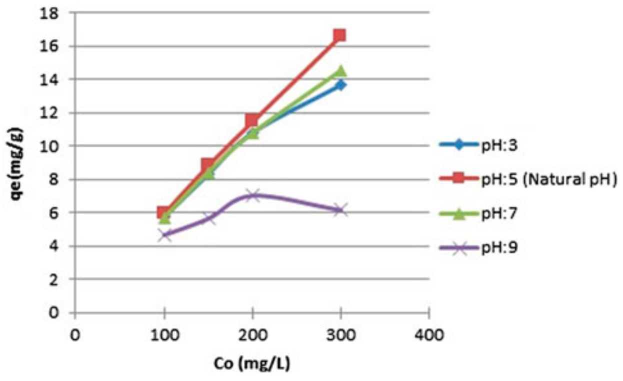

Fig. 3. RB 222 amount adsorbed on WMH as a function of pH values.

3.2. *Effect of electrolytes on the adsorption capacity*

In the various industries, especially in textile industry when dying of fabric with reactive dyes large amounts of salts are generally employed [10]. So the effect of various salt concentrations on the adsorption of dyes should be investigated in order to optimize adsorption studies. Fig. 4 shows the amount of dye adsorbed per unit weight of adsorbent of RB 222 adsorption on WMH with NaCl, Na_2SO_4, and without electrolytes. As it is shown from Fig. 5 that the dye solution with NaCl and Na_2SO_4, the amount of dye adsorbed per unit weight of adsorbent values decreased. It was seen that the adsorption amount of dye decreased from 5.95 to 4.99 and to 3.31 mg/g for without salt, with NaCl, and with Na_2SO_4 for the initial dye concentration of 100 mg/L and in natural pH. This could be explained by competition between SO_3^- ions on dye and Cl^- and SO_4^{2-} ions of salts for the available adsorption sites. As SO_4^{2-} is more negative than Cl^-, adsorption efficiencies containing equal molar NaCl and Na_2SO_4 is not equal and adsorption efficiency with Na_2SO_4 is lower [10].

3.3. *Adsorption equilibrium study*

Equilibrium data are basic requirements for design of adsorption. Common isotherm models such as Freundlich and Langmuir have been used to describe the equilibrium characteristics of adsorption [6,30]. In this study, the most widely used isotherms equations, Langmuir and Freundlich adsorption isotherms were employed.

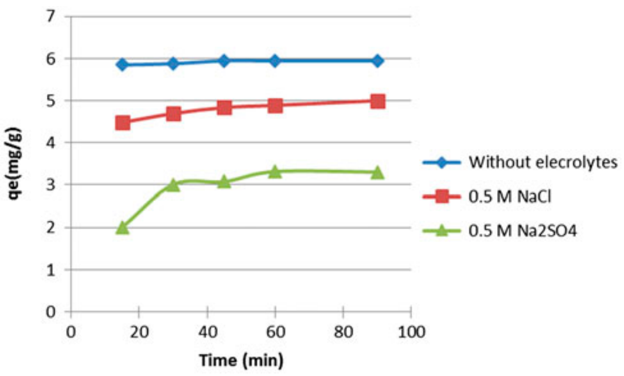

Fig. 4. The amount of dye adsorbed per unit weight of adsorbent of RB 222 adsorption on WMH with NaCl, Na_2SO_4, and without electrolytes (C_0 = 100 mg/L, pH: 5.0 ± 0.1 (natural pH).

The Langmuir equation is used to estimate the maximum adsorption capacity corresponding to complete monolayer coverage on the adsorbent surface. The linearized equation of Langmuir is represented according to Eq. (1) [31]:

$$\frac{C_e}{q_e} = \frac{1}{q_{\max} K_L} + \frac{C_e}{q_{\max}} \tag{1}$$

where q_e (mg/g) is the amount of RB 222 adsorbed per unit mass of adsorbent particles at equilibrium and C_e (mg/L) is the equilibrium liquid concentration of RB 222, K_L is the equilibrium constant (L/mg) and $q_{\max}$ is the amount of adsorbate required to form monolayer (mg/g) [6].

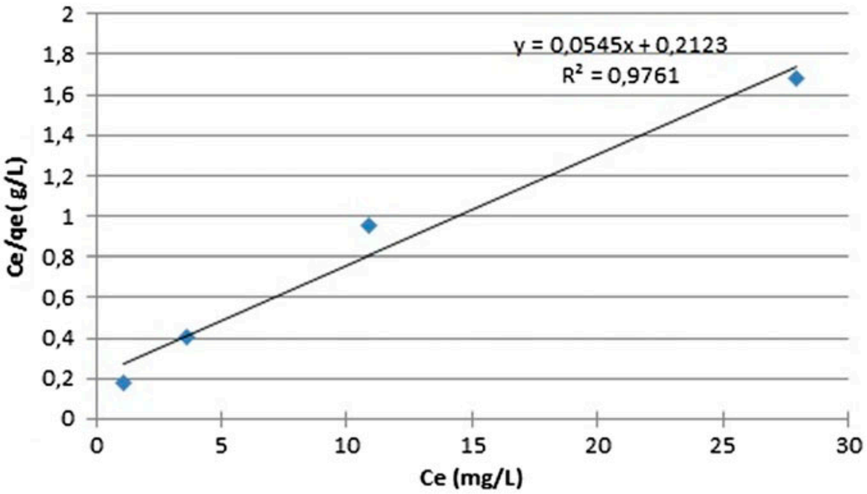

Fig. 5. Langmuir isotherm plots for the adsorption of RB 222 on WMH (pH: 5.0 ± 0.1 (natural pH), t = 90 min, C_0 = 100, 150, 200, and 300 mg/L).

The experimental data are then fitted into the above equation for linearization by plotting C_e/q_e against C_e. From the data of C_e/q_e against C_e, q_{max} and K_L values can be determined. The characteristics of the Langmuir isotherm could be expressed by a separation factor, R_L which is defined as Eq. (2) [6]:

$$R_L = \frac{1}{(1 + K_L C_0)} \tag{2}$$

R_L values indicate the type of isotherm. Favorable adsorption is indicated by $0 < R_L < 1$ [4,29].

Evaluation of Freundlich adsorption isotherm is also important step which provides approximate estimation of sorption capacity of the adsorbent. The Freundlich model is an empirical equation used to estimate the adsorption intensity of the sorbent toward the adsorbate. The Freundlich model and the linearized equation of Freundlich are given according to Eq. (3) [32]:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \tag{3}$$

If a plot of $\ln C_e$ against $\ln q_e$ yields a straight line it indicates the adaptation of the Freundlich model. The value of n indicates the affinity of the adsorbate toward the adsorbent. $1/n$ and K_f can be calculated from the slope and intercept, respectively [6,32].

Langmuir and Freundlich isotherm plots are presented in Figs. 5 and 6 for the adsorption of RB 222 on WMH. According the calculations, the q_{max} and K_L values for Langmuir isotherm are found as 18.38 mg/g and 0.257 L/mg, respectively. K_f and $1/n$ values for the Freundlich isotherm are found as 5.83 and 0.305, respectively. The R^2 values show that Freundlich model yields better fit than the Langmuir model for the adsorption of RB 222 on WMH in this study. K_f and $1/n$ values in Freundlich isotherm model indicate the adsorption capacity and adsorption intensity, respectively. The higher the value of $1/n$, the higher will be the affinity and the heterogeneity of the adsorbent sites. Values of $1/n$ less than 1 show the favorable nature of adsorption of RB 222 on the adsorbents [30]. $1/n$ values less than 1 showed that RB 222 adsorption on WMH is in a favorable nature.

3.4. Adsorption kinetic study

In the kinetics experiments, the adsorption reached equilibrium after 90 min and no significant increase in RB 222 adsorption was observed after that time. Kinetics of RB 222 adsorption on the WMH were analyzed using pseudo-first-order and pseudo-second-order models.

The Lagergren equation has been one of the most used equations for pseudo-first-order kinetics and the integrated of the Lagergren equation for the boundary conditions of t = 0, q_t = 0, and t = t, q_t = q_t, is given according to Eq. (4) is expressed by the equation [33]:

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \tag{4}$$

k_1 is the pseudo-first-order adsorption rate coefficient (min^{-1}), q_e is the amount adsorbed (mg/g) at equilibrium, and q_t is the amount adsorbed (mg/g) at time t.

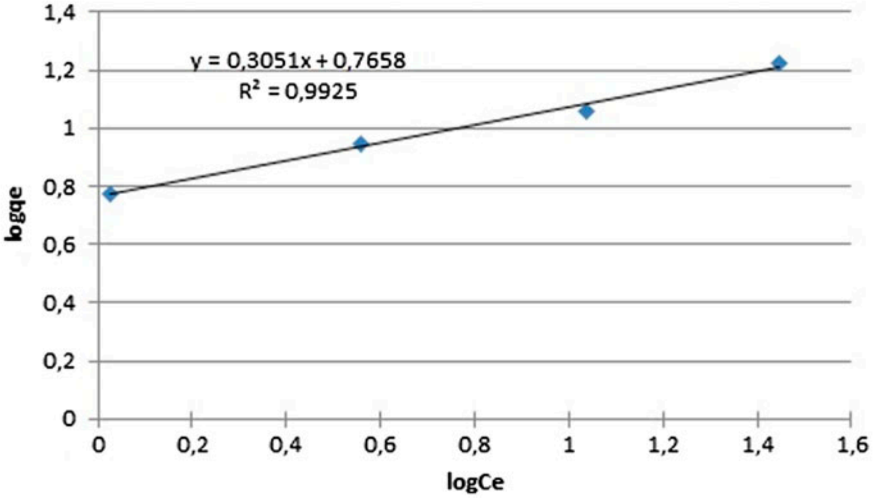

Fig. 6. Freundlich isotherm plots for the adsorption of RB 222 on WMH (pH: 5.0 ± 0.1 (natural pH), t = 90 min, C_0 = 100, 150, 200, and 300 mg/L).

The straight line of the plot of $\log(q_e - q_t)$ vs. t show the applicability of the pseudo-first-order equation for the system. The values of $q_{e,\text{calculated}}$ and k_1 can be determined from the slope and intercept of the plots.

The pseudo-second-order adsorption kinetic rate equation and integrated form of the equation for boundary conditions $t = 0$ to $t = t$ and $q_t = 0$ to $q_t = q_t$ is expressed by the Eq. (6). h (mg/g.min) is the initial adsorption rate in the Eq. (5) below [6]:

$$\frac{t}{q_t} = \frac{1}{h} + \frac{1}{q_e}t \tag{5}$$

Table 1 shows the adsorption kinetics of RB 222 on WMH. The experimental q_e values and R^2 of the pseudo-first-order equation show that adsorption of RB 222 on WMH is not a first-order reaction. As it is seen form the Table 1 that experimental q_e values agree with the calculated ones and the correlation coefficients R^2 of the model for the linear plots are very close to 1 at various concentrations. So, the pseudo-second-order kinetic model yields the best fit and the adsorption rates of RB 222 on WMH fit to pseudo-second-order kinetic model well (Fig. 7). These results suggest that the rate of sorption for WMH is dependent on the availability of the sorption sites rather than the concentration of the sorbate in the bulk solution [34]. The rate coefficient, k_2 decreases with the increasing initial adsorbate concentration as a rule, where k_2 is interpreted as a time-scaling factor. Thus, higher is the initial concentration of adsorbate, the longer time is required to reach an equilibrium, in turn, the k_2 value decreases [35]. In this study, it is found that k_2 values decreases with increasing initial concentration for adsorption of RB 222 on WMH.

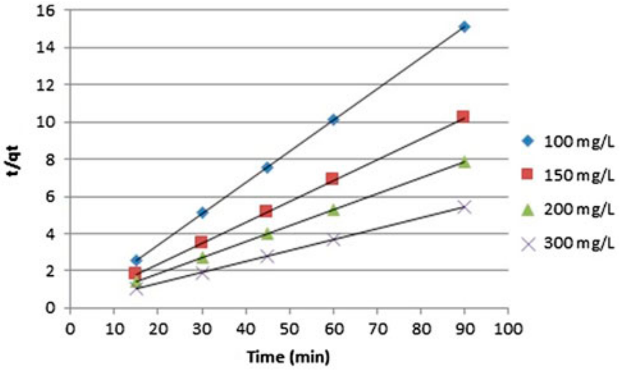

Fig. 7. Plot of pseudo-second-order equation for adsorption of RB 222 on WMH at different initial concentrations (natural pH, t = 90 min, C_0 = 100, 150, 200, 300 mg/L, m = 16.6 g/L).

3.5. Desorption studies

Desorption studies help to explain the adsorption mechanism and necessary in order to regenerate the adsorbent and recovery the adsorbed compounds [4]. It was found that the percent desorption was slightly low (<1.2%) in the pH range 2.5–11.5 for the waste sludge loaded with 16.6 mg/g RB 222 dye. Near the pH_{pzc} which about pH 4.9, the least desorption which could be accepted as no desorption was occurred. It could be explained by the low dissolution and low deterioration of the structure of WMH on the pH_{pzc}.

Table 1

Kinetic parameters for the removal of RB 222 by WMH (t = 90 min, C_0 = 100, 150, 200, 300 mg/L, m = 16.6 g/L, pH: 5 ± 0.1, natural pH)

Pseudo-first-order model

C_0 (mg/L)	$q_{e,exp}$ (mg/g)	$q_{e,\ calc}$ (mg/g)	k_1 (1/min)	R^2
100	5.947	0.927	0.159	0.761
150	8.819	0.514	0.028	0.851
200	11.455	1.008	0.038	0.902
300	16.602	3.81	0.049	0.999

Pseudo-second-order model

C_0 (mg/L)	$q_{e,calc}$ (mg/g)	h (g/mg·min)	k_2 (g/mg·min)	R^2
100	5.974	17.98	0.504	0.999
150	8.913	8.71	0.109	0.999
200	11.614	9.32	0.069	0.999
300	17.035	7.30	0.025	0.999

Low desorption suggests that chemisorptions might be the major mechanism of dye removal by the adsorbent.

4. Conclusion

The influence of pH and electrolytes on the removal of RB 222 in aqueous solution by metal hydroxide sludge was evaluated. The results indicated that waste metal hydroxide sludge could be used as low-cost adsorbent to remove RB 222 from aqueous solution. Equilibrium data were well described by both Langmuir and Freundlich models. The maximum adsorption capacity was determined as 18.38 mg dye/g adsorbent at natural pH without pH adjustment. The adsorption capacity of metal hydroxide sludge was also determined after adding NaCl and Na_2SO_4 salts. With these electrolytes it is found that adsorption capacity was decreased from 5.95 to 4.99 and to 3.31 mg/g for the initial dye concentration of 100 mg/L. Kinetic data were adequately fitted by the pseudo-second-order kinetic model. It is found that k_2 values decreases with increasing initial concentration for adsorption of RB 222 on WMH.

The waste metal hydroxide sludge showed to be an efficient adsorbent for RB 222 without pH adjustment. But its performance was worse when treating dye solutions added electrolytes because of competitive effect between dye anions and Cl^- and SO_4^{2-} ions for the available adsorption sites. So, the presence of these salts must be taken into account in removing reactive dyes by metal hydroxides.

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