



Adsorption of Acid Dyes from Aqueous Solutions by Calcined Alunite and Granular Activated Carbon

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Abstract. Dyestuff production units and dyeing units have always had a pressing need for techniques that allow economical pretreatment for color in the effluent. The effectiveness of adsorption for dye removal from wastewaters had made it an ideal alternative to other expensive treatment options. This paper deals with an investigation on alunite, existing wide reserves in Türkiye and in the world, for dye removal. Calcined alunite was utilized for this study and its performance evaluated against that of granular activated carbon (GAC). The use of calcined alunite for the removal of Acid Blue 40 and Acid Yellow 17 (AB 40 and AY 17) from aqueous solution at different calcination temperature and time, particle size, pH, agitation time and dye concentration has been investigated. The adsorption followed by Langmuir and Freundlich isotherms. The process follows first order adsorption rate expression and the rate constant was found to be 7.65×10^{-2} and $5.74 \times 10^{-2} \text{ min}^{-1}$ for adsorption of AB 40 and AY 17 on calcined alunite, and 8.41×10^{-2} and $10.04 \times 10^{-2} \text{ min}^{-1}$ for adsorption of AB 40 and AY 17 on GAC, respectively. The equilibrium saturation adsorption capacities were 212.8 mg dye/g calcined alunite and 151.5 mg dye/g calcined alunite for AB 40 and AY 17, respectively. The adsorption capacities were found to be 57.47 mg and 133.3 mg dye per g of GAC for AB 40 and AY 17, respectively. The results indicate that, for the removal of acid dye, calcined alunite was most effective adsorbent, although comparable dye removals were exhibited by GAC.

Keywords: acid dye, calcined alunite, adsorption dynamics, isotherm, color removal

1. Introduction

Most of the industries like textile, leather, plastics, paper, food, cosmetics, etc., use dyes and pigments to color their products. The colored wastewaters of these industries are harmful to the aquatic life in rivers and lakes due to reduced light penetration and the presence of highly toxic metal complex dyes. Most of the used dyes are stable to photodegradation, biodegradation and oxidizing agents (Ramakrishna and Viraraghavan, 1997). For this reason, the removal of contaminants from the such colored effluents is require prior to discharge into receiving waters.

Both biological and physical/chemical methods have been employed for color removal from textile efflu-

ents. The former has not been very successful due to the essential non-biodegradable nature of most of the dyes (Juang et al., 1997). The physical/chemical methods that have been proven to be successful are adsorption, chemical oxidation, froth flotation, coagulation/flocculation, membrane filtration and electrochemical treatment agents (Poots et al., 1978; Ramakrishna and Viraraghavan, 1997). Of these methods, adsorption has been found to be an efficient and economically cheap process for removing dyes using various adsorbents (Asfour et al., 1985; Desai et al., 1997).

Alunite is one of the minerals of the jarosite group. Mineral of the alunite has the formula $\text{KAl}_3(\text{SO}_4)_2(\text{OH})_6$. Alunite formed when volcanic roks changed hydrothermally, it occurs with SiO_2 mineral. For this reason, there is about 10–50% SiO_2 in

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alunite composition (Özacar and Şengil, 1999). Alunite gives thermal decomposition reaction products such as Al_2O_3 , $\text{Al}_2(\text{SO}_4)_3$ and K_2SO_4 when it is calcined at 973–1023 K. Alunite exists in substantial deposits in Giresun-Şebinkarahisar, Kütahya-Şaphane and İzmir-Foça in Türkiye. The deposits in Türkiye are estimated to be 37 million tonnes (Şengil, 1995). Alunite can be adsorbent properties, after a suitable process. Alunite-type layered compounds are useful as adsorbents for removing color components from liquids (Şengil et al., 1995; Özacar et al., 2000).

The present study is aimed at studying the adsorption capacity of locally available alunite from Kütahya-Şaphane, Türkiye for dye removal from aqueous solutions, and its performance was evaluated against granular activated carbon (GAC). Batch study was conducted on a laboratory scale using synthetic dye wastewaters made up from commercial grade two acidic dyes. The scope included calcination temperature and time of alunite, particle size, pH, contact time and isotherm studies for all dye adsorbent combinations. The Langmuir and Freundlich isotherm models were tested for their applicability.

2. Materials and Methods

The alunite ore was obtained from the stocks of Dostel Aluminium Sulphate Ltd in Kütahya-Şaphane, Türkiye. The analysis of alunite ore has been carried out by chemical methods and its composition is given in Table 1. The GAC used in this study was Filtrasorb 400 supplied by Chemviron Carbon, UK. Two dyes were used as adsorbates. The dyes used in this study were Acid Blue 40 (AB 40, CI 62125) and Acid Yellow 17 (AY 17, CI 10310). These dyes are commercial grade and were used without further purification. All other chemicals used in the studies were obtained from Merck Chemical Co.

Alunite was prepared by grinding it in a laboratory type ball-mill. The fractions finer than 100 mesh were used in these studies. The alunite samples were calcined in muffle furnace at the temperature from 373 to 1073 K for 15–120 min.

Table 1. The chemical composition of original alunite ore in wt%.

Al_2O_3	SiO_2	SO_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	H_2O
20.85	48.19	17.91	0.07	0.17	0.17	0.10	4.59	0.06	7.89

The dye solutions were prepared by dissolving the different dyes in distilled deionized water at concentrations of 75 mg/L. To determine the suitable calcination temperature and time of alunite, batch adsorption studies were conducted by shaking 100 mL of each dye solution with 1 g of the calcined alunite for 60 min at the various calcination temperatures and then at the suitable calcination temperatures for each dyes similar studies were conducted for various calcination times. The dye solutions were shaken in 250 mL glass-stoppered flask on a Nüve SL 350 shaker operating at 200 rpm. Prior to measurement for color, the dye solutions were filtered through a $1.25\ \mu\text{m}$ glass fiber filter. Alunite was calcined at optimum calcination temperature and time, and sieved to different size fractions using ASTM standard sieves. The BET surface areas were determined based on N_2 adsorption isotherms using sorptometer (Porous Materials Inc., Model BET-202A). The specific surface areas of different particle size are given in Table 2. Adsorption studies were also conducted at different size fractions. Batch pH studies were conducted by shaking 100 mL of each dye solution with 1 g of the calcined alunite for 30 min a range of pH values from 0.5 to 10. 1 N HCl and 1 N NaOH were used for pH adjustment. Blanks were run simultaneously, without any adsorbent to determine the impact of pH change on the dye solutions. At the optimum calcination temperature and time, particle size and pH, contact time studies were conducted by shaking 1 g of the calcined alunite adsorbent in 100 mL of each dye solution, for increasing period of time, until no more dye was removed and equilibrium was achieved. Isotherm studies were conducted in 100 mL of each dye solution, having different concentrations of dye varying from 25 to 200 mg/L, for a time period to

Table 2. BET specific surface areas for the different alunite particle size ranges.

Calcination			
Temperature (K)	Time (min)	Size range (μm)	BET specific surface area (m^2/g)
873	30	90–150	42.8
		150–250	42.2
		250–315	39.8
		315–500	31.1
		500–710	26.4
GAC (Filtrisorb 400)			1100*

*Supplied by the manufacturers.

the equilibrium time for that particular adsorbent-dye combination. Blank runs, with only the adsorbent in 100 mL of deionized water; were conducted simultaneously at similar conditions to account for any color leached by the adsorbent and adsorbed by the glass container.

All concentrations were measured at the wavelength corresponding to maximum absorbance, λ_{\max} , which for AB 40 is 614 nm and for AY 17 is 435 nm using spectrophotometer (Shimadzu UV-150-02).

3. Results and Discussion

3.1. Effect of Calcination Temperature and Time

The alunite was calcined at the temperature from 373 to 1073 K for 60 min. The adsorption experiments with these calcined alunites, using dye solutions with a concentration of 75 mg/L, were run. The results obtained is shown in Fig. 1.

As can be seen from Fig. 1, the adsorption of dye on calcined alunite changes with calcination temperature of the alunite and is highest at 873 K calcination for acid dyes. The adsorption of dyes on the calcined alunite is related to solid phase reactions which happen during calcination. The thermal decomposition of alunite ($\text{KAl}_3(\text{SO}_4)_2(\text{OH})_6$) occurs in two main stages (Bayliss and Koch, 1955; Özacar, 1995). The first stage, which completes at 873 K, is the dehydration of the alunite.

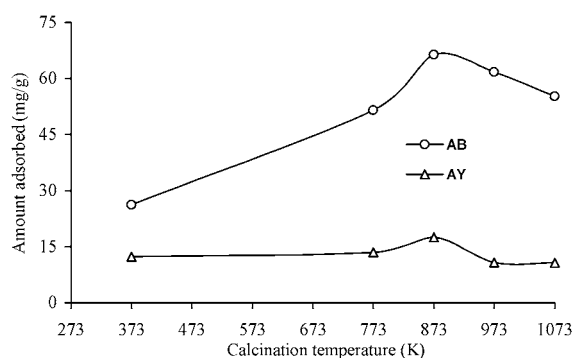
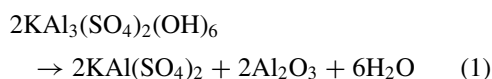


Figure 1. Effect of calcination temperature on adsorption of acid dyes by calcined alunite. Conditions: 75 mg/L concentration, 90–710 μm particle size, 60 min calcination, 1 g/100 mL dose, 30 min agitation, 298 K temperature and pH 6.2.

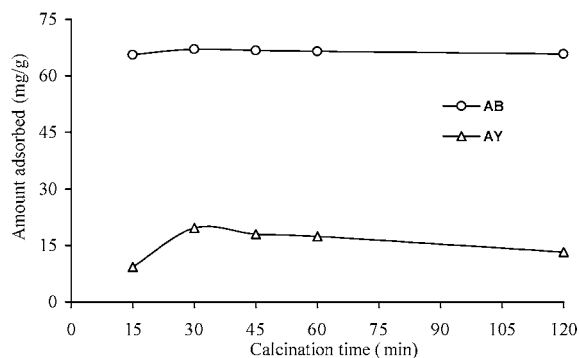
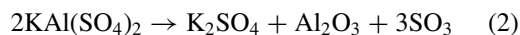


Figure 2. Effect of calcination time on adsorption of acid dyes by calcined alunite. Conditions: 75 mg/L concentration, 90–710 μm particle size, 873 K calcination, 1 g/100 mL dose, 30 min agitation, 298 K temperature and pH 6.2.

The second stage, which begins at about 973 K, is the loss of sulfur trioxide,



The decomposition of aluminum sulphate was completed at 1073 K (Özacar and Şengil, 1999). The maximum adsorption of acid dyes is reached after dehydration of alunite at 873 K calcination.

The alunite was calcined at 873 K for different times. The effect of calcination time on the adsorption of acid dyes from aqueous solutions on calcined alunite is shown in Fig. 2. The optimum calcination time was found to be 30 min at 873 K for adsorption of acid dyes. The calcined amount of alunite depends on calcination time. This time shows difference with respect to chemical composition of alunite. For example, the dehydration time of Pegam alunite is 25–30 min at 793–813 K. The optimum decomposition time of Ping Yang alunite is 10 min at 973 K (Gülensoy and Şengil, 1989).

3.2. Effect of Particle Size

A series of experiments have been carried out with a constant initial dye concentration of 75 mg/L and with various particle sizes of the alunite. Figure 3 shows the experimental results obtained from series of experiments performed, using different alunite particle size ranges. The result shown in Fig. 3 indicate that the saturation capacity of dye adsorption by alunite was increased by decreasing the particle size. This behavior can be attributed to the relationship between the effective specific surface area of the adsorbent particles

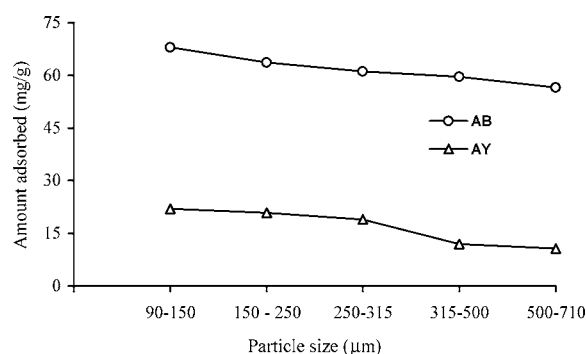


Figure 3. Effect of particle size on adsorption of acid dyes by calcined alunite. Conditions: 75 mg/L concentration, 873 K and 30 min calcination, 1 g/100 mL dose, 30 min agitation, 298 K temperature and pH 6.2.

and their sizes. The effective surface area increased as the particle size decreased and as a consequence, the saturation capacity per unit mass of the adsorbent increased.

As shown in Fig. 3, the adsorption capacity for acid dyes increased with the decrease in the particle diameter. At a particle size range of 90–150 μm the values of saturation capacities are 67.95 and 22.08 mg dye/g alunite for AB 40 and AY 17, respectively. The adsorption of dye from solution with concentration of 75 mg/L have been to increase from 73.4 to 90.6%, and from 14.3 to 29.4% for AB 40 and AY 17, respectively, with decrease in particle size of alunite from 500–710 μm to 90–150 μm at 298 K and pH 6.2.

This can be explained by the fact that for small particles a large external surface area is presented to the dye molecules in the solution which results in a lower driving force per unit surface area for mass transfer than when larger particles are used. Since C_0 is constant and the mass of alunite is constant, the external particle surface area increases as particle size decreases (McKay et al., 1981; El-Geundi, 1991; Annadurai and Krishnan, 1997).

3.3. Effect of pH

Figure 4 shows the comparative removals of the two acidic dyes achieved by calcined alunite and GAC. Ionic dyes upon dissolution release colored dye anions/cations in solution. The adsorption of these charged dye groups onto the adsorbent surface is primarily influenced by the surface charge on the adsorbent which in turn is influenced by the

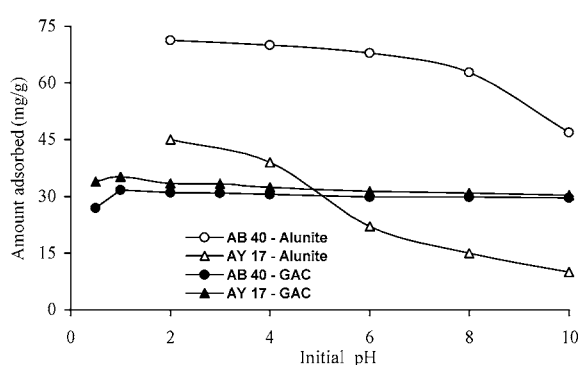
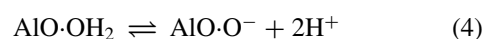
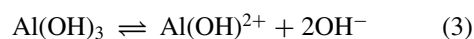


Figure 4. Effect of pH on adsorption of acid dyes by calcined alunite and GAC. Conditions: 75 mg/L concentration, 90–150 μm particle size, 873 K and 30 min calcination, 1 g/100 mL dose, 30 min agitation and 298 K temperature.

solution pH agents (Ramakrishna and Viraraghavan, 1997).

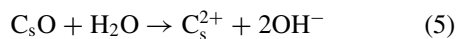
The chief constituents of calcined alunite are metal oxides mainly of Si and Al. These metal oxides form metal-hydroxide complexes in solution and the subsequent acidic or basic dissociation of these complexes at the solid-solution interface leads to development of a positive or negative charge on the surface agents (Ramakrishna and Viraraghavan, 1997). Activated alumina in the alunite are normally contains varying amounts of water molecules especially those which either exist as surface hydroxyl groups or adsorbed water.



In water, these OH^- ions are released from the surface and the cationic centers remaining on the surface would be the source of attraction for anionic dyes (Desai et al., 1997). At acidic pHs, an increasing concentration of the H^+ ion in dye solution, the surface OH^- ions would get neutralized by protonation which facilitates the diffusion of dye molecules in the vicinity of the adsorbent. The maximum removal for AB 40 and AY 17 with calcined alunite was hence observed at pH 2, when the negative charge on the surface is very much reduced due to the excess of protons in solution. For AB 40 and AY 17, the percent removal increased from 62.6 to 95% and from 13.3 to 60%, respectively, with decrease in pH from 10 to 2.

From the experimental results on Filtrasorb 400 chemistry, the reaction on the activated carbon surface

follows the following pathway (Al-Degs et al., 2000):



where C_sO is the surface carbon oxide and C_s^{2+} are the charged carbon particles. The pH for the adsorption solutions (i.e. the pH of the dye solution and carbon particles after equilibrium) was hence increasing the positive charges on the surface of the GAC. This has the effect of attracting more negatively charged functional groups located on the acid dye during the adsorption process. For AB 40 and AY 17, the percent removal increased from 39.5 to 42.2% and from 40.4 to 46.9%, respectively, with decrease in pH from 10 to 1.

3.4. Effect of Contact Time

Adsorption isotherms are usually determined under equilibrium conditions. A series of contact time experiments for two acid dyes have been carried out with a constant initial dye concentration of 75 mg/L, particle size of 90–150 μm , pH 2, 1 g of calcined alunite and temperature 298 K. Figure 5 shows the contact time necessary for AB 40 and AY 17 to reach saturation to be over 90 min. The removals exhibited for each of the dye-adsorbent combinations were compared with the performance of GAC under similar conditions. Amount of GAC exactly equal to the calcined alunite were shaken with similar quantities of dye solutions for times equal to the equilibrium time obtained for each of the dye-calcined alunite combination at pH 1.

The distribution of dye between the adsorbent and the dye solution, when the system is in a state equi-

librium, is important to establish the capacity of the adsorbent for the dyestuff (McKay et al., 1985).

Calcined alunite exhibited equilibrium times of 90 min for AB 40 and AY 17 with dye removals between 96.6% and 72.3%. Similarly GAC showed removal efficiencies of change of between 44.3% and 48.4% at equilibrium times of 90 for AB 40 and AY 17, respectively. Such short times coupled with high removals indicate a high degree of affinity for the dye groups towards chemisorption. The initial concentration of dye has little influence of the time of contact necessary to reach equilibrium (Poots et al., 1978; Khare et al., 1987; Gupta et al., 1988, 1990).

As can be seen from Fig. 5, alunite-AY 17 exhibits a different kinetic behavior to the others. A possible explanation for the difference in adsorption of the dyes can be given on the basis of their molecular weight and structural complexity. Desai et al. (1997) showed in their study on adsorption of acid dyes by neutral alumina that complex molecular structures adsorb less and take more time for saturation of adsorption. In other words, the equilibrium adsorption and adsorption kinetics are dependent upon the molecular dimensions of dyes.

3.5. Adsorption Dynamics

The rate constant of adsorption is determined from the first order rate expression given by Lagergren (Gupta et al., 1990; Annadurai and Krishnan, 1997).

$$\log(q_e - q) = \log q_e - \frac{k_{ad}}{2.303} t \quad (6)$$

where q_e and q are the amounts of dye adsorbed (mg/g) at equilibrium and at time t (min), respectively, and k_{ad} is the rate constant of adsorption (1/min). Linear plots of $\log(q_e - q)$ vs t both calcined alunite-acid dyes and GAC-acid dyes combinations were obtained and shown in Fig. 6. The values of k_{ad} for different dye-adsorbent combinations were calculated from the slopes of the straight lines (Fig. 6) and given in Table 3. This shows that the adsorption process follows the first order rate expression.

3.6. Adsorption Isotherms

Figures 7 and 8 show adsorption isotherms, the relationship between the amount of acid dyes adsorbed per unit mass of calcined alunite and GAC (q_e) and

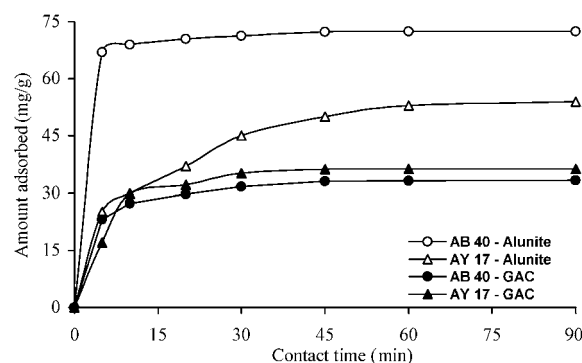
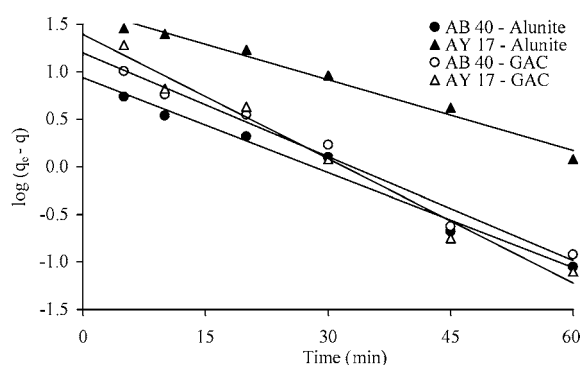
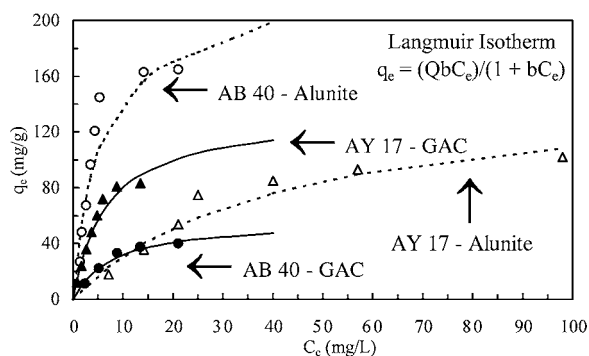


Figure 5. Effect of contact time on adsorption of acid dyes by calcined alunite and GAC. Conditions: 75 mg/L concentration, 90–150 μm particle size, 873 K and 30 min calcination, 1 g/100 mL dose, 298 K temperature and pH 2 for calcined alunite and pH 1 for GAC.

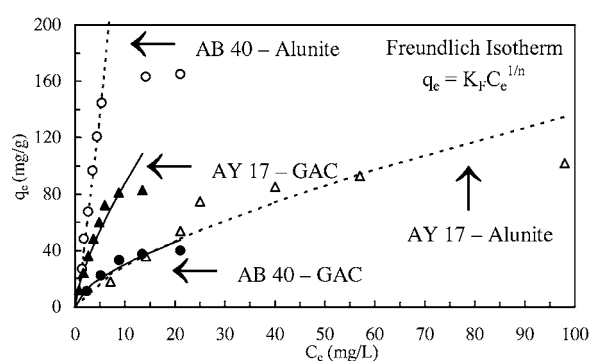
Table 3. Lagergren rate constant and Langmuir and Freundlich constants for adsorption of two acid dyes.

Adsorbent	Dye	Lagergren		Langmuir constant			R_L	Freundlich constant		
		k_{ad} (min ⁻¹)	r^2	Q (mg/g)	b (L/mg)	r^2		K_F	n	r^2
Calcined alunite	AB 40	7.65×10^{-2}	0.981	212.8	0.198	0.921	0.026	20.26	0.847	0.973
	AY 17	5.74×10^{-2}	0.981	151.5	0.025	0.912	0.167	6.32	1.498	0.880
GAC	AB 40	8.41×10^{-2}	0.978	57.47	0.123	0.970	0.119	7.70	1.679	0.928
	AY 17	10.04×10^{-2}	0.979	133.3	0.149	0.936	0.063	17.38	1.419	0.952

Figure 6. Lagergren plot for adsorption of acid dyes on calcined alunite and GAC. Conditions: 75 mg/L concentration, 90–150 μ m particle size, 873 K and 30 min calcination, 1 g/100 mL dose, 298 K temperature and pH 2 for calcined alunite and pH 1 for GAC.Figure 7. Adsorption isotherms of acid dyes by calcined alunite and GAC. (O, Δ , \bullet , \blacktriangle) experimental data and (—), (----) Langmuir isotherm. Conditions: 90–150 μ m particle size, 873 K and 30 min calcination, 1 g/100 mL dose, 298 K temperature and pH 2 for calcined alunite and pH 1 for GAC.

their final concentrations in the aqueous phase (C_e). The plots of dye uptake against equilibrium concentration (Figs. 7 and 8) indicate that adsorption increases initially with concentration but then reaches saturation.

Adsorption isotherms were analyzed according to the linear form of the Langmuir isotherm agents (Desai

Figure 8. Adsorption isotherms of acid dyes by calcined alunite and GAC. (O, Δ , \bullet , \blacktriangle) experimental data and (—), (----) Freundlich isotherm. Conditions: 90–150 μ m particle size, 873 K and 30 min calcination, 1 g/100 mL dose, 298 K temperature and pH 2 for calcined alunite and pH 1 for GAC.

et al., 1997; Al-Degs et al., 2000; Namasivayam et al., 2001).

$$\frac{C_e}{q_e} = \frac{1}{bQ} + \frac{C_e}{Q} \quad (7)$$

where C_e is the concentration of dye solution (mg/L) at equilibrium, q_e is the amount of dye adsorbed per unit weight of adsorbent (mg/g). The constant Q signifies the adsorption capacity (mg/g) and b is related to the energy of adsorption (L/mg). The values of Q and b were calculated from the slope and intercept of the linear plot, C_e/q_e vs C_e (Fig. 9) and are represented in Table 3. The applicability of the Langmuir isotherm suggests the monolayer coverage of both acid dyes on the surface of calcined alunite and GAC. The essential characteristics of Langmuir equation can be expressed in terms of a dimensionless separation factor R_L , which is defined by Al-Degs et al. (2000) and Namasivayam et al. (2001), as:

$$R_L = \frac{1}{1 + bC_0} \quad (8)$$

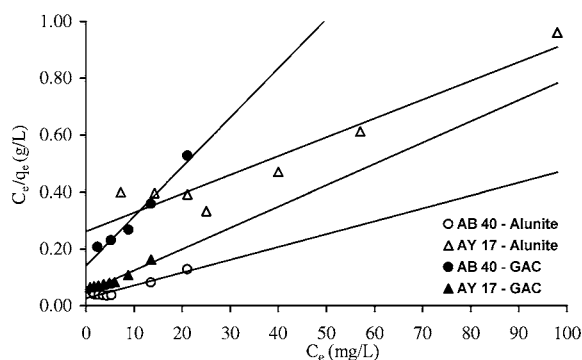


Figure 9. Linear Langmuir plots for the adsorption of acid dyes.

where C_0 is the highest initial dye concentration (mg/L) and b is the Langmuir constant, R_L values indicate the shape of isotherm. The R_L value between zero and one indicate favorable adsorption. The R_L values were found to be between zero and one for both the dyes for each adsorption (Table 3).

The linearized Freundlich isotherm equation is shown in Eq. (9) agents (Ramakrishna and Viraraghavan, 1997; Namasivayam et al., 2001).

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (9)$$

where K_F and n are Freundlich constant. The linear plot of $\log q_e$ vs $\log C_e$ (Fig. 10) indicates the applicability of the Freundlich isotherm and for the present system exhibits a monolayer coverage of the adsorbate on the outer surface of the adsorbent. The K_F and n values were calculated from the intercept and slope of the plots and are listed in Table 3.

Figures 7 and 8 also show a comparison between the theoretical Langmuir and Freundlich isotherms, re-

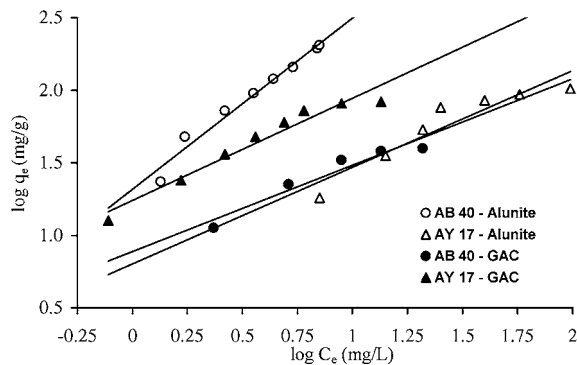


Figure 10. Linear Freundlich plots for the adsorption of acid dyes.

spectively, and the experimental data. Both adsorption isotherm models were found to generate a satisfactory fit to the experimental data as indicated by correlation coefficient values (r^2). But the Langmuir model shows a better fit to adsorption data than the Freundlich model (Figs. 7 and 8).

The Q values for the two dyes in Table 3 reveal that calcined alunite had a greater affinity (or capacity) for AB 40 than AY 17 while activated carbon F-400 had a greater capacity for AY 17. The different affinities of the dyes with the adsorbents result from the different degree of interaction between the adsorbates and the adsorbents. At the same time, the difference in adsorption capacities between the two anionic dyes comes from its different molecular structures and different molecular weights. The molecular weights of AB 40 (anthraquinone dye) and AY 17 (monoazo dye) are 473 and 548 g/mol, respectively.

Al-Degs et al. (2000) explained that the differences in capacities of adsorbents for same adsorbate are caused by their surface properties. Activated carbon has a high adsorption capacity or reactivity towards a range of organic pollutants. This reactivity arises from the complexity of the chemical surface groups compared to other surfaces, for example, inorganic solids such as calcined alunite and metal oxide surfaces consist mainly of oxygen atoms and hydroxyl groups. It is primarily the hydroxyl groups that determine the chemistry (acid-base character) and the reactivity of these surfaces. In contrast, the surface of carbon materials can contain not one but, at least, five markedly different types of surface groups such as carboxylic, lactonic, phenolic, carbonyl and etheric types. This diversity of surface groups make the surface chemistry (acid-base character) much more versatile than that of other adsorbents. The adsorption capacity of calcined alunite is higher than that of GAC for the same dyes at the same conditions. These results indicate that calcined alunite has good capabilities as an adsorbent for acid dyes.

4. Conclusion

The use of calcined alunite and GAC as adsorbents for two acid dyes, AB 40 and AY 17, was studied. The optimum calcination temperature and time of alunite were found to be 873 K and 30 min, respectively, for adsorption of acid dyes. The adsorbed amounts of acid dyes increased with decreasing particle size of alunite. The surface charge on the adsorbent and the

solution pH play a significant role in influencing the capacity of an adsorbent towards dye ions. Having an excess of positive charge on their surfaces, calcined alunite and GAC have a greater capacity to adsorb acid dyes. The adsorbed amounts of acid dyes increased with increase in contact time and reached the equilibrium at 90 min. Adsorption of dyes followed first order rate kinetics. Adsorption capacities of AB 40 and AY 17 are 57.47 mg and 133.3 mg/g GAC, respectively. Calcined alunite, however has considerable adsorption capacities are 212.8 and 151.5 mg dye/g calcined alunite for AB 40 and AY 17, respectively. Thus calcined alunite has considerable potential as an adsorbent for dyestuffs in a commercial system. Adsorption followed both Langmuir and Freundlich isotherms.

GAC is an expensive material and regeneration is essential, whereas alunite is so cheap that regeneration is not necessary. Based on the above good results this relatively cheap, low-cost material is recommended as effective and cheap adsorbent for dyestuffs removal from textile effluents.

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