



STATISTICAL INVESTIGATION OF ADSORPTION OF TWO REACTIVE TEXTILE DYES BY VARIOUS ADSORBENTS

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ABSTRACT

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Textile industry, in which uses the dyestuffs containing coloured and complex chemical compounds, is both water consumer and water pollutant. The removal of these compounds from the wastewaters is one of the most important problems in the textile industry. In this study, the adsorption of two reactive dyes (Red Px and Yellow P) onto Aşkale and Balkaya lignites, Bensan clay and powdered active carbon (PAC) from aqueous solution was statistically investigated. The adsorption time, dye concentration, solid/liquid ratio and mixing rate were chosen as parameters. The effects of these parameters on the amount of dye adsorbed by the adsorbents were determined. The results obtained have been statistically evaluated by using the stepwise method and SPSS Sortware version (9.1). The experimental observations and statistical evaluations shown that the effective parameters on the adsorption are equilibrium dye concentration and solid/liquid ratio. It was found that the adsorptive behaviours of both lignites and clay are similar to each other, but powdered active carbon displays different adsorptive behaviour. Finally, the empirical equations showing the relation between amount of dye adsorbed and the effective parameters were developed.

Key Words : Textile dyes, Adsorption, Adsorption kinetics, Decolorization

REAKTİF İKİ TEKSTİL BOYALARININ FARKLI ADSORBENTLERLE ADSORPSİYONUNUN İSTATİSTİKSEL İNCELENMESİ

ÖZET

Renkli ve karmaşık kimyasal bileşikleri içeren boyar maddeleri kullanan tekstil endüstrisi hem temel bir su kullanıcısı hem de kirleticisidir. Atık sudan bu bileşiklerin uzaklaştırılması tekstil endüstrisindeki en önemli problemlerden biridir. Bu çalışmada, sulu çözeltiden aktif karbon, Bensan kili, Aşkale ve Balkaya linyitleri üzerine 2 reaktif boyanın (Red Px ve Yellow P) adsorpsiyonu istatistiksel olarak araştırıldı. Parametreler olarak adsorpsiyon zamanı, boya konsantrasyonu, katı/sıvı oranı ve karışım hızı seçildi. Adsorbentler tarafından adsorplanan boyanın miktarı üzerine bu parametrelerin etkileri belirlendi. Elde edilen sonuçlar SPSS Sortware versiyonu (9.1) ve Stepwise metodu kullanılarak istatistiksel olarak değerlendirildi. Deneysel gözlemler ve istatistiksel değerlendirmeler adsorpsiyon üzerine etkili parametrelerin katı/sıvı oranı ve denge boya konsantrasyonu olduğunu gösterdi. Hem linyitlerin hem de kilin adsorpsiyon davranışlarının birbirine benzer olduğu fakat aktif karbonun farklı adsorpsiyon davranışı gösterdiği bulundu. Sonunda, etkili parametreler ve adsorplanan boyanın miktarı arasında ilişkiyi gösteren ampirik denklem geliştirildi.

Anahtar Kelimeler : Tekstil boyları, Adsorpsiyon, Adsorpsiyon kinetiği, Renk giderme

1. INTRODUCTION

Dyes are released into the environment in industrial effluents from two major sources, the textile and the dyestuff industries (Cripps et al., 1990). A necessary criteria for the use of these dyes is that they must be highly stable in light and during washing. They must also be resistant to microbial attack. Therefore, they are not readily degradable and are typically not removed from water by conventional wastewater treatment systems (Brown and Laboureur, 1983; Pagga and Brown, 1986; Cripps et al., 1990; Ganes et al., 1994; Sheng and Chi, 1994; Smith et al., 1997; Young and Yu, 1997; Daniş, et al., 1999). Textile industrial effluents are an important source of water pollution (Ahmet and Ram, 1992). Over 7×10^5 tons and approximately 10.000 different dyes and pigments are produced annually world wide. It is estimated that from 10 to 15 % of the dye is lost in the effluent during the dyeing process (Young and Yu, 1997). Wastewater discharges from textile dyes houses are complex chemical solutions which are very highly coloured (Sheng and Chi, 1994). The effects of coloured effluents in aquatic environments are not only aesthetic, but they can interfere with the transmission of sunlight and lead to a reduction in self-purification ability and also a decrease in the numbers and biodiversity of flora and fauna (Sheng and Chi, 1994; Daniş et al., 1999). However, coloured discharges may cause the increasing of Biological and Chemical Oxygen Demand (BOD and COD) (Sheng and Chi, 1994). Toxic constituents of discharged effluents may play an important role in reducing of the quality of the aquatic environment (Cripps et al., 1990; Spadora et al., 1992). They also exhibit great structural variety and are not uniformly susceptible to microbial attack. These dyes are not typically degraded under aerobic conditions. However, under anaerobic conditions, the azo linkage can be reduced to form aromatic amines which are colourless but which can be toxic and carcinogenic (Cripps et al., 1990). On the other hand, ozonation is a technique that has been often suggested in the literature in recent years as a potential alternative. Ozonation is not sufficient for high strength dye waste effluents. However, ozonation is alone sufficient to totally eliminate the colour and reduce the turbidity only for low strength dye waste effluents. Adsorption has been found to be an efficient and economically cheap process to remove pollutants such as dye and other chemicals and to control the BOD (Gupta and Bhattacharya, 1985; Allen et al., 1989; Ahmed and Ram, 1992; Nassar et al., 1995; Rita et al., 1996; Youssef et al., 1996). Inorganic oxides, such as those of aluminium,

silica and carbon have been extensively used as adsorbents (Allen, 1987; McKay and Al-Duri, 1988; Gupta et al., 1988; Navar and Doma, 1989; Ahmed and Ram, 1992). The adsorption process using active carbon which is a porous material with a very large surface area has shown to be effective in the removal of many nondegradable organic pollutants existent in wastewater (Abu Zeid et al., 1995; Sulaiman et al., 1997). In addition, it is known that clays and lignites may be used as alternative adsorbents in the adsorption processes (Gürses et al., 1995). In this study, the statistical investigation of adsorption of two reactive dyes, Red Px and Yellow P (which are the samples chosen of a common dyestuff class used in Turkish Textile Industry) by different adsorbents such as lignite, clay and active carbon has been aimed. It has also been tried to develop the empirical equations showing relation between the amount of dye adsorbed and the parameters investigated for different dyes and adsorbent types.

2. EXPERIMENTAL

2. 1. Material

In this study; as adsorbents, lignites from Erzurum-Balkaya and Aşkale coal basin in Turkey and commercial powder active carbon (Merck cat. No. 2184) and a clay sample (Bensan) were selected. The lignites were milled and sieved to 425 μm and then characterized by chemical analysis. Ultimate analysis of the lignites were performed using Turkish and ASTM standards and the results together with their other some characteristic properties are given in Table 1. The approximately wt. % 80 of the powder active carbon and the clay used in this work have the particle size of 150 μm . The results of the some analyses for these last two adsorbents were shown in Table 2. In the experiments, the adsorption of the two reactive azo dyes, procion Red Px and procion Yellow P (produced by ICI Inc. In England) were investigated by these adsorbents. These dyes are two representative samples of common dye stuff class used in Turkish Textile Industry. In addition, the two principal oxygen-containing functional groups, carboxylic and phenolic for the some adsorbents were determined by using the methods which are described previously (Miller et al., 1983; Gürses et al., 1987). The results of these analyses together with the other results for the both lignites and PAC were given in Table 1 and 2, respectively.

Table 1. The Results of the Some Analysis For Aşkale and Balkaya Lignites Used in This Study and Their Some Characteristic Properties

Analysis	Aşkale	Balkaya
Proximate Analysis (wt. % as Received)		
Moisture	4.46	8.00
Ash	29.64	22.20
Volatile matter	34.28	29.40
Fixed carbon	31.62	40.40
Ultimate Analysis (wt. % Daf ^a)		
Carbon	70.86	72.50
Hydrogen	4.28	4.10
Sulfur	4.76	2.30
Nitrogen	2.20	2.00
Oxygen (diff)	17.90	19.10
Oxygen Functional Groups (wt. % Daf ^a)		
Carboxylic groups	0.58	1.20
Phenolic groups	8.91	1.60
Surface area(BET,N ₂ ,m ² g ⁻¹)	2.20	2.80

^a: Dry, ash-free basis

Table 2. The Results of the Some Analysis For Powder Active Carbon (PAC) and Bensen Clay and Their Some Characteristic Properties

Analysis	PAC ^a	Bensen clay ^b
Chemical analysis (wt. %)		
SiO ₂	-	62.7
CaO	-	2.4
Al ₂ O ₃	-	17.1
MgO	-	1.9
Na ₂ O	-	0.5
K ₂ O	-	1.6
Residue on ignition (600°C)	<2	-
Loss in drying (120°C)	<10	9.0
Surface area(BET,N ₂ , m ² g ⁻¹)	900	49.22
Oxygen functional groups (wt. % daf ^c)		
Carboxylic groups	10.7	-
Phenolic groups	6.1	-

^a: For Merck chemical agent catalogue, ^b As received basis, ^c: Dry, ash-free basis

2. 2. Methods

Batch adsorption experiments for the study of the kinetics of adsorption were carried out using 100 mL of the aqueous solutions of both dyes having 100 mgL⁻¹ initial concentration at the constant shaking speed (238 rpm) and at 20 °C, for several adsorption periods. Because, the hydrophobic particles of both the lignites and powdered active carbon could be completely wetted by strongly shaking the mixture of solid-liquid, a high mixing rate of 238 rpm in this work has been preferred. In this work has been preferred. In this experiments the solid/liquid ratios for the first three adsorbents(Aşkale, Balkaya, Clay) and PAC were 3.0/100 g mL⁻¹ and 0.5/100 g mL⁻¹, respectively. After each adsorption period, the aliquots of 5 mL from the adsorption mixture were taken, decanted and centrifuged at 5000 rpm for 15 minutes. The concentrations of the dyes in the supernatant solutions were spectrophotometrically determined to their blank solutions at their maximum wavelengths (227.2 nm for Yellow P and 226.8 nm for Red Px). In order to obtain the adsorption isotherms, 100 mL of aqueous dye

solutions having the different initial concentrations and the constant solid/liquid ratios were added onto erlenmeyer flasks of 100 mL. These were then shaken on a thermostated-shaker, at the mixing rate of 238 rpm and 20 °C for 1 h. At the end of the period 1h, the dye concentration in the supernatants was determined as in the previous step. While the effect of solid/liquid ratio is being investigated, the experiments for the several solid/liquid ratios were conducted at the initial dye concentration of 100 mgL⁻¹, the mixing rate of 238 rpm and 20 °C for 1h. On the other hand, the effect of mixing rate on the amount of dye adsorbed has been investigated under the constant conditions as the adsorption time of 1h, the initial dye concentration of 100 mgL⁻¹, the temperature of 20 °C and the solid/liquid ratios of 3.0/100 g mL⁻¹ for the first three adsorbents and 0.5/100 g mL⁻¹ for PAC. In both cases, the dye concentration in the supernatants was determined as in the previous cases. The amount of dye adsorbed per 1g of the adsorbent (x, mg g⁻¹) was calculated as follows:

$$x(\text{mg g}^{-1}) = (C_0 - C_e) \cdot V/m \quad (1)$$

Where; C_0 , C_e and m , V are the initial dye concentration (mgL^{-1}), the equilibrium dye concentration (mgL^{-1}) and the amount of adsorbent(g), total solution volume (mL), respectively.

3. RESULTS AND DISCUSSION

The adsorption of the both dyes on the adsorbents as a function of adsorption time for a sample of clay, the lignites of Balkaya and Aşkale and powdered activated carbon are individually given in Figures 1-3, respectively. From these Figures, it may be seen that after about adsorption time of 1h, no significant change occur with time in adsorption capacity and also the adsorption of both dyes onto all adsorbents rapidly takes place at the beginning of adsorption process. This could be due to presence of intensive electrostatic and hydrophobic interactions which especially play a significant role in the adsorption of dye molecules on activated carbon surface and effective surface diffusion instead of pore diffusion (Motta et al., 1994; Zhang and Ritter 1997).

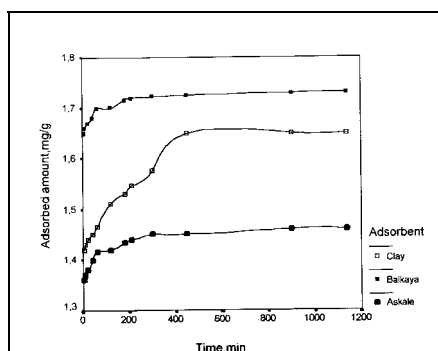


Figure 1. Variation of the adsorption of Yellow P dye on the various adsorbents with adsorption time (20°C , mixing rate : 238 rpm, solid/liquid ratio: 3gL^{-1} and $C_0=100\text{mgL}^{-1}$)

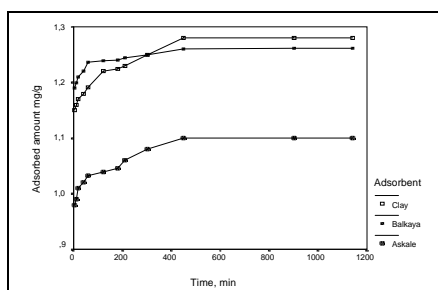


Figure 2. Variation of the adsorption of Red Px dye on the various adsorbents with adsorption time (20°C , mixing rate : 238 rpm, solid/liquid ratio : 3gL^{-1} and $C_0=100\text{mgL}^{-1}$)

The extreme short equilibrium adsorption times for some adsorption process in which the mechanism of physical adsorption, i.e. electrostatic and hydrophobic interactions is predominant have been reported (Gürses et. al., 1992; Couillard, 1994; Gürses et al., 1995).

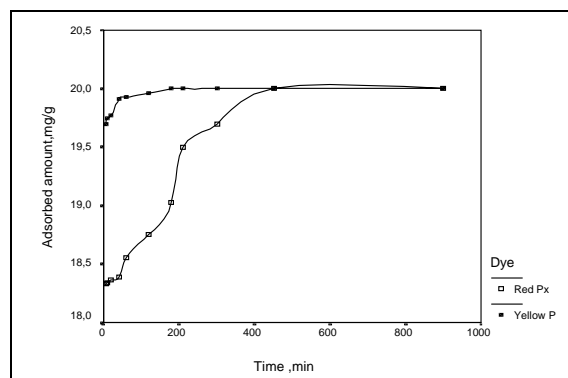


Figure 3. Variation of the adsorption of Yellow P and Red Px dyes on the powdered active carbon with adsorption time (20°C , mixing rate : 238 rpm, solid / liquid ratio : 0.5gL^{-1} and $C_0=100\text{mgL}^{-1}$)

In order to investigate the variation of adsorption of both dyes on the various adsorbents with the equilibrium dye concentrations, the experiments with the different initial dye concentrations for adsorption period of 1h were carried out. The isotherms for Red Px and Yellow P on Aşkale and Balkaya lignites and clay, and also both dyes on the powdered active carbon are shown in Figures 4, 5 and 6, respectively. From Figures 4 and 5, it can be seen that the amount of dye adsorbed increases with the increasing of the equilibrium dye concentration. The adsorption yield of Yellow P dye on Balkaya lignite is higher than that of the other adsorbents except to PAC. This could indicate the effective hydrophobic interactions between Yellow P molecules and the surface of Balkaya lignite which have the higher carbon content and the lower percentage of oxygen functional groups (Table 1) and so is more hydrophobic than Aşkale lignite and clay. As can be seen from Figure 6, the adsorption yield of Yellow P on the surface of PAC is also higher than that of the other dye, supporting the hydrophobic adsorption mechanism due to the fact that the most possible adsorption mechanism for both dyes in the case of PAC is hydrophobic bonding.

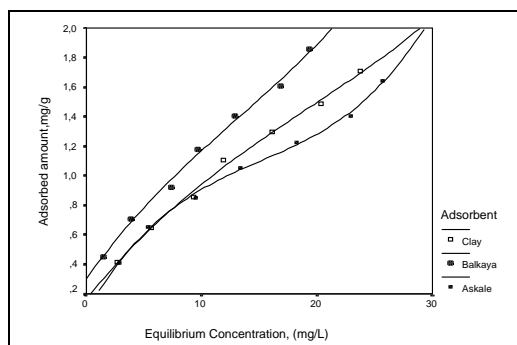


Figure 4. Variation of the adsorption of Yellow P dye on the various adsorbents with equilibrium dye concentration (20 °C, adsorption time of 1h, mixing rate : 238 rpm and solid / liquid ratio : 3 g/L⁻¹)

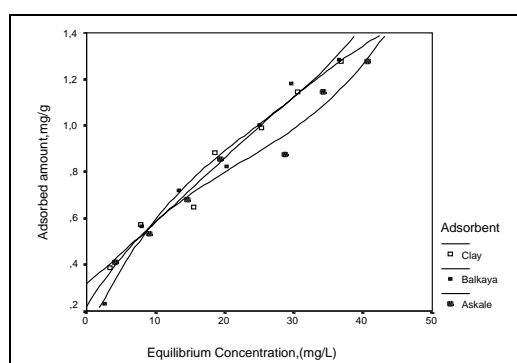


Figure 5. Variation of the adsorption of Red Px dye on the various adsorbents with equilibrium dye concentration (20 °C, adsorption time of 1h, mixing rate : 238 rpm and solid / liquid ratio : 3 g/L⁻¹)

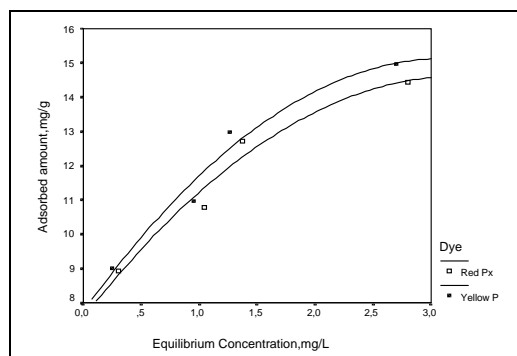


Figure 6. Variation of the adsorption of Yellow Pand Red Px dyes on the powdered active carbon with equilibrium dye concentration (20 °C, adsorption time of 1 h, mixing rate : 238 rpm and solid/liquid ratio : 0.5 g/L⁻¹)

In addition, although the clay used in this work has extensive surface area comparison to the both lignites (Table1), the tendencies of adsorption of the both dyes onto the surface of clay are similar that of the surfaces of the lignites. This can be attributed to its extensive surface area and the weak hydrophobic

interactions between dye molecules and the surface of clay. The variations with solid/liquid ratio of the adsorption of the dyes of Yellow P and Red Px on Aşkale and Balkaya lignites, the clay and powdered active carbon have been graphically shown in Figures 7, 8 and 9, respectively. From these Figures, it can be seen that in the cases of all dyes and adsorbents, the amount of dye adsorbed decreases with the increasing of solid/liquid ratio and the decreasing trends are similar to each other. At the lower range of solid/liquid ratios, the decreasing tendency of the amounts adsorbed for all of the adsorbents is extremely high. This similarity implies to the fact that the decreasing tendency can be correlated to the bulk behaviours of both the dye molecules and the grains of the adsorbents but not their characteristic properties. By considering the probability which the adsorption of dye molecules onto the surfaces of the adsorbent may predominantly occur with the hydrophobic interactions, it can be said that the interactions between the grains of the adsorbents begin to be predominate with the increasing of solid/liquid ratio.

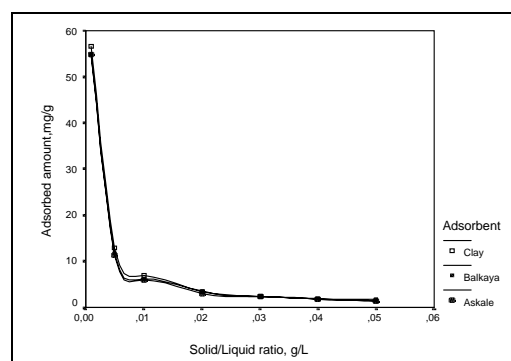


Figure 7. Variation of the adsorption of Yellow P dye on the various adsorbents with solid/liquid ratio (20 °C, adsorption time of 1 h, mixing rate : 238 rpm and C₀ : 100 mg/L⁻¹)

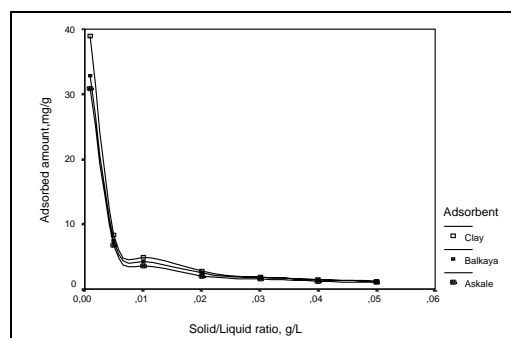


Figure 8. Variation of the adsorption of Red Px dye on the various adsorbents with solid/liquid ratio (20 °C, adsorption time of 1 h, mixing rate : 238 rpm and C₀ : 100 mg/L⁻¹)

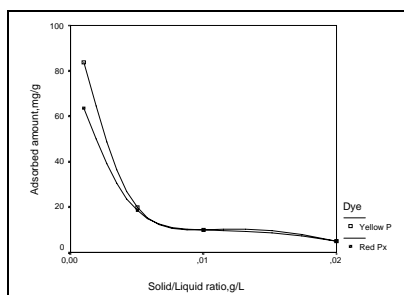


Figure 9. Variation of the adsorption of Yellow P and Red Px dyes on the powdered active carbon with solid/liquid ratio (20 °C, adsorption time of 1 h, mixing rate : 238 rpm and C_0 : 100 mgL⁻¹)

At the adsorption of phenols by powdered active carbon, it has been reported that the sorption rate increased with the decreasing in the number of carbon grains and the increasing of macropore volume (Peprowica, 1990).

The variation of adsorbed amounts of Yellow P and Red Px with the mixing rate for various adsorbents is given in Figures 10 and 11, respectively. Again, the variation of adsorbed amount of Yellow P and Red Px with the mixing rate for the activated carbon is given in Figure 12. These Figures show that the amounts of dye adsorbed in the cases of all of the dyes and adsorbents slightly increased with the increase of mixing rate. This is probably due to the diffusion of the dye molecules from aqueous solution to the surfaces of the solid adsorbents may extremely easily occurs at least the range of variation of mixing rate in this work. The relative increases in the amounts of dye adsorbed with the increased mixing rate may also be attributed to the decrease in the hydrophobicity of the particles of the adsorbents.

On the other hand, in order to support the above qualitative explanations for the adsorption of two the reactive dyes on the various adsorbents, the mathematical equations were developed.

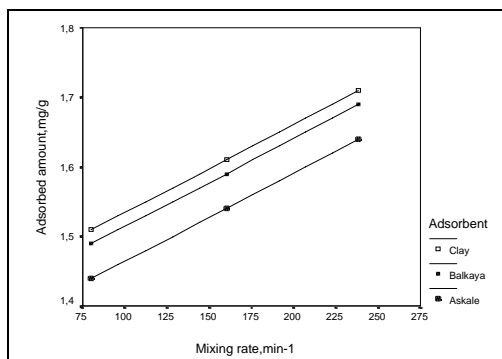


Figure 10. Variation of the adsorption of Yellow P dye on the various adsorbents with mixing rate (20 °C, adsorption time of 1 h, solid/liquid ratio : 3 gL⁻¹ and C_0 : 100 mgL⁻¹)

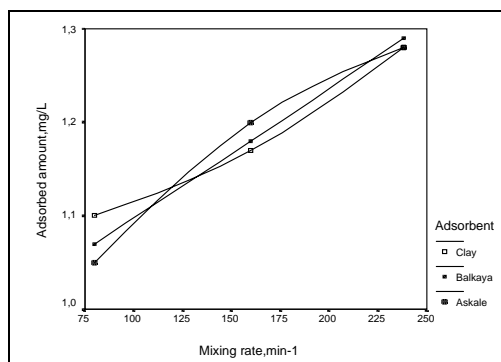


Figure 11. Variation of the adsorption of Red Px dye on the various adsorbents with mixing rate (20 °C, adsorption time of 1 h, solid/liquid ratio : 3 gL⁻¹ and C_0 : 100 mgL⁻¹)

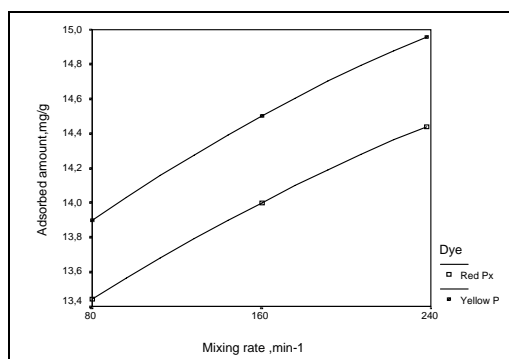


Figure 12. Variation of the adsorption of Yellow P and Red Px dyes on the powdered active carbon with mixing rate (20 °C, adsorption time of 1 h, solid/liquid ratio : 0.5 gL⁻¹ and C_0 : 100 mgL⁻¹)

These show the relation between the amount of dye adsorbed and the variables such as adsorption time, equilibrium dye concentration, solid/liquid ratio and mixing rate. For this, all of the variables were taken as a function of the amount of dye adsorbed in the following form:

$$x = f(T, C, R, V) \quad (2)$$

Equation (2) was then rewritten by rearranging the variables in the form of dimensionless groups, as follows:

$$x = k \cdot (T^t \cdot C^c \cdot V^v / R^r) \quad (3)$$

where, x, T, C, V and R are the amount of dye adsorbed per 1 g adsorbent (mg g⁻¹), equilibrium dye concentration (mgL⁻¹), adsorption time (min.), mixing rate (min⁻¹) and solid/liquid ratio (g L⁻¹), respectively. The k constant and the exponents (T, C, V and R) in Equation 3 have been statistically calculated by using the stepwise method and SPSS software version 9.01. The statistical evaluations

indicated that amongs the variables investigated in this work, equilibrium dye concentration and solid/liquid ratio were effective variables on the adsorption of reactive dyes by the different adsorbents. Finally, three different mathematical equations for the adsorptions of Yellow P on Aşkale and Balkaya lignites and the clay, Red Px on Aşkale and Balkaya lignites and the clay and also the adsorption of both the dyes on PAC were individually obtained as follows:

$$x \text{ (mg g}^{-1}\text{)} = 13.8 (C^{0.321}) (R^{-1.032}) \quad (4)$$

$$x \text{ (mg g}^{-1}\text{)} = 9.58 (C^{0.397}) (R^{-0.941}) \quad (5)$$

$$x \text{ (mg g}^{-1}\text{)} = 509 (C^{0.211}) (R^{-0.586}) \quad (6)$$

The constants for Equations 4 and 5 are depend on both of the temperature and the type of dye. Whereas, the constant for Equation 6 is only depend on the temperature. In addition, theoretical values calculated from Eqs. 4, 5 and 6 of the amount of dye adsorbed by the adsorbents have been graphically compared with their experimental values in Figures 13, 14 and 15, respectively. The high R- squares values for the comparisons in these figures may suggest that the predicted values of the amounts of dye adsorbed are closely agreed with their observed values. As a result, it can be said that the equations obtained could provide a true correlation between the amount of dye adsorbed and the variables such as equilibrium dye concentration and solid/liquid ratio. In addition, the experimental observations and statistical evaluations show that the samples of the lignite and clay used in this work have almost the same effectiveness on the removal of reactive dyes from the aqueous solutions and also they can be used as an alternative for the more expensive adsorbents such as powdered active carbon.

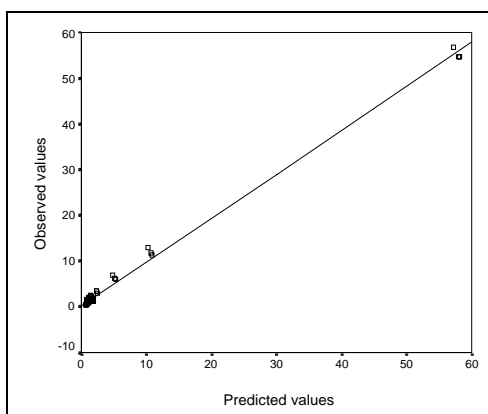


Figure 13. Comparison of the observed values for the adsorption of Yellow P dye on Balkaya and Aşkale lignites and clay with the values predicted from Equation 4 ($R^2 = 90.6$)

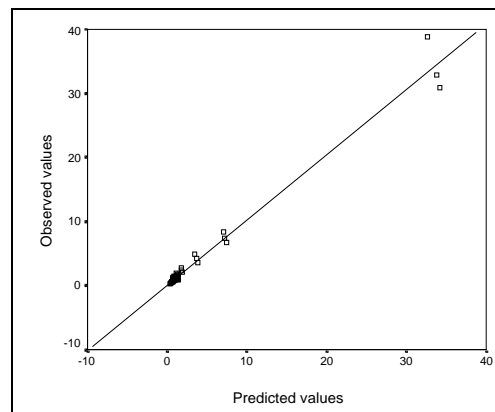


Figure 14. Comparison of the observed values for the adsorption of Red Px dye on Balkaya and Aşkale lignites and clay with the values predicted from Equation 4 ($R^2 = 92.9$)

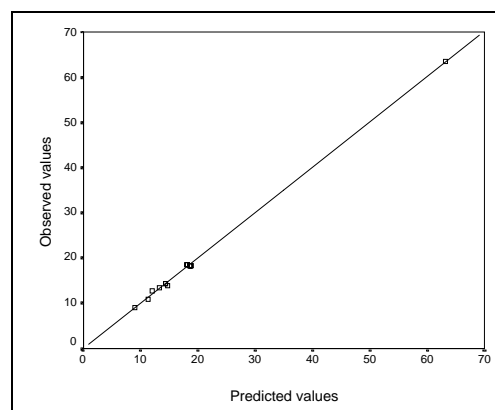


Figure 15. Comparison of the observed values for the adsorption of Yellow P and Red Px dyes on the powdered active carbon with the values predicted from Equation 4 ($R^2 = 95.7$)

4. ACKNOWLEDGEMENT

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