

# Dyeing Kinetics of Methacrylic Acid Grafted Polyester Fabric with Astrazonrot Violet and Rhodamine Red Basic Dyes

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**ABSTRACT:** The low-temperature dyeing kinetics of radiation-grafted poly(ethylene terephthalate) fabric were studied. The effects of the graft yield (GY), dye concentration, and dyeing temperature on the color difference (CD) of methacrylic acid grafted polyester fabric were studied for astrazonrot violet (AV) and rhodamine red (RR) basic dyes. CD increased sharply with an increase in GY and tended to level off at higher degrees of grafting. The best dyeing conditions were achieved for both dyes at pH 11.5. CD of the grafted fabric increased rapidly as the dyeing time increased; this was followed by a relatively slow dyeing rate within a few minutes, which depended on the concentration and temperature of the dye bath. The initial dyeing rates and rate constants for the AV dye were higher than those for the

RR dye. The dyeing process followed 0.14-order kinetics and was independent of the dyeing temperature or the type of dye. The dyeing rates and rate constants increased with an increase in the dyeing temperature. An Arrhenius-type plot of the natural logarithm of the dyeing rate constant versus the inverse of the absolute temperature yielded apparent activation energies of 4.9 and 13.8 kJ/mol and pre-exponential rate constants of 9.4 and 100.6 (CD/GY)s<sup>-1</sup> for the AV and RR dyes, respectively. The mechanism of the dyeing process for the two dyes was diffusion-controlled, and their dyeing rates depended on the type of basic dye. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 94: 1070–1076, 2004

**Key words:** activation energy; polyesters; radiation

## INTRODUCTION

Extensive investigations into the improvement of the physical and chemical properties of polymeric substrates by graft copolymerization with vinyl monomers have been published. Considerable literature on the subject was reviewed by Chapiro<sup>1</sup> and Hebeish and Guthrie.<sup>2</sup> Grafting cotton with polyacrylonitrile, polyacrylamide, and poly(methyl methacrylate) affects its mechanical properties.<sup>3</sup> The moisture regain, water sorbency, and electrical conductivity of grafted polymeric materials depend on the polymer–monomer combination. Increases and decreases in physical properties have been reported for the grafting of natural and synthetic materials with different vinyl monomers.<sup>4–12</sup> The dyeing properties of cotton, polyester, and polyamide fabrics have shown substantial improvements from the radiation grafting of the fabrics with the proper monomers. Grafting the fabrics with dimethylaminoethyl methacrylate considerably increases the color strength of reactive drimalan red dye.<sup>12</sup> Ny-

lon-6 fabrics grafted with *N*-vinyl pyrrolidone have increased dyeability toward solar orange direct dye.<sup>7</sup> The radiation grafting of cotton with poly(acrylic acid) significantly improves the affinity of sandocryl blue basic dye to the fabric.<sup>6</sup>

Basic dyes are known to have no affinity toward polyester fabrics. This drawback has been overcome by the addition of acidic groups to the polymer macromolecules via the radiation grafting of the fabrics in acrylic acid<sup>6</sup> and methacrylic acid (MAA) solutions.<sup>7</sup> In these investigations, we studied the effect of the pH of the dye bath and the degree of grafting on the color strength of the dyed fabrics. The best results were obtained at pHs 3.9 and 11.5 for cotton and polyester fabrics, respectively. An increase in the graft yield (GY) substantially increased the color strength of the fabrics.

This article presents studies of the low-temperature dyeing kinetics of MAA-grafted polyester poly(ethylene terephthalate) (PET) fabric with astrazonrot violet (AV) and rhodamine red (RR) basic dyes. The effects of the GY, pH of the dye bath, dye concentration (*C*), and dyeing temperature (*T*) on the color difference (CD or  $\Delta E^*$ ) of the dyed fabrics were investigated. The kinetic parameters [reaction order (*n*), dyeing rate (*R<sub>d</sub>*) and dyeing rate constant (*k<sub>d</sub>*), and the activation energy of the dyeing process (*Q*)] were determined.

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## EXPERIMENTAL

### Materials

Thermally stabilized (heat-treated at 493 K for 1.5 min) low-density polyester fabric, a product of Hankook Synthetic Inc. (Kangwondo, Korea), was mill-scoured in a solution containing 0.001 g/L Data Scour WS-100 and sodium carbonate (0.5 g/L) at boiling for 1 h. The fabric was thoroughly washed with hot water, dried at the ambient temperature, and then used for grafting.

The MAA monomer and other chemical reagents were used as-received. AV (C.I. 48020) and RR (C.I. 45170), basic dyes produced by Sandoz (Basle, Switzerland), were used.

### Methods

#### Radiation grafting

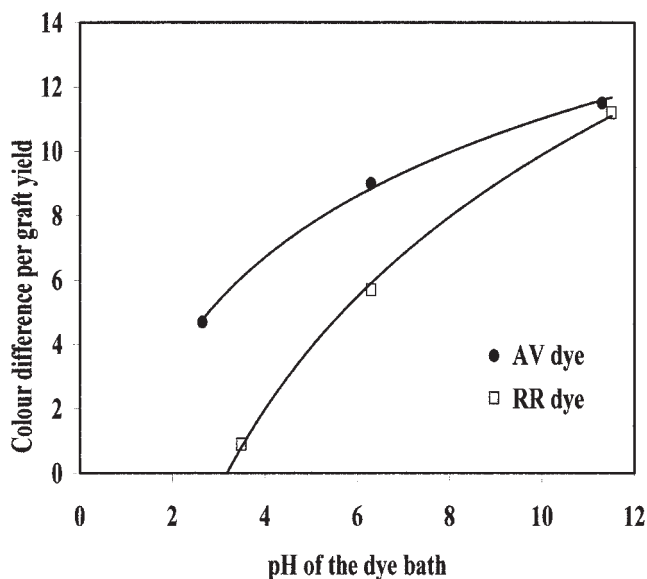
Grafting was carried out by the direct irradiation method in a  $^{60}\text{Co}$   $\gamma$  source at a dose rate of 1.98 Gy/s with different doses and MAA concentrations to achieve a wide range of GYs. Dry and weighed polyester samples (ca. 0.7 g) were swollen in chloroform overnight before being put in wide-mouth tubes with ground-glass stoppers. Also put into each tube were methanol, the monomer, and a 2 wt % grafting solution of chloroform,<sup>6</sup> so that the fabric-to-liquor ratio was 1:40. The polyester solution was deaerated with bubbling nitrogen for 5 min. The grafted fabrics were removed from the reaction tube after irradiation to the desired dose. The homopolymer was extracted from the grafted fabrics with boiling water. The samples were then dried at 313 K in a vacuum oven to a constant weight. The degree of grafting was determined as the percentage increase in the weight with the following relationship:

$$\text{Degree of grafting} = 100[(W_g - W_0)/W_0]$$

where  $W_0$  and  $W_g$  are the weights of the initial and final grafted samples, respectively.

#### Dyeing procedure and CD measurements

A 1% stock dye solution was prepared by the pasting of the dye in acetic acid before the addition of the required distilled water. Aqueous dye solutions containing 2 wt % fabric dye were prepared from the dyestuffs at a fabric-to-liquor ratio of 1:50. The pH of the dye bath was adjusted, and the dyeing process was carried out in the presence of a 10 g/L dye solution, sodium sulfate, and a 0.1 g/L Sandozin (Sandoz, Basle, Switzerland) NIT solution as a wetting agent. The temperature of the dye bath was then raised to 363 K and kept constant for 1 h. After the dyeing, the samples were rinsed in hot water containing a non-



**Figure 1** Effect of pH on the CD of grafted PET fabric dyed with AV and RR basic dyes.

ionic detergent and in tap water and were allowed to dry. This dyeing procedure was applied to samples dyed at different pHs and GYs. The samples dyed at different concentrations and different temperatures for kinetic studies were rinsed in water before being heated at 323 K for 15 min.

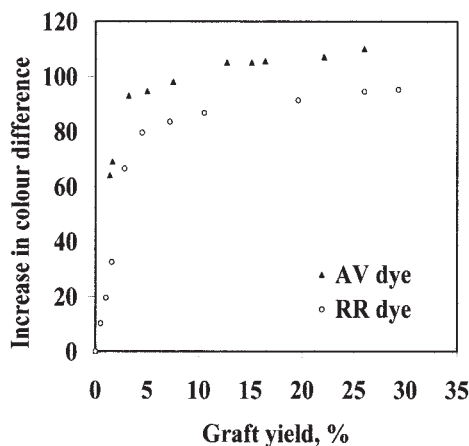
A computerized microcolorimeter unit made by Dr. Lange (Germany) was used for the color measurements. The  $L^*$ ,  $a^*$ , and  $b^*$  system used was based on the Commission International de l'Eclairage (CIE) color triangle (CIE units X, Y, and Z). In this system, the  $L^*$  value represents the dark-white axis,  $a^*$  represents the green-red axis, and  $b^*$  represents the blue-yellow axis. The  $L^*$ ,  $a^*$ , and  $b^*$  values of the grafted fabrics before dye sorption were measured and taken as references. The CD intensity of the grafted samples after dyeing was determined as follows:

$$\Delta E^* = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$$

## RESULTS AND DISCUSSION

### Effect of the pH

The effect of the pH of AV and RR dye solutions on CD of grafted PET fabrics is shown in Figure 1. The results indicate that both dyes yielded high color strengths in alkaline solutions. The affinity of the grafted fabrics toward the AV dye solution increased from CD/GY = 4.7 to CD/GY = 9.0 as the pH increased from 2.65 to 6.3. A further increase in the pH to 11.3 increased CD to 11.32. The CD/GY ratio for fabrics dyed in RR solutions increased from 0.9 to 5.7 as the pH increased from 3.49 to 6.3. A sudden increase in CD/GY to 11.43 was obtained



**Figure 2** Effect of the degree of grafting on the CD of PET fabric dyed in AV and RR dye solutions adjusted to pH 11.5.

as the pH increased to 11.5. These results showed the higher affinity of the AV dye toward grafted PET fabrics in comparison with the RR dye, although the degree of grafting of the latter was almost twice that of the AV dye. Consequently, all the dyeing experiments were carried out at a pH as close as possible to 11.5 for both dyes.

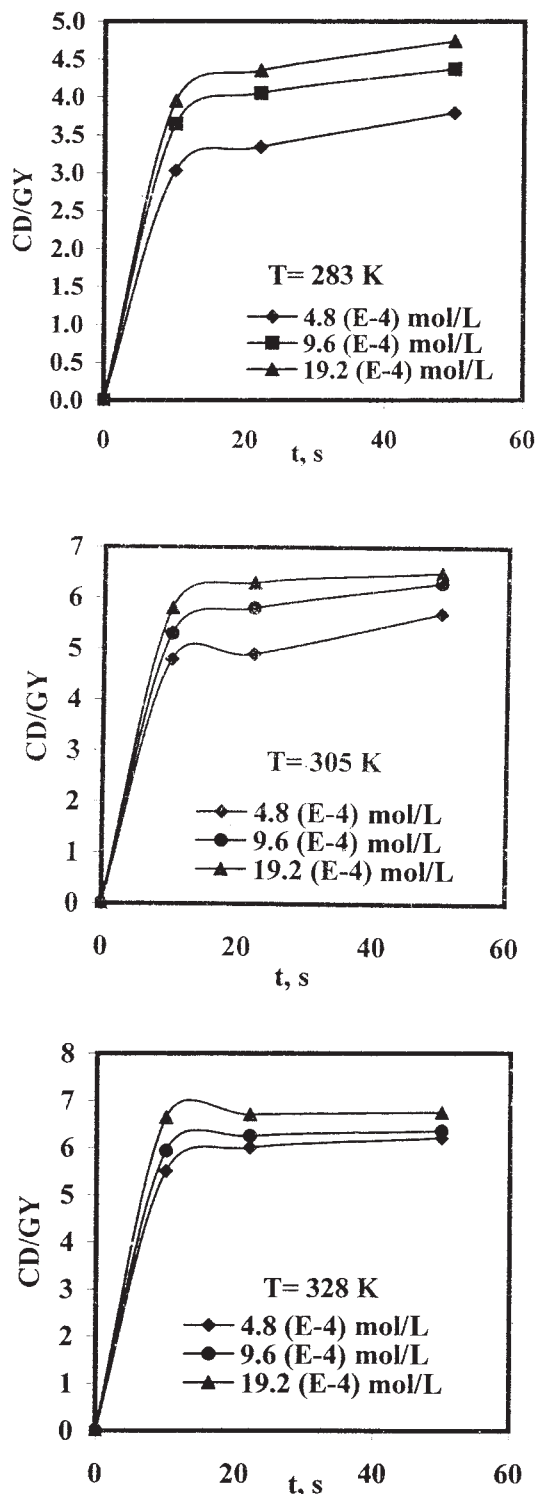
#### Effect of the degree of grafting

The effect of the degree of grafting (GY) on the CD of PET fabrics dyed in AV and RR dye solutions is shown in Figure 2. The results showed a fast linear increase in CD with an increase in GY up to  $\Delta E^*$  values of 75 and 69 for the AV and RR dyes, respectively. A further increase in GY produced a decrease in the rate of change of the color strength with the change in the degree of grafting, with a tendency to level off at GYs higher than 10.5%. These results indicated that dyeing grafted PET fabrics in AV dye solutions produced CD values higher than those for RR solutions with the same degree of grafting. This supported the results presented in Figure 1 and emphasized the higher affinity of grafted PET fabrics toward the AV dye in comparison with the RR dye. Moreover, the best dyeing conditions were achieved at GYs much lower than 10%. Consequently, the studies on the dyeing kinetics of grafted fabrics in AV and RR dye solutions were carried out at pH 11.5 and on samples with GYs of approximately 5%.

#### Dyeing kinetics

In the kinetic study, the CD of the dyed polyester fabrics was measured by the  $\Delta E^*$  value per unit of GY to avoid the contribution of the degree of grafting in the CD measurements. Preliminary experiments were carried out to study the dependence of the CD on the dyeing time. Dyeing time intervals ranging from 1 to

10 min were tried, and the corresponding CD values per unit of GY were measured. All the samples gave almost the same CD value. Consequently, intervals between 10–30 s were used to measure the dyeing time. In each experiment, grafted and ungrafted sam-



**Figure 3** Dependence of CD of grafted PET fabric on the dyeing time ( $t$ ) for different concentrations of AV dye solutions adjusted to pH 11.5 at 283, 305, and 328 K.

**TABLE I**  
Dependence of  $R_d$  [(CD/GY) $\text{min}^{-1}$ ] on  $C$ , and  $T$ , for AV Dye Solutions Adjusted to pH 11.5

	C (mol/L)		
	$4.8 \times 10^{-4}$	$9.6 \times 10^{-4}$	$19.2 \times 10^{-4}$
$R_d$ at 283 K	25.12	28.85	33.03
$R_d$ at 305 K	28.81	31.80	35.56
$R_d$ at 328 K	33	35.30	40.27

ples were dyed together in the same dye bath, and the net increase in CD per unit of GY was calculated.

AV dye

The dependence of CD/GY of grafted PET fabrics on the dyeing time for  $C$  values of  $4.8 \times 10^{-4}$ ,  $9.6 \times 10^{-4}$ , and  $19.2 \times 10^{-4}$  mol/L is shown in Figure 3 for AV dye solutions adjusted to pH 11.5 and dyed at 283, 305 and 328 K. The general features of the curves are nearly the same, except that the color strength increases with increasing  $C$  and  $T$ . The color strength increases linearly as the dyeing time increases up to about 10 s; this is followed by a slower increase with a tendency to level off at 50 s. The initial  $R_d$  value was calculated from the slope of the linear part of the color-strength/dyeing-time relationship. Table I shows the dependence of  $R_d$  on  $C$  and  $T$  for AV dye solutions.

The results show that as  $C$  increased,  $R_d$  increased. The relative rates of dyeing ( $R_{9.6 \times 10^{-4}}/R_{4.8 \times 10^{-4}}$ ) at concentrations of  $9.6 \times 10^{-4}$  and  $4.8 \times 10^{-4}$  mol/L and at 283, 303, and 328 K were nearly constant and independent of  $T$ , with an average value of 1.12. The ratio was approximately the same even when  $C$  was doubled from  $9.6 \times 10^{-4}$  and  $19.2 \times 10^{-4}$  mol/L. This indicated that doubling  $C$  did not affect the  $R_d$  ratios as given. Consequently,  $n$  had to be calculated.

It is known that the initial  $R_d$  value is related to  $C$  according to the following equation:

$$R_d = k_d C^n \tag{1}$$

Taking the logarithm of both sides of eq. (1) gives

$$\log R_d = \log k_d + n \log C \tag{2}$$

The plot of  $\log R_d$  versus  $\log C$  produces a straight line, the slope of which is  $n$  and the intercept of which yields  $\log k_d$ . Figure 4 shows logarithmic plots of  $R_d$  versus  $C$  at different values of  $T$ , as given in Table I. The relationship is linear, and the equations relating the different parameters, as displayed on the computer chart, are as follows:

$$(\log R_d)_{283\text{ K}} = 0.1379 \log C + 1.8653 \tag{3}$$

$$(\log R_d)_{305\text{ K}} = 0.1412 \log C + 1.9284 \tag{4}$$

$$(\log R_d)_{328\text{ K}} = 0.1428 \log C + 1.9890 \tag{5}$$

Equations (3)–(5) show that the values of  $n$  are almost constant and independent of  $T$ , with an average value of 0.14. The values of the intercepts yield  $\log k_d$ . Consequently, the  $R_d$  dependence on  $C$  can be written as follows:

$$(R_d)_{\text{AV dye}} = k_d C^{0.14} \tag{6}$$

The values of  $k_d$  for the different  $T$  values were calculated from the intercepts of eqs. (3)–(5) and are given in Table II. The results showed that  $k_d$  increased with an increase in  $T$ .

It is interesting to compare the CD/GY values under different dyeing conditions, as given in Figure 3, with those obtained from the initial slopes of CD versus GY, as presented in Figure 2. The values con-

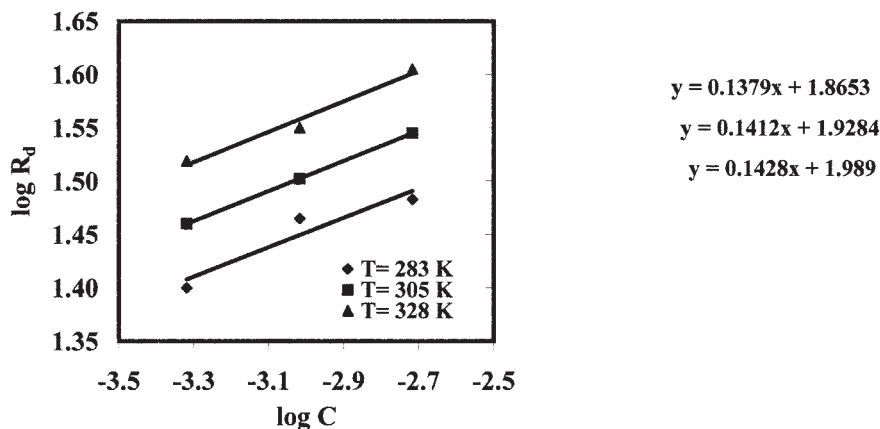


Figure 4 Logarithmic plots of  $R_d$  versus  $C$  at different  $T$  values for the AV dye.

**TABLE II**  
**Values of  $K_d$  [(CD/GY)min<sup>-1</sup>] of the AV Dye at Different  $T$  (K) Values**

	$T$		
	283	305	328
$k_d$	70.81	83.4	95.3

sidered were those obtained at  $C = 4.8 \times 10^{-4}$  mol/L, which was almost equal to the  $C$  value corresponding to 2% of weight of fabric (owf), in a 1:50 fabric-to-liquor ratio. The CD/GY values obtained under different dyeing conditions (Fig. 3) indicated an increase in CD/GY from 3.1 to 4.79 as  $T$  increased from 283 to 303 K for samples dyed for 10 s. The corresponding value for samples dyed at 363 K for 3600 s, as obtained from Figure 2, was 16, indicating that 19–30% of the CD was obtained at such low  $T$  values and extremely short dyeing times.

RR dye

The dependence of the CD of the grafted PET fabric on the dyeing time for  $C$  values of  $4.8 \times 10^{-4}$ ,  $9.6 \times 10^{-4}$ , and  $19.2 \times 10^{-4}$  mol/L is shown in Figure 5 for RR dye solutions adjusted to pH 11.5 and dyed at 305, 328, and 353 K. The general features of the curves are the same as those for the AV dye, except that the corresponding CD/GY values are much lower. The initial  $R_d$  values were calculated at the corresponding  $T$  values and are given in Table III. The results indicate that as  $C$  increased,  $R_d$  increased. The relative value of  $R_d$  at the corresponding  $C$  and  $T$  values was constant with an average value of 1.14 as  $C$  was doubled, as shown in Table III. The results show that the relative values of  $R_d$  were independent of  $T$  and were similar to those for the AV dye. This indicates that the order of the two basic dyes was very close.

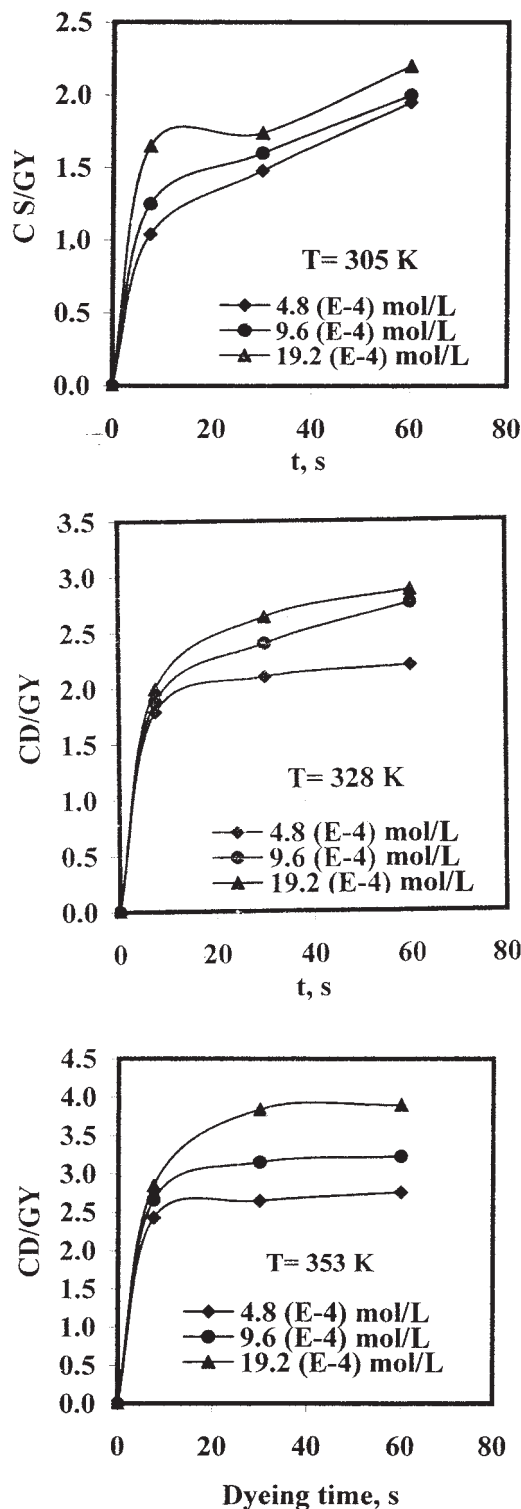
Logarithmic plots of  $R_d$  versus  $C$  at different values of  $T$  are shown in Figure 6. The relationship was linear, and  $n$  was independent of  $T$  and yielded an order of 0.14. The values of  $\log k_d$  at 305, 328, and 353 K were 1.397, 1.568, and 1.734, respectively. Consequently, the equation relating  $R_d$  to  $C$  for the RR dye could be presented as follows:

$$(R_d)_{RR \text{ dye}} = k_d C^{0.14} \tag{7}$$

The values of  $k_d$  for the RR dye at different  $T$  values were calculated as explained before for the AV dye. The values of  $k_d$  at the corresponding  $T$  values are given in Table IV. The results in Table IV show that  $k_d$  increased with an increase in  $T$ .

The CD/GY values, similar to those reported for the AV dye, were calculated from Figures 2 and 5 at  $C = 4.8 \times 10^{-4}$  mol/L and at different  $T$  values for RR.

The results indicate that 11–16% of the color strength of the samples dyed at 363 K for 3600 s was obtained for samples dyed at temperatures ranging from 305 to 328 K and at a dyeing time of 10 s.



**Figure 5** Dependence of CD of grafted PET fabrics on the dyeing time ( $t$ ) for different concentrations of RR dye solutions adjusted to pH 11.5 at 305, 328, and 353 K.

**TABLE III**  
**Values of  $R_d$  [(CS/GY)min<sup>-1</sup>] at Different  $C$  (mol/L)**  
**Values and  $T$  Values for RR Dye Solutions**  
**Adjusted to pH 11.5**

	C		
	$4.8 \times 10^{-4}$	$9.6 \times 10^{-4}$	$19.2 \times 10^{-4}$
$R_d$ at 305 K	8.34	10.02	12.3
$R_d$ at 328 K	12.59	14.13	16.02
$R_d$ at 353 K	19.44	21.3	23.28

**TABLE IV**  
**Values of  $k_d$  [(CD/GY)min<sup>-1</sup>] of the RR Dye**  
**at Different Values of  $T$  (K)**

	T		
	305	328	353
$k_d$	26.8	37.52	52.4

**Activation Energy  $Q$**

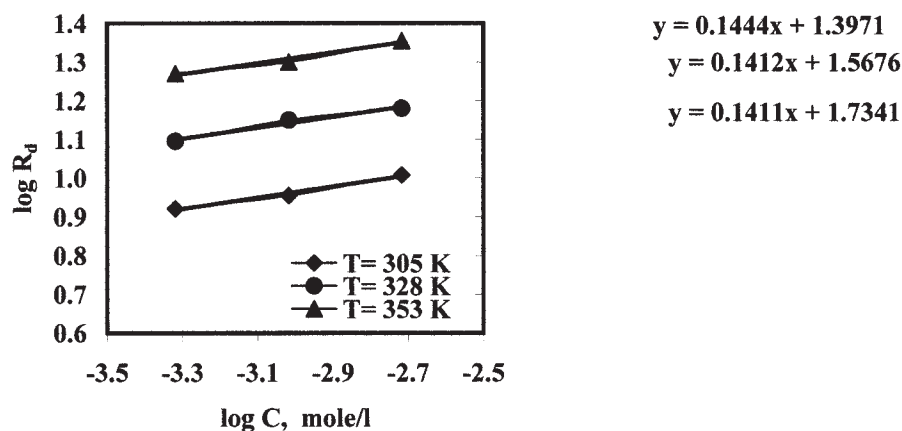
The results given in Tables II and IV for the dependence of  $k_d$  for the AV and RR dyes on  $T$  indicate that  $k_d$  increased with an increase in  $T$ . Moreover, the values of  $k_d$  for the AV dye were about 2.8 times higher than those for the RR dye for the same  $T$  value. The dependence of  $k_d$  on  $T$  is presented by an Arrhenius-type plot of the natural logarithm of  $k_d$  versus the inverse of  $T$  in Figure 7. The relationship is linear, and the slope gives the value of  $Q/R$ , where  $R$  is the universal gas constant.  $Q$  values of 4.9 and 13.8 kJ/mol were calculated for the AV and RR dyes, respectively. The intercept of the relationship is the natural logarithm of the pre-exponential rate constant ( $A$ ). The values of  $\ln A$  for the AV and RR dyes were 6.364 and 8.705, respectively. The corresponding values of  $A$  were 566 and 6035 (CD/GY)min<sup>-1</sup>. The general equation relating  $k_d$  to  $A$ ,  $T$ , and  $Q$  for the AV and RR dyes is

$$(k_d)_{AV \text{ dye}} = 9.43 e^{-4.9(\text{kJ/mol})/RT} (\text{CD/GY})\text{s}^{-1} \quad (8)$$

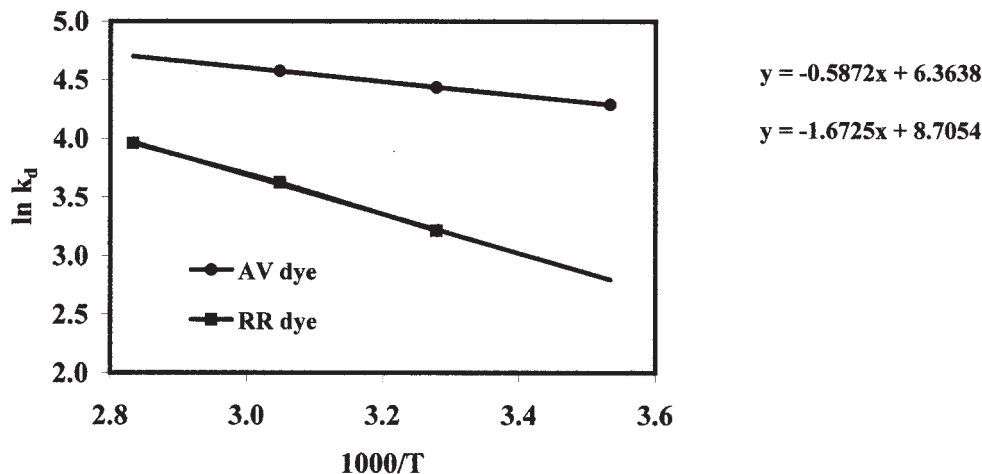
$$(k_d)_{RR \text{ dye}} = 100.6 e^{-13.8(\text{kJ/mol})/RT} (\text{CD/GY})\text{s}^{-1} \quad (9)$$

The results obtained for the AV and RR dyes showed that the order of the dyeing process was the same for both dyes and was independent of  $T$  or the type of dye. This indicates that the dyeing process for the

grafted PET fabrics with the basic dyes was of the same nature, although  $k_d$  for the AV dye was higher than that for the RR dye. The differences in  $R_d$  for the dyes could be attributed to the different molecular structures and molecular weights of the dyes. The molecular weight of the RR dye was 478.5, whereas that for the AV dye was 417. Moreover, the calculated values for  $Q$  of the two dyes (4.9 and 13.8 kJ/mol) were high enough to explain the reaction between the grafted polyester fabric and the basic dyes. This reaction took place at an extremely high rate and required an extremely small  $Q$  value. This suggested that the calculated rates and apparent activation energies were those for the diffusion of the dye molecules through grafted and dyed PET fabrics to reach grafted areas (dye sites) in the bulk of the fabric matrix. Because the diffusion of the dye and its subsequent reaction with grafted PET molecules were dependent processes, the slowest process (diffusion through grafted and dyed fabrics to reach dye sites) was rate-controlling. The small values of the rate constant for the RR dye, in comparison with those for the AV dye, and its high activation energy can, therefore, be attributed to the bigger structure (triphenyl and diphenyl for the AV and RR dyes, respectively) and higher molecular weight of the RR dye molecule. These factors were responsible for retarding the diffusion of the RR dye through the dyed and grafted PET fabric. Once the surface layer of the grafted fabric reacted with the dye, the newly formed F-COO-dye structure started to



**Figure 6** Logarithmic plots of  $R_d$  versus  $C$  at different  $T$  values for the RR dye.



**Figure 7** Arrhenius-type plot of the natural logarithm of  $k_d$  versus the inverse of the absolute temperature ( $1000/T$ ) for AV and RR dyes.

oppose the dye diffusion and slowed down the dyeing process. This explained the leveling off of the CD values after about 15 s of dyeing time. An increase in  $C$  increased the concentration gradient and hence increased the driving force for the diffusion process, which resulted in higher CD values at constant dyeing times. The diffusion process was temperature-dependent in a way similar to that given by an Arrhenius equation. The exponential dependence of  $k_d$  on the absolute temperature emphasized that the rate-controlling process was diffusion. The results also indicated that dyeing MAA-grafted PET fabrics with basic dyes could be carried out at temperatures lower than those usually applied in industry and at extremely short dyeing times. This considerably reduced the energy consumption for heating the dye bath and cut down the time necessary for dyeing the PET fabric.

### CONCLUSIONS

The increase in the pH of the dye solution considerably increased the CD of MAA-grafted polyester fabrics dyed with AV and RR basic dyes. The best dyeing was achieved at pH 11.5 for AV and RR aqueous solutions.

The CD of the grafted fabric increased rapidly with an increase in GY and leveled off as the degree of grafting exceeded 8% and the dyeing time passed 10 s.

The initial  $R_d$  value increased with an increase in  $C$  and  $T$  of the dye bath. The values of  $R_d$  for the AV dye were higher than those for the RR dye.

The dyeing process followed 0.14-order kinetics and was independent of  $T$  and the type of dye.

$k_d$  increased exponentially with an increase in  $T$  and followed an Arrhenius-type relationship.  $Q$  values of 4.9 and 13.8 kJ/mol and  $A$  values of 9.43 and 100.6 (CD/GY) $s^{-1}$  were calculated for the AV and RR dyes, respectively.

The mechanism of the dyeing process for the two dyes was diffusion-controlled, and the  $R_d$  values depended on the type of basic dye. Quite high  $R_d$ 's were found even at temperatures as low as 283 K (AV dye), and the completion of the dyeing process occurred in a matter of minutes. About 19–30% of the color strength of the samples dyed at 363 K for 1 h was obtained for the AV dye at dye bath temperatures ranging from 283 to 328 K and at a dyeing time of 10 s.

The relatively low  $T$  values and the extremely low dyeing times required to dye MAA-grafted PET fabrics with basic dyes cut the dyeing cost to a minimum and showed the importance of studying the dyeing kinetics for the economy of the process.

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