



Thermodynamic Sorption Parameters and Kinetics of Dyeing Disazo Dyes Derived from 4-aminobenzoic acid and 4-amino-3-nitrotoluene on Polyester Fibre and Polyamide Fibre

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Authors' contributions

This work was done in collaboration with all authors. Author JOO designed the study, carried out the synthesis, wrote the protocol; managed literature searches and wrote the first draft of the manuscript. Authors MKY and USA carried out the dyeing, plotting of the rate curves and interpreted them. All the authors read and approved the final manuscript.

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ABSTRACT

The thermodynamic and kinetic properties of disazo dyes on polyester fibre and polyamide (nylon) fibre were investigated. The disazo dyes showed high affinity values on the two substrates under study. Some of the affinity values obtained on polyester fabric are -10.25, -12.10, -9.80, -10.35, -14.15 Kj.mol^{-1} at 363K and those obtained from nylon fabric are -10.03, -10.16, -10.26, -10.04, -10.3 Kj.mol^{-1} at 343K for dyes I-V respectively. However, the affinity values of the dyes were slightly higher on polyester fibre than on nylon 6 fibre. It was also found that the enthalpy of dyeing, and entropy of dyeing values were slightly higher on polyester fibre than on the nylon 6 fibre. The enthalpy of dyeing values obtained on polyester fabric for dyes I-V are -838, -174.7, -15.9, -129.1, -741.8 Kj.mol^{-1} and those obtained on nylon 6 fabric for the same set of dyes are -223.4, -50.4, -65.9, -62.9, -88.0, Kj.mol^{-1} . The entropy values obtained are -203, -448, -100, -53, -317, -177 $\text{Jmol}^{-1}\text{k}^{-1}$ at 363k on polyester fabric and -613, -114, -158, -149, -221 $\text{Jmol}^{-1}\text{k}^{-1}$ at 343K on nylon 6 fibre. The activation energy of diffusion results showed that there was

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high barrier to the mobility of dye molecules within the nylon polymer matrix due to steric effects and hydrogen bond formation between some functional groups of the dyes and the nylon polymer. The results of this study indicated that the dye-fibre interactions are thermodynamically allowed, which described the suitability of these practical dye applications on the synthetic fibres.

Keywords: Thermodynamic parameters; disazo dyes; dyeing Kinetics, polyester fibre, polyamide fibre, activation energy.

1. INTRODUCTION

The amount of dye absorbed by a textile fibre at equilibrium when there is variation in temperature allows thermodynamic and kinetic sorption quantities such as standard affinity, heat of dyeing, entropy of dyeing and activation energy of diffusion to be evaluated [1-4]. The standard affinity, $\Delta\mu^0$ is the transfer of one mole of dye from its standard state in solution to its standard state in the fibre. The heat-content change during the dyeing process is the ΔH^0 , whereas the degree of disorder of the dye molecules during the dyeing process corresponds to the entropy change, ΔS^0 [5-7]. However, the mass transfer of dye molecules from the external aqueous phase to the surface and interior of the substrate and subsequent distribution of dye within the substrate up to its saturation equilibrium value correspond to the diffusion process. The thermodynamic and kinetics of dyeing are influenced by the structural features in both the dye and polymer [2,8-9].

In a study involving disazo disperse dyes derived from 1, 4-diaminobenzene and 4-amino-3-nitrotoluene, and from 4-aminophenol and 4-aminobenzoic acid on polyester and nylon 6 fibres [7], the thermodynamic absorption parameters were described. The results of the study revealed that the affinity values of the dye on polyester fibre were higher than those of the nylon 6 fibre. It was also found that the binding forces between the dye and the fibre were those of hydrogen bonding mechanism.

In another study [10], the thermodynamic analysis of 1, 4- diaminoathraquinone adsorption on polyethylene terephthalate, using many alkane media such as from pentane to decane, was reported. The authors established that the standard affinity $\Delta\mu^0$ and diffusion coefficient D of the dyes decreased with increasing number of carbon atoms while activation energy (E_D) increased with increasing number of carbon atoms.

In spite of the many studies carried out on the physical Chemistry of azo dyes, only a few studies describe the thermodynamic and kinetic sorption parameters of disazo disperse dyes when applied on polyester fibres and polyamide fibres. Most of the work carried out earlier in this area were based solely on monoazo disperse dyes [4]. Thus, the aim of the present study was to evaluate the practical suitability (in terms of thermodynamic and kinetics of dyeing) of a series of disazo disperse dyes on polyester and nylon. The dyes were previously prepared in [8].

2. EXPERIMENTALS

2.1 Preparation of Dye Dispersion

As described in the sub-section above, the dyes were previously prepared and formulated to be applied to polyester fibre and nylon 6 fibres. The dye dispersion is composed of a dispersing agent, Diwatex 40p (moderately sulphonated Kraft Lignin, 20% on the weight of the dye) and a solvent N, N- dimethylformamide (DMF). Each dye (1.0g) was dissolved in dimethylformamide and later made up to the mark (100ml) with the dispersing agent.

2.2 Materials

Plain weave nylon 6 fabric having 3696 ends/ m and 3307 picks/ m was used. One hundred percent polyester (Terylene) was also used in the dyeing.

2.3 Standard Affinity, Heat of Dyeing and Entropy of Dyeing

Sixteen identical dyebaths were prepared by the diazotization and coupling reactions of 4-amino phenyl azo-4-methyl-2-nitrobenzene derivatives and sixteen 0.04g polyester fabrics were used. Ten milliliters of dye liquor was placed in each dyebath to give a 10% shade with a liquor ratio 80:1. The pH of the dyeing system was adjusted to 5.5 using 2% acetic acid. The absorbance readings were recorded on a camspec spectrophotometer at a wavelength of 420nm. The dyebaths were placed in a water bath that was graduated to read up to 110°C. The polyester fabric (not pretreated) was entered into each dyebath and timing started when the temperature of the dye liquor equilibrated with that of the dyebath. Dyeing was allowed to continue for six hours. After dyeing, the concentration of dye $[C_f]$, remaining in the dyebath was measured. The dyed samples were rinsed thoroughly with deionized water. After complete drying, the dyed samples were placed in a thimble for extraction using formic acid (50%). The concentration of extracted dye, $[C_i]$ was measured using spectrophotometric method. The procedure above, was repeated for the dyeing and stripping of nylon 6 fabrics at 353⁰k and 363⁰k respectively at pH 4 adjusted with 2% acetic acid. The standard affinity, $\Delta\mu^0$, (K.Jmo1⁻¹) measured at 363K and 373K was estimated using Eqn 1 [11-13].

$$\Delta\mu^0 = -RT \ln \frac{[C_f]}{[C_s]} \quad (1)$$

Where R is the gas constant (8. 317 JK⁻¹mol⁻¹), T is the absolute temperature (K), $[C_f]$, the concentration of dye in the fibre at equilibrium (g kg), (C_s) , the concentration of dye in the solution at equilibrium (gl⁻¹). The heat of dyeing, ΔH^0 (KJ.mol⁻¹) was calculated using Eqn 2 [14].

$$\Delta H^0 = \frac{T_2 \Delta\mu_1^0 - T_1 \Delta\mu_2^0}{T_2 - T_1} \quad (2)$$

Where T_1 and T_2 are the absolute temperatures 1 and 2 and $\Delta\mu_1^0$ and $\Delta\mu_2^0$ (KJ.mo1⁻¹) are the standard affinities at T_1 and T_2 respectively.

Eqn 3 was finally used to estimate the entropy of dyeing, ΔS^0 (Jmol⁻¹) on sorption [15].

$$\Delta\mu^0 = \Delta H^0 - T\Delta S^0 \quad (3)$$

2.4 Diffusion (Sorption) Coefficient and Activation of Energy Diffusion

Sixteen identical dyebaths were prepared in a manner similar to those described in section 2.3. Sixteen nylon 6 samples (0.05g) were used. Dye dispersion was placed in each dyebath to give 20% shade at a liquor ratio 70:1. The dyeing system was adjusted to pH 4.0 using 2% acetic acid. The nylon 6 fabrics were entered into each dyebath set at 343K and 353K respectively. Constant agitation was applied during the dyeing process. The timing started when the temperature of the dye liquor equilibrated with that of the dyebath and the first sample was removed from the dyebath at intervals of 10mins and the last sample was removed after six hours of dyeing. Each dyed sample was washed with warm water and allowed to dry at room temperature. The concentrations of the dyes were measured after stripping the dyed samples with formic acid at λ_{\max} (400nm) in a 10mm quartz absorption cell campsec UV-vis spectrophotometer. The various absorbance at 10, 20, 30, 40, 50, 60, 70, 80, 90 and 100mins were $[C_1]$, and the absorbance after six hours of dyeing were $[C]_{\infty}$. All measurements of dye solution were conducted at room temperature and the rate curves of C_1/C_{∞} against $t^{1/2}$ were plotted from where the slopes were obtained. The values of diffusion coefficients (D) were estimated using Eqn 4 [12].

$$D = \frac{[C_1] \pi r^2}{[C_{\infty}] 16} \quad (4)$$

And the values of the activation energy of diffusion (E_D) were calculated in accordance with Eqn 5 [3].

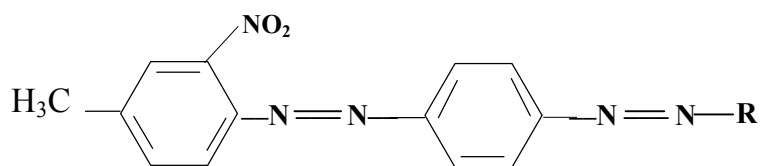
$$E_D = \left[R \left/ \frac{1}{T_1} - \frac{1}{T_2} \right. \right] \ln \left[\frac{D_1}{D_2} \right] \quad (5)$$

Where T_1 and T_2 are the absolute temperatures 1 and 2, R is the universal gas constant ($8.317 \text{ JK}^{-1} \text{ mol}^{-1}$), E_D is the activation energy of diffusion (Jmol^{-1}) and D_1 and D_2 are the diffusion coefficients at the two temperatures T_1 and T_2 . It must be noted that the difference in the temperatures for the dyeing of the two different fibres is due to the fact that polyester has a more crystalline structure compared to nylon with less amorphous regions. Thus, polyester fibre requires higher temperatures for dyeing to occur than nylon fibre. It should also be noted that only the kinetic sorption parameters of nylon fibre were carried out.

3. RESULTS AND DISCUSSION

3.1 Thermodynamic Parameters

The results of the thermodynamic studies including the standard affinity, enthalpy of dyeing, and entropy of dyeing are summarized in Tables 1, 2, 3 and 4. Fig. 1 shows the structures of the dyes while Figs. 2 to 17 give the representative rate curves of dye I to XVI on nylon respectively. The dyeing affinity values of the dye analogues on nylon 6 fabrics and polyester fabrics were found to be generally lower at higher temperature (373K) than at lower temperature (363K).



Dye	R
i.	
ii.	
iii.	
iv.	
v.	
vi.	
vii.	
viii.	
ix.	
x.	

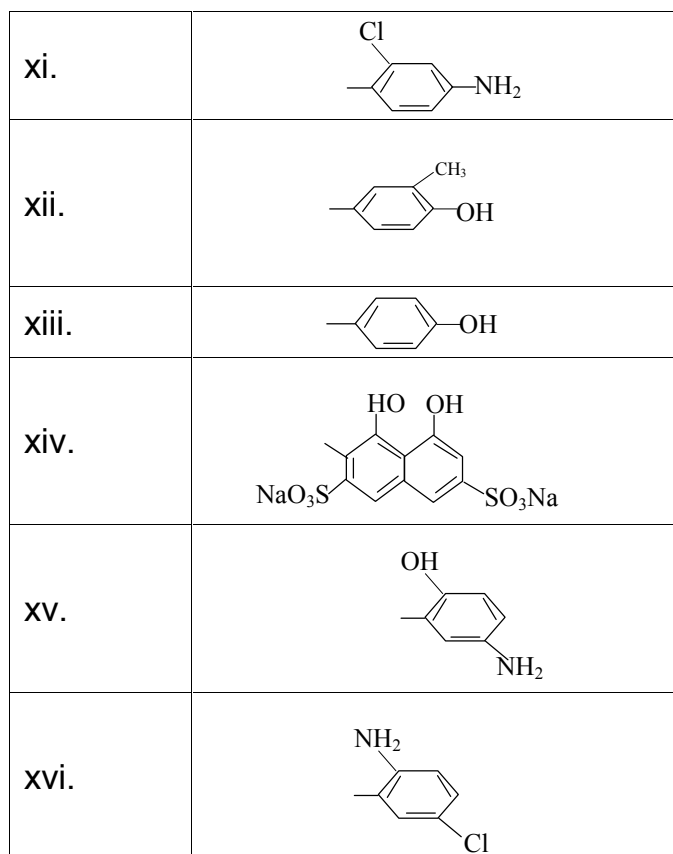


Fig. 1. Dye Structures

Table 1. Affinity values of dyes (I-XVI) on polyester fabric

Dye	$\Delta\mu^0(\text{KJ.mol}^{-1})$	
	363K	373K
I	-10.25	-8.22
II	-12.10	-7.62
III	-9.80	-8.80
IV	-10.35	-9.82
V	-14.15	-10.98
VI	-10.08	-8.31
VII	-8.52	-7.16
VIII	-13.41	-7.43
IX	-10.02	-8.01
X	-9.90	-8.35
XI	-8.75	-7.89
XII	-12.83	-8.45
XIII	-9.76	-9.19
XIV	-9.84	-8.84
XV	-12.73	-6.23
XVI	-9.76	-9.34

This is in agreement with earlier findings [14] that dye affinities decrease with increasing temperatures. The affinity values as shown in Tables 1 and 2 for the two fibres are large and negative which indicate that the dyes have high affinities for the fibres under study. It was also observed that dye XIV exhibited the highest affinity value of $-14.07 \text{ KJ.mol}^{-1}$ on nylon 6 fibres, whereas the same dye gave affinity value of -9.84 KJmol^{-1} on polyester fibre. The reason for this is that dye XIV contained the salt group (SO_3Na) in the structure. Similarly, dye VII gave higher affinity value of $-10.84 \text{ KJ mol}^{-1}$ on nylon 6 fibre than that of $-8.52 \text{ KJ.mol}^{-1}$ on polyester fibre due to the presence of the sulphonate group ($-\text{SO}_3\text{Na}$) in the dye structure.

Table 2. Affinity ($\Delta\mu^0$) values of dyes (I-XVI) on nylon fibre

Dyes	$\Delta\mu^0 (\text{KJ.mol}^{-1})$	
	343K	353K
I	-10.03	-9.04
II	-10.16	-9.02
III	-10.26	-8.68
IV	-10.04	-8.55
V	-10.13	-7.93
VI	-9.46	-7.38
VII	-10.84	9.08
VIII	-12.93	-12.70
IX	-10.50	-7.89
X	-9.88	-7.82
XI	-10.20	-8.51
XII	-9.90	-9.27
XIII	-9.97	-7.63
XIV	-14.07	-9.25
XV	-11.45	-4.86
XVI	-10.28	-4.83

Table 3. Thermodynamic sorption parameters of dyes (1-XVI) on polyester fabric

Dye	$\Delta H^0 (\text{KJ.mol}^{-1})$	$\Delta S^0 (\text{Jmol}^{-1} \text{K}^{-1})$	
	$\Delta\mu^0 (\text{KJ.mol}^{-1})$	363K	373K
I	-83.8	-203	-203
II	-174.7	-448	-448
III	-45.9	-100	-100
IV	-29.7	-53	-53
V	-129.1	-317	-317
VI	-741.8	-177	-177
VII	-57.8	-136	-136
VIII	-230.5	-598	-598
IX	-82.8	-201	-201
X	-66.3	-155	-155
XI	-40.0	-86	-86
XII	-171.8	-438	-438
XIII	-30.6	-57	-57
XIV	-46.0	-100	-100
XV	-248.7	-650	-650
XVI	-25.0	-42	-42

These suggest that dyes containing acidic groups have higher affinity for the polyamide fibres than for the polyester fibres [15]. This is the reason why polyester fibres are best coloured by disperse dyes. In general, the high affinity values of the dyes on the two

substrates suggest that they have high hydrophobicity which has been found to increase affinity [16]. Since the calculated thermodynamic sorption parameters obtained indicated that the dye-fibre combination process gave negative values of standard affinity, $\Delta\mu^0$. The dye-fibre interactions is, therefore, thermodynamically favorable.

Table 4. Thermodynamic sorption parameters of dyes (1- XVI) on nylon fibre

Dye	R ²		ΔH^0 (KJ.mol ⁻¹)	ΔS^0 (Jmol ⁻¹ K ⁻¹)	
	343K	353K	-223.4	343K	353K
I	0.8471	0.8695	-50.4	-613	-613
II	0.9130	0.9790	-65.9	-114	-114
III	0.8936	0.9513	-62.9	-158	-158
IV	0.8372	0.9022	-88.0	-149	-149
V	0.8740	0.9575	-83.0	-221	-221
VI	0.9195	0.9215	-72.9	-208	-208
VII	0.8401	0.8490	-20.8	-176	-176
VIII	0.8630	0.8731	-102.5	-22	-22
IX	0.8432	0.8545	-82.6	-261	-261
X	0.7256	0.8511	-69.7	-206	-206
XI	0.7738	0.8344	32.0	-169	-169
XII	0.8361	0.9141	-92.4	-82	-82
XIII	0.8006	0.8102	-184.3	-234	-234
XIV	0.6860	0.9235	-244.2	-482	-482
XV	0.8348	0.8776	-202.9	-659	-659
XVI	0.6757	0.8248		-526	-546

The changes in the heat-content (enthalpy) of the system when dye is sorbed are summarized in Tables 1 and 2 for the two fibres, and they show large negative values of enthalpy. This corresponds to the high potential affinities of the dyes on the nylon 6 fibre and polyester fibre. The ΔH^0 values varied from -25.0KJ.mol^{-1} to -248.7KJ.mol^{-1} on polyester fibre and from -20.8KJ.mol^{-1} to -244.2KJ.mol^{-1} on nylon 6 fibre. The changes in enthalpy values found for polyester fibre were generally slightly higher than those found for nylon 6 fibre. In addition, enthalpy values were negative which suggested that dyeing process is exothermic hence, raising the temperature leads to lower potential affinity and less dye being absorbed at equilibrium [17-18].

The entropy values for the sorption of the dyes into nylon 6 fibre were found to be between $-659\text{Jmol}^{-1}\text{K}$ and $-22\text{Jmol}^{-1}\text{K}$ and between $-650\text{Jmol}^{-1}\text{K}$ and $-42\text{Jmol}^{-1}\text{K}$ for polyester fibre. This indicated that the entropies of dyeing of the polyester fibre are slightly higher than those determined for the nylon fibre. The negative entropy (ΔS^0) values suggested that the randomness of dye molecules decrease at the solid/solution interface during sorption of the dyes. This could also explain the reason for the decrease in diffusion rate of the dyes. The values in Tables 1 and 2 showed that the sorption of each dye by the substrates is a spontaneous and exothermic process [19].

4. Diffusion Coefficient (D) and Activation Energy of Diffusion (E_D)

The results in Table 5 represent the diffusion coefficient of the dyes on nylon 6 fibre at 343K and 353K. And the results show that the diffusion of dyes increased as the temperature was raised from 343K to 353K. Thus, diffusion of dyes occurred by random motion from regions of high concentration to regions of lower concentration in the polymer matrix on raising the temperature.

Table 5. Diffusion coefficient (d) and activation energy of diffusion data of dyes on nylon 6 fabrics

Dye sample	D (cm ² / min) at		Activation energy (E _D KJ/mol ⁻¹)	Molecular Weight (Cal)
	343K	353K		
I	2.059 x 10 ⁻⁵	2.525 x 10 ⁻⁵	2.0524 x 10 ⁻⁴	390.4
II	3.878 x 10 ⁻⁵	5.676 x 10 ⁻⁵	3.8343 x 10 ⁻⁴	394.8
III	2.071 x 10 ⁻⁵	4.674 x 10 ⁻⁵	8.1937 x 10 ⁻⁴	390.4
IV	6.350 x 10 ⁻⁵	6.872 x 10 ⁻⁵	7.9519 x 10 ⁻³	377.4
V	4.046 x 10 ⁻⁵	4.517 x 10 ⁻⁵	1.1094 x 10 ⁻⁴	411.4
VI	1.825 x 10 ⁻⁵	2.355 x 10 ⁻⁵	2.5656 x 10 ⁻⁴	410.4
VII	1.926 x 10 ⁻⁵	8.242 x 10 ⁻⁵	1.4636 x 10 ⁻⁵	512.5
VIII	3.285 x 10 ⁻⁵	4.585 x 10 ⁻⁵	3.3548 x 10 ⁻⁴	375.4
IX	5.314 x 10 ⁻⁵	6.251 x 10 ⁻⁵	1.6350 x 10 ⁻⁴	376.4
X	1.583 x 10 ⁻⁵	1.630 x 10 ⁻⁵	3.5058 x 10 ⁻³	411.4
XI	1.953 x 10 ⁻⁵	2.131 x 10 ⁻⁵	8.7800 x 10 ⁻³	394.8
XII	2.124 x 10 ⁻⁵	1.077 x 10 ⁻⁴	1.6342 x 10 ⁻⁴	375.4
XIII	4.072 x 10 ⁻⁶	1.429 x 10 ⁻⁵	1.2636 x 10 ⁻⁴	361.4
XIV	2.525 x 10 ⁻⁵	2.935 x 10 ⁻⁵	1.5151 x 10 ⁻⁴	631.5
XV	3.291 x 10 ⁻⁵	1.002 x 10 ⁻⁵	1.1211 x 10 ⁻⁵	376.4
XVI	2.293 x 10 ⁻⁵	5.021 x 10 ⁻⁵	7.9111 x 10 ⁻⁴	394.8

The results of the activation energy of diffusion (E_D) of the dyes on nylon 6 fibre are summarized in Table 3 and show that the amount of energy required to assist the dye in entering the fibre in order to overcome the surface barrier of the substrate is 7.919 x10⁻³ KJ.mol⁻¹ for dye IV and a value of 8.7800 x 10⁻³ KJ.mol⁻¹ for dye XI. The difference between the values indicates that there is a barrier to the diffusion of each dye in the polymer matrix which could be due to many factors in a conventional aqueous dyeing system. Such factors are steric factors or effects connected with the molecular size of the dye, which means that smaller molecules have higher mobility than larger molecules [19]. Apart from the aforementioned factors, the possible interactions between the dye molecules and the polymer matrix can have an important role in the diffusion process. For instance, the H-bonds formation between some functional groups of the dye such as -OH, -NH₂ and -OCH₃ and the polymer may be attributed to the high dye affinity values obtained in this study. This bond formation may also reduce the dye mobility in the polymer matrix and hence slow down the rate of diffusion of the dye molecules into and within the substrate. It was also observed from Table 5 that the higher the value of the activation energy of diffusion, the higher the diffusion coefficient for a given temperature gradient. This means that the higher the activation energy the higher the barrier to diffusion of dye molecules in the polymer matrix. Thus, dyes iii, iv, xi, and xvi which exhibited higher activation energy of diffusion values than the others, may have experienced considerable amount of energy barrier to push between absorbing sites. On the other hand, it was also observed that dye vii showed the second least activation energy of diffusion value yet it had the second largest molecular size of all the dyes. The reason for this may be due to the fact that it had a more planar shape and thus encountered fewer barriers in moving between absorbing sites. It has also been found out [20] that there is inverse relation between dye substantivity and diffusion rates in the fibre and vice versa. Thus, these dyes under investigation show low diffusion rates in the nylon 6 fibre, which suggests that they have high substantivity.

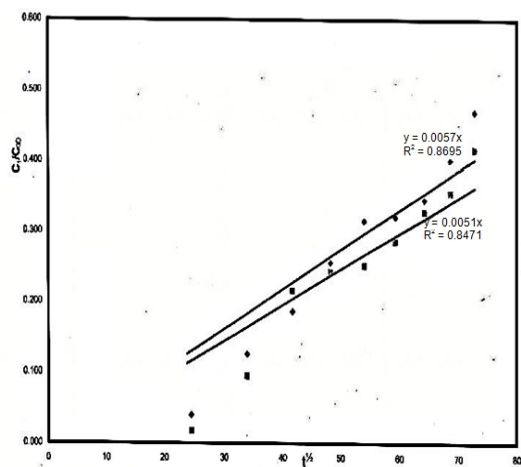


Fig. 2. Rate curves of Dye I on mylon

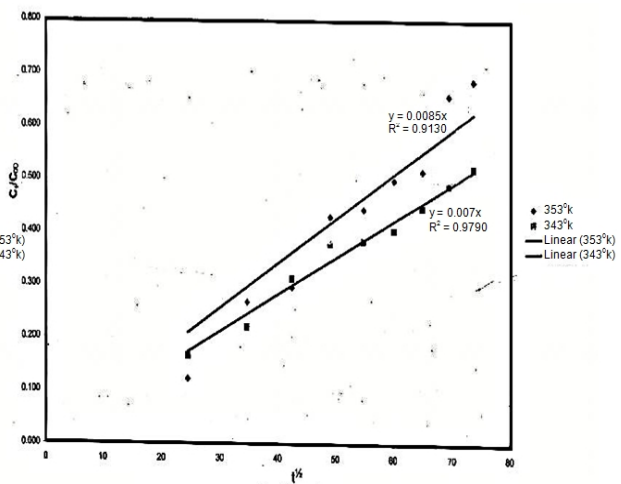


Fig. 3. Rate curves of Dye II on mylon

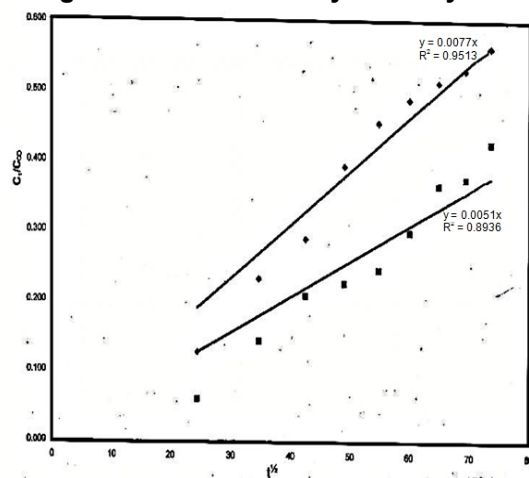


Fig. 4. Rate curves of Dye III on mylon

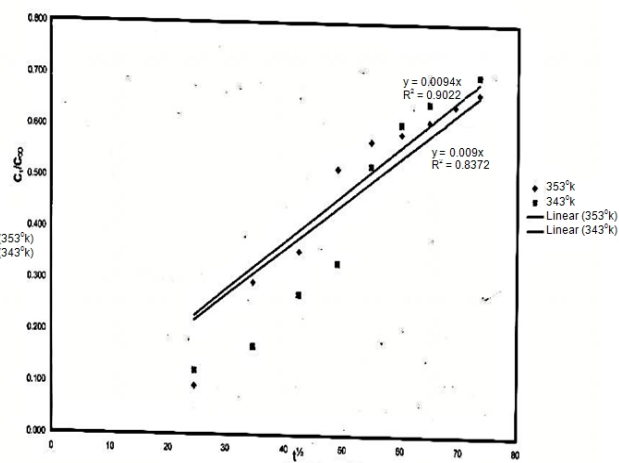


Fig. 5. Rate curves of Dye IV on mylon

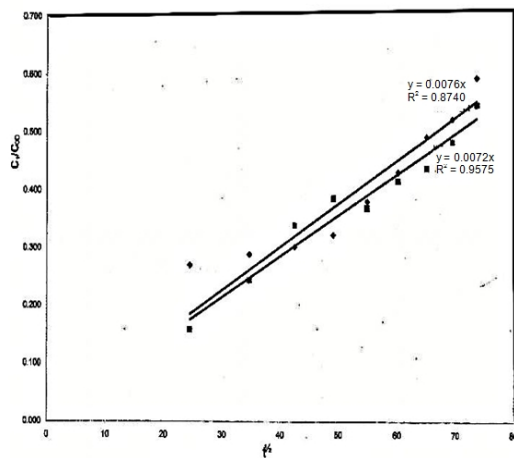


Fig. 6. Rate curves of Dye V on mylon

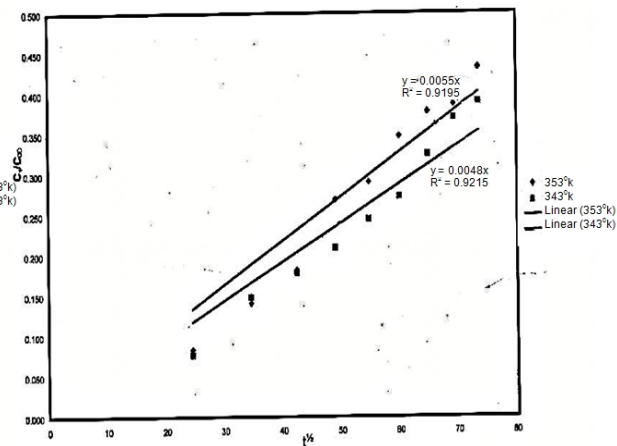


Fig. 7. Rate curves of Dye VI on mylon

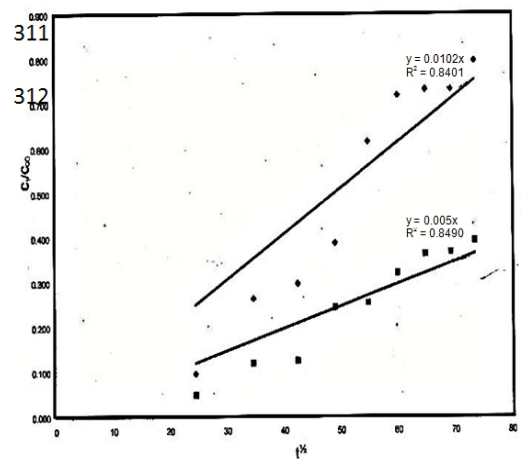


Fig. 8. Rate curves of Dye VII on mylon

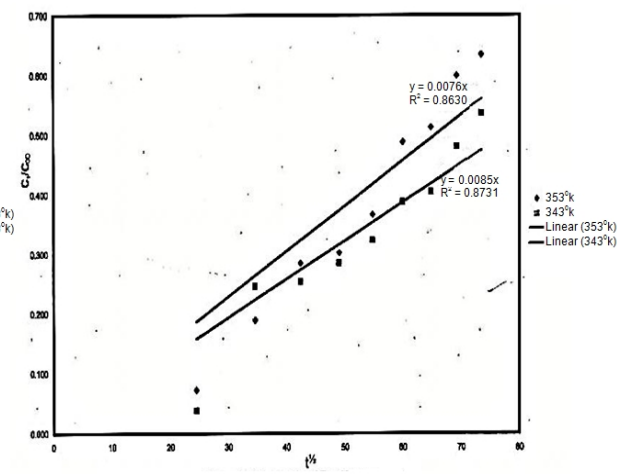


Fig. 9. Rate curves of Dye VIII on mylon

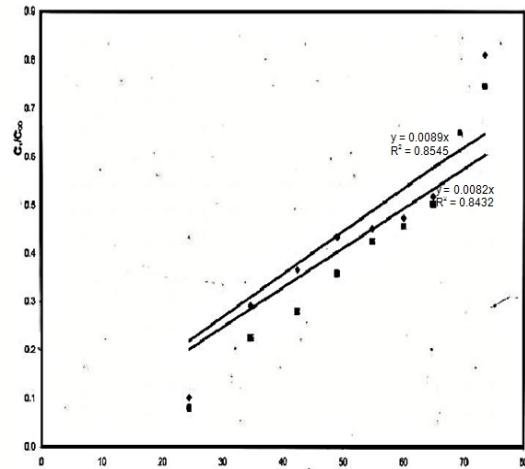


Fig. 10. Rate curves of Dye IX on mylon

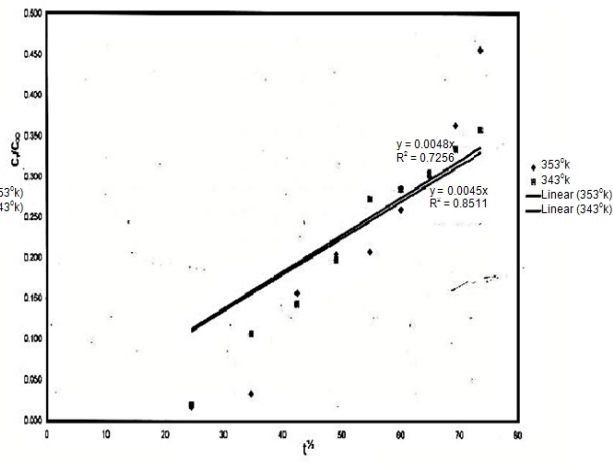


Fig. 11. Rate curves of Dye X on mylon

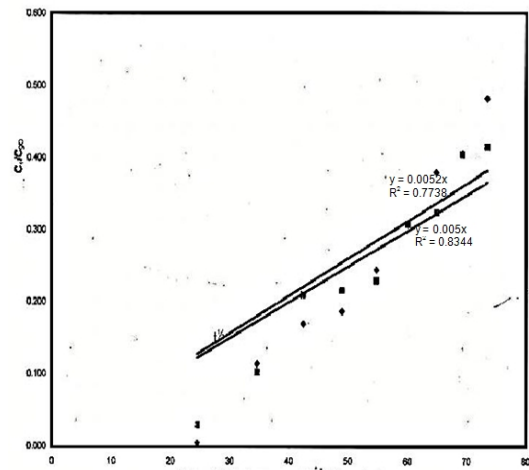


Fig. 12. Rate curves of Dye XI on mylon

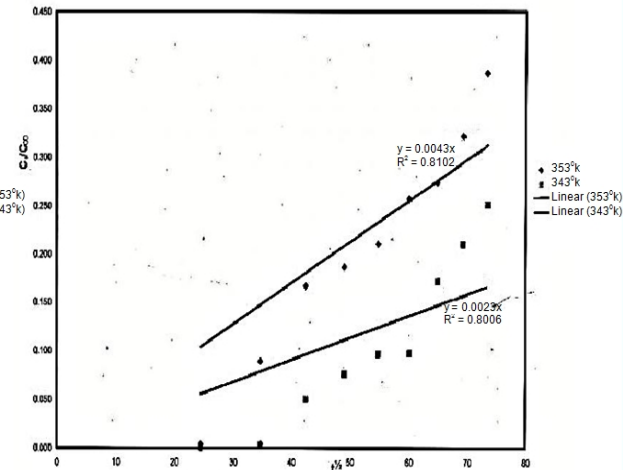


Fig. 13. Rate curves of Dye XII on mylon

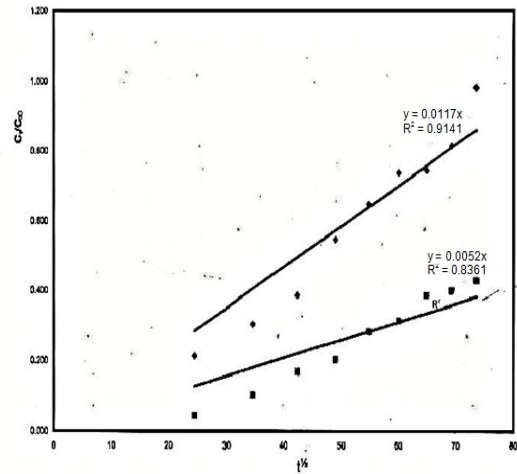


Fig. 14. Rate curves of Dye XIII on mylon

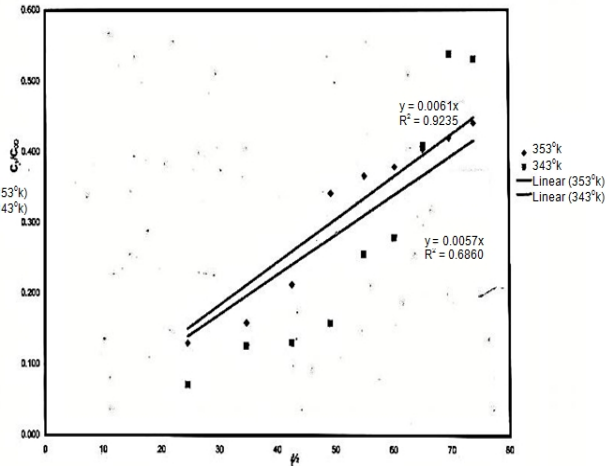


Fig. 15. Rate curves of Dye XIV on mylon

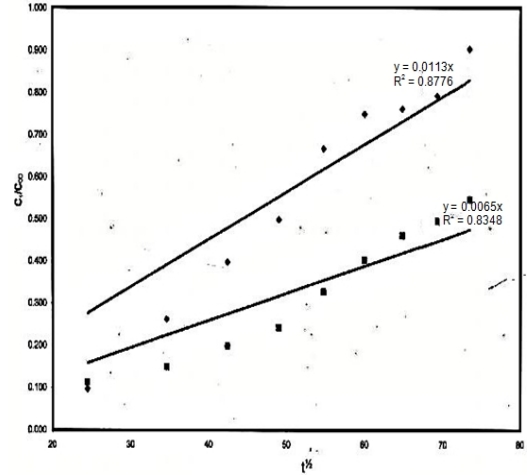


Fig. 16. Rate curves of Dye XV on mylon

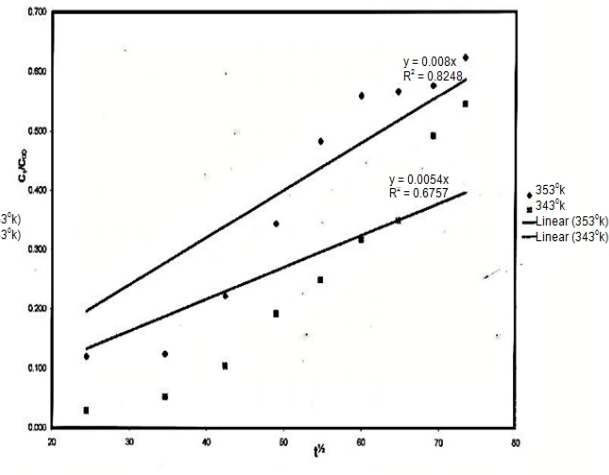


Fig. 17. Rate curves of Dye XVI on mylon

5. CONCLUSION

In conclusion, the results show that the dye-fibre attraction in both polyester fibre and nylon 6 fibre is clearly that of polar and hydrophobic binding forces. In addition, the linear relation between saturation adsorption of the dyes on polyester and nylon 6 suggests that the mechanism of dyeing of these fibres is the same. However, it was found that the dyes under consideration, generally exhibited slightly higher affinity values for the polyester fibre than for the nylon fibre, except for those dyes that contain acidic (SO_3H) and salt (SO_3Na) groups in their structures. The values of the activation energy of diffusion show that there is a high barrier to the mobility of the dye molecules into and within the nylon polymer matrix due to steric effects and hydrogen bond formation between some functional groups of the dye and the polymer. Finally, the results of the study indicate that the dye-fibre interactions are thermodynamically favorable.

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COMPETING INTERESTS

Authors have declared that no competing interests exist.

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