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ScienceDirect
Dyes and Pigments 75 (2007) 378–384
DYES and PIGMENTS
www.elsevier.com/locate/dyepig

Optimization of dye incorporation into modified poly(ethylene terephthalate) knitted fabrics by response surface methodology

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Received 8 May 2006; accepted 12 June 2006
Available online 22 August 2006

Abstract

Poly(ethylene terephthalate) can be modified by UV light and/or *N*,*N*-dimethylacrylamide to better incorporate disperse dyes. In this work, PET knitted fabrics were modified by DMAAm and UV light and then dyed with azo and anthraquinone disperse dyes. Factorial designs were performed at two levels using the following factors: DMAAm treatment time, dyeing time, and UV light exposure time. The best dyeing conditions were obtained with anthraquinone dye in the dyeing of UV light → DMAAm-modified PET knitted fabrics. In this case, the highest amount of incorporated dye was 6.3 mg/g under the following conditions: 77 min UV light exposure time; 15 min and 85 °C DMAAm treatment time and temperature, and 164 min and 85 °C dyeing time and temperature. The diffusion coefficients obtained were in the order of 10^{-5} cm^2/min. The results showed that the azo dye diffuses faster into modified PET than the anthraquinone dye does.

Keywords: Poly(ethylene) terephtalate; Disperse dye; UV light; *N*,*N*-Dimethylacrylamide; Response surface methodology; Dyeing

1. Introduction

Dyeing technology is based on physico-chemical equilibrium processes namely, diffusion and sorption of dye molecules and ions. Poly(ethylene terephthalate) (PET) fibers are the most important synthetic textile fibers in the world due to their high demand [1]. PET dyeing by disperse dye process comprises four stages [2]: (i) dissolution of dye molecules, which change from dispersed to dissolved state, (ii) molecule transport towards the fiber through the solution, (iii) transport by diffusion through the hydrodynamic boundary layer and immediate adsorption onto the fiber surface, and (iv) diffusion into the fiber. Because of the strong dyeing bath stirring in industrial processes, diffusion into the fiber is usually considered the process-determining step. For this reason, on studying the kinetics of PET dyeing, many researchers focus on the determination of the dye diffusion coefficient (D) into the polymer [3–5]. Moreover, due to the hydrophobic nature and the compact molecular structure [6,7] of PET fibers, the conventional dyeing methods present several problems; high-temperature dyeing [8], excessive use of water, and discharge of several chemical additives [9] and difficulties involved in dyeing blends of PET and natural fibers, which do not stand high temperatures, all of which have led to the search of solutions and the publication of several works [10–12]. Studies developed by our group [13,14] show that modifying PET fibers and films with *N*,*N*-dimethylacrylamide(DMAAm) improves dye sorption with significantly shorter dyeing times.

UV light has been used to modify PET surface [15]. PET irradiation with UV light promotes chemical surface

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doi:10.1016/j.dyepig.2006.06.015

modifications and improves wettability. In recent studies [16], it was observed that PET treatment with UV light and *N*,*N*-dimethylacrylamide (DMAAm) decreases surface tension and favors dye incorporation.

Response surface methodology (RSM) [17] has proved to be valuable in studies involving a great variety of problems. An important example is the determination of optimum experimental conditions. In RSM, a polynomial response surface is generally used to describe the relationship between a response variable Y and predicting variables X. The functional relationship for a quadratic model with k independent variables is often:

$$Y = \xi_0 + \sum \xi_i X_i + \sum \xi_{ii} (X_i)^2 + \sum \xi_{ij} X_i X_j + E, \quad i,j = 1,2,3\ldots k$$

In this work, factorial design was used to evaluate the effect of the factors associated to pre-treatment and dyeing UV light- and DMAAm-modified PET in relation to dye incorporation. RSM was used to determine the best treatment and dyeing conditions for PET knitted fabrics. Dye incorporation results were analyzed along with the diffusion coefficients calculated for each dyeing system studied.

2. Experimental

2.1. Materials

This work was conducted with commercial poly(ethylene terephthalate) knitted fabrics (provided by Seda Têxtil Ltda) made of 174/22 dTex yarns with 18 μM diameter filaments. Dyes (Dy Star): Navy Blue Dianix ER-FS 200 (CI Disperse Blue 79) and Red Dianix E-FB (CI Disperse Red 60). Modifier: *N*,*N*-dimethylacrylamide (Fluka). Fig. 1 shows the dye, modifier, and PET chemical structures.

2.2. Pre-treatment of PET knitted fabrics

Non-modified PET knitted fabrics were first washed for 6 h and dried. Then, they were subjected to UV light and DMAAm treatments.

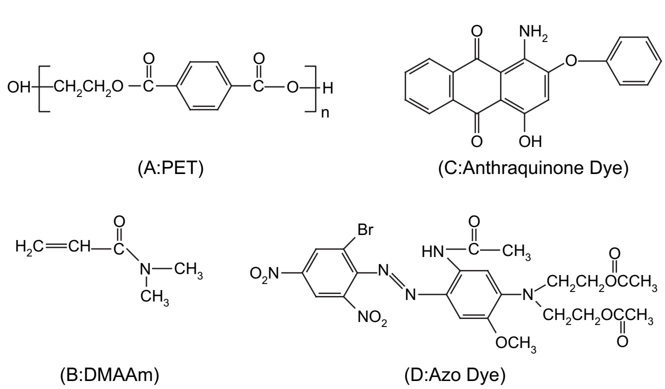

Fig. 1. (A): Poly(ethylene terephthalate), (B): *N*,*N*-dimethylacrylamide, (C): CI Disperse Red 60 (Red Dianix E-FB), and (D): CI Disperse Blue 79 (Navy Blue Dianix ER-FS 200).

UV light irradiation: non-modified and DMAAm-modified knitted fabrics were placed in a booth with a 250 W high-pressure mercury vapor lamp from EMPALUX as a UV light source. Lamp emission peaks were at 254, 263, 297, 303, and 365 nm [18]. The samples were placed at a fixed distance of 5.5 cm from the source. The UV light booth was kept closed and did not have vents. Under these conditions, the sample was heated and consequently subjected to thermal treatment. To exemplify, Fig. 2 shows the sample-heating curve inside the booth during UV light treatment.

DMAAm treatment: non-modified knitted fabric and UV light-treated knitted fabric were immersed into the modifying solvent, DMAAm, at 85 °C for different lengths of time.

Two types of pre-treatment were used in this work:

(i) DMAAm sorption followed by UV light exposure (DMAAm → UV light)
(ii) UV light exposure followed by DMAAm sorption (UV Light → DMAAm).

2.3. PET knitted fabric dyeing

Non-modified and DMAAm → UV light- and/or UV → DMAAm light-modified PET knitted fabrics were dyed under different conditions, according to factorial designs and star designs. The PET knitted fabrics were immersed into a dye aqueous dispersion 1:150 (g/mL). It was used at a concentration of 3 wt.% of dye in relation to the PET knitted fabrics. Dyeing was performed, under stirring, in a glass cell coupled to a thermostatized water bath at 85 °C.

Table 1 shows the factors and values for the levels used in the complete factorial designs for both dye types. The main and the interaction effects of DMAAm treatment time, UV radiation exposure time, and dyeing time on the Dm/KFm (dye mass/knitted fabric mass) response were analyzed.

The best modification and dyeing conditions were determined by response surface methodology (RSM). Factors and

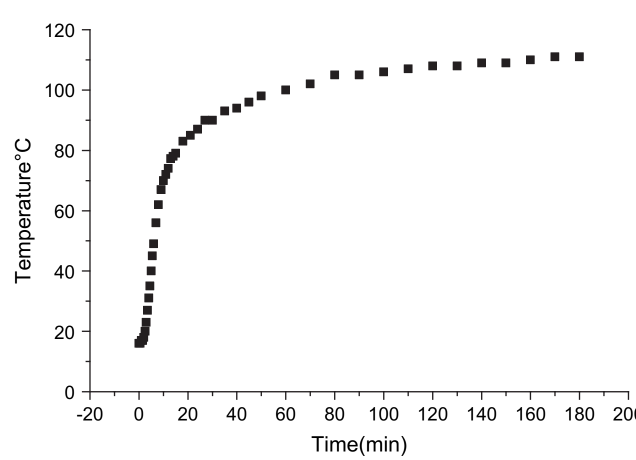

Fig. 2. Heating curve of PET sample inside the booth during UV light treatment.

Table 1
Factors and values of the low (−1) and high (1) levels of the 2^3 complete factorial design used in PET knitted fabric modification and dyeing

Factors	Level (1)	Level (−1)
A = DMAAm treatment time (min)	60	15
B = UV radiation exposure time (min)	90	30
C = dyeing time (min)	180	30

values of the five levels used in the star designs are shown in Table 2.

2.4. Monitoring of dye incorporation into PET and knitted fabrics

The quantities of dye sorbed by PET were determined by UV−vis spectroscopy. Dye was extracted from PET with *N*,*N*-dimethylformamide (DMF) and later absorbance was determined for each of the dye solutions extracted with DMF.

3. Modeling of experimental data and calculation of diffusion coefficient

The continuity equation that describes diffusion in a transient regime was used for fitting concentration data as a function of time [19].

$$\frac{\partial q}{\partial t} = D\frac{\partial^2 q}{\partial z^2} \tag{1}$$

with the following boundary and initial conditions:

(a) at $t = 0$:

$$q = 0 \tag{2}$$

(b) at $z = 0$ and $t > 0$:

$$q = q_e \tag{3}$$

(c) at $z = L$ and $t > 0$:

$$q = q_e \tag{4}$$

where q is the dye concentration in PET (mg/g), q_e is the equilibrium dye concentration in PET (mg/g), D is the dye diffusion coefficient in PET (m^2/min), L is the PET thickness (m), t is the time (min), and z is the axial coordinate.

It was assumed that dye concentration on the PET surface was constant and in equilibrium with liquid phase dye concentration.

The method of lines that approximates space derivatives by finite differences was used for solving Eq. (1). The resulting ordinary differential equation system was solved using Petzold's DASSL [20] FORTRAN subroutine. The values of the dye concentration profiles in PET were used to determine the medium dye concentration. Dye diffusion coefficient was determined by minimization of the following objective function:

$$F_{obj} = \sum_{j=1}^{n}\left(q_j^{Exp} - q_j^{Mod}\right)^2 \tag{5}$$

where n is the number of experimental data, q_j^{Exp} is the experimental dye concentration in PET (mg/g) and q_j^{Mod} is the measured dye model concentration in PET (mg/g).

Table 2
Factors and values of star design levels used in the modification and dyeing of PET fabrics (DMAAm treatment time = 15 min)

Time factors (min)	Level ($2^{1/2}$)	Level (1)	Level (0)	Level (−1)	Level ($-2^{1/2}$)
X_1 = UV radiation exposure	102	90	60	30	18
X_2 = dyeing	211	180	105	30	0

4. Results and discussion

4.1. Optimization of PET knitted fabric pre-treatment and dyeing parameters

The factorial design described in Table 1 was used to evaluate the influence of each studied variable on the incorporation of both dyes in DMAAm → UV light- and UV light → DMAAm-modified PET knitted fabrics. The results are presented in Table 3.

Analysis of the data in Table 3 shows that the amount of anthraquinone dye incorporated in the modified knitted fabric is larger than the amount of azo dye under most of the factorial design conditions. The data in Table 3 are used to obtain Table 4 and analyze the main and the interaction effects of DMAAm treatment time, UV radiation exposure time, and dyeing time on the Dm/KFm response.

Table 3
Codified variables and Dm/KFm for DMAAm → UV light- and UV light → DMAAm-modified PET knitted fabrics dyed with azo and anthraquinone dyes

Experiments	Codified Variables			Dm/KFm response (mg/g)			
				DMAAm → UV light		UV light → DMAAm	
	A	B	C	Azo dye	Anthraquinone dye	Azo dye	Anthraquinone dye
1	−1	−1	−1	1.0	1.4	2.4	2.7
2	1	−1	−1	1.0	2.7	1.7	3.0
3	−1	1	−1	2.3	1.9	3.2	3.5
4	1	1	−1	1.2	2.3	3.4	3.5
5	−1	−1	1	2.3	3.0	3.3	4.6
6	1	−1	1	1.8	2.2	3.9	4.1
7	−1	1	1	2.3	3.3	4.2	5.7
8	1	1	1	2.6	4.7	5.4	5.6

A = DMAAm treatment time; B = UV light exposure time; C = dyeing time. Mean error. UV light → DMAAm − azo: 0.4, anthraquinone: 0.2; DMAAm → UV light − azo: 0.4, anthraquinone: 0.4.

Table 4
Main and interaction effect of variables A, B, and C of the 2^3 complete factorial design described in Table 3

Variables	DMAAm → UV light		UV light → DMAAm	
	Azo dye	Anthraquinone dye	Azo dye	Anthraquinone dye
A	–[a]	0.57	0.36	–[a]
B	0.58	0.71	1.22	1.00
C	0.86	1.23	1.55	1.83
A × B	–[a]	–[a]	0.38	–[a]
A × C	–[a]	–[a]	0.55	–[a]
B × C	–[a]	0.70	–[a]	–[a]
A × B × C	–[a]	–[a]	–[a]	–[a]

A = DMAAm treatment time; B = UV light exposure time; C = dyeing time.
[a] Non-significant effects that are not considered by normal plot analysis.

Table 4 indicates that the main effect of the dyeing time factor is relevant for the response in all the studied dyeings. UV radiation exposure time has a positive main effect on the Dm/KFm response for all studied systems. DMAAm treatment time has a non-significant main effect on the dyeing of DMAAm → UV light-modified PET knitted fabric with the azo dye and also UV light → DMAAm-modified PET knitted fabric with the anthraquinone dye. In the other cases, the main effect of DMAAm treatment time is smaller than the main effects of UV light exposure time and dyeing time variables.

The interaction between the dyeing time and UV radiation exposure time factors is synergic for the dyeing of DMAAm → UV light-modified PET knitted fabric with anthraquinone dye. In the same way, the interaction effect between the dyeing time and DMAAm treatment time is synergic for UV light → DMAAm-modified PET knitted fabric dyeing with azo dye.

After the factorial designs were completed, star designs [21,22] were performed in order to calculate the best modification and dyeing conditions in relation to the UV light exposure time and dyeing time factors. With the star planning, it is possible to obtain a relationship between the dependent variable (response) and the independent variables (factors). In the case of these two factors, this design consists of a complete 2^2 factorial extended by four axial points and three central points, which totalized 11 experiments. The DMAAm treatment time factor was not used in the star design, since it did not have a significant main effect on the Dm/KFm response. Factors and values of the five levels used in the star design are shown in Table 2. The values of the Dm/KFm responses used in the star design are given in Table 5.

By regression, it was possible to fit a second-order model describing the relationship between the responses and the factors in Table 5. Each of the response columns in Table 5 was adjusted to a second-order model $Y = \xi_0 + \xi_1 X_1 + \xi_2 X_2 - \xi_3(X_1)^2 - \xi_4(X_2)^2 + \xi_5 X_1 X_2$ by multiple regression based on the least square method. Table 6 shows the variance analysis (ANOVA) for the fit models. The quality of the model fit is evaluated by the parameter $R^2 = SQ_R/SQ_T$ and its statistical significance is determined by the F test. Parameter R^2 estimates the variation percentage in the responses that can be explained by the fit model. According to Kim et al. [23], R^2 values over 85% provide an excellent explanation for the relationship between independent and dependent variables. In order to perform variance analysis, the deviations of the data obtained in the experiments from the model were subdivided according to the specific variation source and hypothesis tests were performed. To test the significance of the model

Table 5
Results of the star design used to determine the best modification and dyeing conditions for DMAAm → UV light- and UV light → DMAAm-modified PET fibers

Experiments	Codified variables		Response Dm/KFm (mg/g)			
			DMAAm → UV light		UV light → DMAAm	
	X_1	X_2	Azo dye	Anthraquinone dye	Azo dye	Anthraquinone dye
1	−1	−1	1.0	1.4	2.4	2.7
2	1	−1	2.3	1.9	3.2	3.5
3	−1	1	2.3	3.0	3.3	4.6
4	1	1	2.3	3.3	4.2	5.7
5	0	0	1.9	2.2	4.3	4.9
6	0	0	2.5	2.7	5.4	5.2
7	0	0	2.3	2.9	4.6	6.3
8	$-2^{1/2}$	0	1.7	2.7	2.8	2.7
9	0	$2^{1/2}$	2.6	3.2	4.4	6.1
10	$2^{1/2}$	0	3.8	3.0	4.6	5.0
11	0	$-2^{1/2}$	0.00	0.0	0.0	0.0

X_1 = UV light exposure time; X_2 = dyeing time.
Mean error. UV light → DMAAm – azo: 0.4, anthraquinone: 0.2; DMAAm → UV light – azo: 0.4, anthraquinone: 0.4.

Table 6
ANOVA for the model $Y = \xi_0 + \xi_1 X_1 + \xi_2 X_2 - \xi_3(X_1)^2 - \xi_4(X_2)^2 + \xi_5 X_1 X_2$ fitted to the star design data, Table 5, considering the response Y_i = Dm/KFm

	Variation source	Degrees of freedom	Square sum	Square mean	F test	P
DMAAm → UV light	*Azo dye*					
	Regression	5	7.89	1.58	6.91	0.0268
	Residues	5	1.14	0.23		
	Lack of fit	3	1.01	0.34	4.87	0.1750
	Pure error	2	0.14	0.069		
	Total	10	9.04			
	Anthraquinone dye					
	Regression	5	8.93	1.79	14.80	0.0051
	Residues	5	0.60	0.12		
	Lack of fit	3	0.38	0.13	1.15	0.4962
	Pure error	2	0.22	0.11		
	Total	10	9.53			
UV light → DMAAm	*Azo dye*					
	Regression	5	19.01	3.80	5.83	0.0378
	Residues	5	3.26	0.65		
	Lack of fit	3	2.54	0.85	2.47	0.3142
	Pure error	2	0.73	0.36		
	Total	10	22.27			
	Anthraquinone dye					
	Regression	5	30.18	6.04	6.23	0.0331
	Residues	5	4.84	0.97		
	Lack of fit	3	3.72	1.24	2.21	0.3263
	Pure error	2	1.12	0.56		
	Total	10	35.03			

parameters, we divided the square mean due to the regression by the residual square mean and obtained a value for F for the regression. High values of F indicate that the variation in responses can be explained by the fit model. The value of P associated to the value of F is used to indicate whether F is high enough to indicate statistical significance. P-values smaller than 0.05 ($\alpha = 0.05$, or 95% confidence) indicate that the model is statistically significant. ANOVA also allows us to evaluate the amount of variation in the response that cannot be explained by the model. The lack of fit term indicates the variations caused by model inadequacy. Values for $F_{\text{lack of fit}}$ are obtained by dividing the square mean of the lack of fit by the square mean for the pure error. Values for $P_{\text{lack of fit}}$ higher than 0.05 indicate that the model lack of fit is not significant at 95% confidence level.

The results in Table 6 show that the models do not present significant lack of fit at 95% confidence level. Values for $P_{\text{lack of fit}}$ ranged between 0.4962 and 0.1750, all larger than 0.05. $P_{\text{regression}}$ values ranged between 0.0051 and 0.0378, all smaller than 0.05, showing that at least one of the parameters in each model is significant at 95% confidence level. The value of $R^2 = SQ_R/SQ_T$ indicates that even in the worst of the cases 85% (19.01/22.27 = 0.854), the results can be explained by the models.

Fig. 3 shows the response surface obtained for model $Y = \xi_0 + \xi_1 X_1 + \xi_2 X_2 - \xi_3 (X_1)^2 - \xi_4 (X_2)^2 + \xi_5 X_1 X_2$ fit to the results in Table 5 for the response column Y_i = Dm/KFm (anthraquinone dye; treatment: DMAAm → UV light)

Response surfaces were obtained for the other response columns of Table 5 and used to calculate the maximum points for each of the surfaces. Next, experiments were performed under the conditions that the model indicated as leading to the maximum dye incorporation. Table 7 gives the incorporation values for these conditions. Table 7 also contains the points of maximum dye incorporation points to DMAAm → UV light- and UV light → DMAAm-modified PET knitted fabrics.

Table 7 indicates that the UV light → DMAAm pre-treatment is more efficient for the incorporation of both dyes than DMAAm → UV light one. The best dye sorption result was obtained with UV light → DMAAm pre-treatment and anthraquinone dye. The results presented in Table 7 show the efficiency of the statistical models used. The experimental values obtained under maximal incorporation conditions agree with the calculated values.

Table 7
Maximum points obtained by the model $Y = \xi_0 + \xi_1 X_1 + \xi_2 X_2 - \xi_3 (X_1)^2 - \xi_4 (X_2)^2 + \xi_5 X_1 X_2$ adjusted to the results in Table 5

	Treatment	Factors (min)	Dm/KFm responses (mg/g)	
			Model	Experimental
Azo dye	UV light → DMAAm	$X_1 = 78$, $X_2 = 138$	5.2	4.8
	DMAAm → UV light	$X_1 = 102$, $X_2 = 119$	3.5	3.7
Anthraquinone dye	UV light → DMAAm	$X_1 = 77$, $X_2 = 164$	6.3	6.0
	DMAAm → UV light	$X_1 = 90$, $X_2 = 171$	3.3	3.4

Mean error. UV light → DMAAm − azo: 0.4, anthraquinone: 0.2; DMAAm → UV light − azo: 0.4, anthraquinone: 0.4.

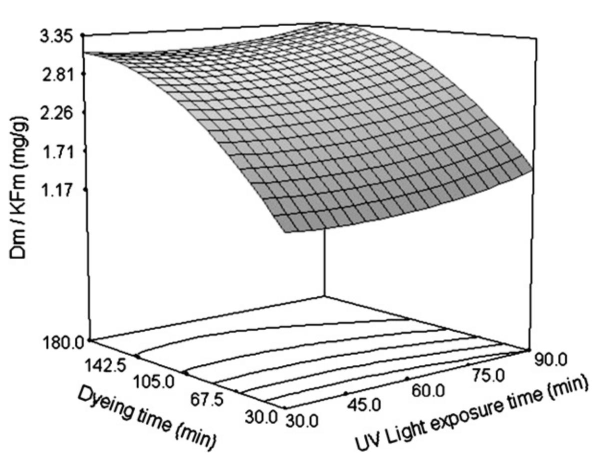

Fig. 3. Response surface for the model fit to the results in Table 5 for the response column Y_i = Dm/KFm (anthraquinone dye; treatment: DMAAm → UV light).

4.2. Calculation of the diffusion coefficients for the dyeing systems

By using the response surfaces, it was possible to obtain dye concentration curves for PET knitted fabrics (Dm/KFm) as a function of dyeing time for several UV light treatment times. Based on the curves for dye concentration in PET knitted fabrics as a function of dyeing time, it was possible to fit the model presented in Section 3 (Eq. (1)).

Fig. 4 shows the Dm/KFm curve as a function of dyeing time calculated using the model and the points obtained by statistical treatment (Fig. 3) for the PET sample treated with UV light for 90 min.

Fig. 4 shows that the statistical model and the phenomenological model curve points are in good agreement.

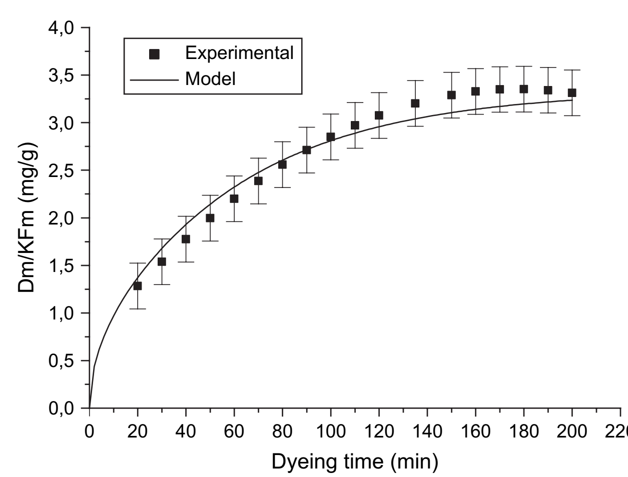

Fig. 4. Dye concentration curves for PET knitted fabrics (Dm/KFm) as a function of dyeing time. The PET samples were treated with UV light for 90 min.

Table 8
Dye diffusion coefficients of DMAAm → UV light- and UV light → DMAAm-modified PET knitted fabrics

Disperse dye	Treatment	UV light exposure (min)	D (cm^2/min) × 10^5	UV light exposure (min)[a]	D (cm^2/min) × 10^5
Azo	DMAAm → UV light	90	3.8 ± 0.2	102	4.9 ± 0.3
	UV light → DMAAm	90	2.0 ± 0.1	78	2.1 ± 0.1
Anthraquinone	DMAAm → UV light	90	1.7 ± 0.1	90	1.7 ± 0.1
	UV light → DMAAm	90	1.7 ± 0.1	77	1.8 ± 0.1

[a] The best UV light treatment times.

Based on the continuity of equation (Eq. (1)), it was possible to obtain the diffusion coefficient for each dye and each type of modified PET knitted fabric. The diffusion coefficients under the same UV light treatment conditions, the 90 min UV light PET treatment diffusion coefficients were calculated for comparison. Diffusion coefficients were also calculated for the best UV light treatment times shown in Table 7.

Table 8 shows the diffusion coefficients of both dye types for PET knitted fabrics modified by DMAAm → UV light and UV light → DMAAm.

The data presented in Table 8 indicate that the azo dye diffuses faster into modified PET than the anthraquinone dye does. The highest diffusion coefficient was obtained for the DMAAm → UV light-modified PET knitted fabric.

Based on the diffusion coefficient values (Table 8) it can be inferred that the anthraquinone dye diffuses more slowly in modified PET knitted fabrics. However, higher anthraquinone dye concentrations were obtained for the UV light → DMAAm-modified PET knitted fabric at the maximum point (Table 7), which makes it possible to obtain more intense colors with anthraquinone dye. On the other hand, azo dye incorporation levels are lower under the optimal conditions determined for UV light → DMAAm-modified PET knitted fabric. Nevertheless, the dye penetrates the fiber the fastest. This is an advantage in practice, because dyeing processes are performed in limited time and with excess dye.

The diffusion coefficient values obtained for DMAAm → UV light-modified PET knitted fabric are higher than the ones usually found in literature for PET fiber dyeing with disperse dyes [4,8,24]. Clerck et al. [4] calculated diffusion coefficients for the CI Disperse Red 60 in PET fabrics made of 167/30 dTex yarns with filaments of 23–24 μM diameter in the order of 10^{-10} cm^2/min. The values shown in Table 8 are in the order of 10^{-5} cm^2/min and were calculated for CI Disperse Red 60 dye (anthraquinone dye) in knitted fabrics of 174/22 dTex yarns with filaments of 18 μM diameter. These results show the potential of the UV light and DMAAm PET modification techniques for dyeing PET knitted fabrics with disperse dyes.

5. Conclusions

The statistical models used proved to be efficient in the evaluation of the influence of several factors in the chemical modification of PET knitted fabric and its dyeing in aqueous medium. Based on statistical treatments, it was observed that the dyeing time factor has a main effect stronger than that of the UV light exposure factor. However, both favor dye incorporation into PET and have a synergic effect. DMAAm treatment time has little effect on PET dye incorporation in an aqueous medium. UV light → DMAAm treatment shows better results than DMAAm → UV light does concerning the dye incorporation of both studied dyes. The evaluation of dye incorporation results through diffusion coefficients showed that both pre-treatments, UV light and DMMAm, have potential use in industrial PET knitted fabric dyeing.

Acknowledgements

The authors gratefully acknowledge the financial support of CAPES, CNPq and Fundação Araucária.

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