THERMODYNAMIC INVESTIGATION OF DISPERSE DYES SORPTION ON POLYESTER FIBERS

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The thermodynamics of sorption during the dyeing process of raw polyester fibers with disperse dye was studied in this article. Dyeing of fibers is carried out by a discontinuous batch process, according to standard procedures with varying of the initial dye concentration and the dyeing temperature. Several of the accepted models for calculation of thermodynamic parameters were used which define and clarify the sorption of disperse dyes on polyester fibers. High functionality of variables for the calculation of basic thermodynamic parameters was determined, which confirmed the validity of the results. Depending on the method of calculation, the change of free energy during the dyeing of the polyester was negative ($-10 - -40 \text{ kJ}\cdot\text{mol}^{-1}$), which corresponds to the spontaneous and mainly the physical process of sorption. A positive value for the change in enthalpy during dyeing ($1.1\times 10^{-2} - 2.9\times 10^1 \text{ kJ}\cdot\text{mol}^{-1}$) reflects the endothermic nature of the interactions, and a relatively lower value characterizes the physisorption phenomenon. A positive value for the change in entropy ($1.1\times 10^{-4} - 1.5\times 10^{-1} \text{ kJ}\cdot\text{mol}^{-1}\cdot\text{K}^{-1}$) confirms that it is a physical sorption.

Keywords: polyester, modification, dyeing, disperse dye, thermodynamics parameters.

Introduction

Polyester fibers are widely used in textile industry for the production of variety of products for clothing or technical purposes. The fiber has a very compact and crystalline structure and is very hydrophobic. It is mainly dyed with disperse dyes with low solubility in water in specific conditions: high temperature and presence of a carrier. Low water solubility and a particle size of the disperse dye play a vital role in the rate of dissolution and the rate of the adsorption dye for a polyester fiber [1,2]. In practice, it is very difficult to achieve adequately stronger and darker shades at dyeing of polyester under normal conditions of temperature and air pressure, it is always the imperative to work on clarifying the sorption process with kinetic and thermodynamic point of view, as well as to develop new methods of dyeing to increase the dyeing rate and improve the dye absorption.

The sorption process is usually complex and mixed and is the result of the joint action of several different types of actions. In the case of physical adsorption, multi-layer adsorption of the adsorbate (dye) on the surface of the adsorbent (polyester fiber) may occur. Adsorbate molecules are bonded by *van der Waals* forces on the surface of the adsorbent and can move translationally over the surface. These forces belong to the group of weaker ones, so that the energy released during physical adsorption is small. Thanks to this energy, the adsorbate particles move on

the surface of the adsorbent, gradually losing energy, and eventually being adsorbed. At little change of enthalpy, the adsorption is insufficient to cause the rupture of chemical bonds, but physisorbed particles retain their chemical identity, i.e. the initial chemical structure, and by desorption, they can return to their initial state in solution. Physical adsorption is most often a reversible process and occurs mainly at lower temperatures [3].

During chemisorption, at the interaction of the adsorbent and the adsorbate, chemical bonds are formed, mostly covalent, during which a monolayer of the adsorbed substance is formed. Chemisorbed particles undergo a chemical change (dissociation reaction), which means that they lose their chemical identity and cannot return to the solution by simple desorption. Chemically adsorbed molecules cannot move on the adsorption surface because the adsorbate forms a strong local bond with the active centers on the adsorbent, changing the composition and properties of the surface. This type of adsorption is characterized by a high activation energy which ranges from 100 to 400 $\text{kJ}\cdot\text{mol}^{-1}$ [4].

Adsorption is a temperature-dependent phenomenon, and the evaluation of thermodynamic parameters reflects the feasibility of the sorption process. The temperature has two main influences on sorption; rising temperature increases the rate of diffusion of adsorbate molecules through the outer

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barrier layer as well as towards intern pores of adsorbent particles. Spontaneity of the system is defined by an assessment of free energy change; there are changes in enthalpy and entropy, which also define the conditions of performing a sorption process, i.e. polyester dyeing in this case [5].

The aim of this research is to increase the success of dyeing by clarifying the dye-polyester interactions, defining the character of sorption, as well as adjusting the results for the needs of working and operational conditions.

Experimental

In the experimental part of the paper, raw, undyed 100% polyester fibers were used, with the following characteristics: length 85 mm, fineness 1.7 dtex, strength 51.7 cN·tex⁻¹, with 30% of elongation to break.

For dyeing of these polyester fibers, C.I. Disperse Yellow 42 disperse dye was used. For checking the thermodynamic parameters, a fed-batch (discontinuous) process was used.

The dyeing process is organized in the bath with the constant volume of 0.1 dm³, the temperature of dyeing varied from 303 to 368 K, the time of dyeing was 60 min while the dye concentration varied from 20 to 120 mg·dm⁻³. Of the additives, Alvion W dispersant (Texticolor, Switzerland) 1 g·dm⁻³ and acetic acid (Tehnohemija ad, Serbia) were used in all cases.

UV-VIS spectrophotometry and the Cary 100 Conc UV-VIS apparatus, Varian (absorption maximum at 440 nm) was used to determine the dye concentration in the solution.

The change of free energy ΔG , in the equilibrium conditions [5], was defined in the following way:

$$\Delta G = -R \cdot T \cdot \ln(K_{eq}) \quad (1)$$

where: K_{eq} is constant of thermodynamic balance (nondimensional parameter).

The ratio between the standard Gibbs change of the free surface energy, the standard change in enthalpy (ΔH) and a standard change in the entropy (ΔS) [5] is as follows:

$$\Delta G = \Delta H - T \cdot \Delta S \quad (2)$$

From the equations (1) and (2) the following equation is obtained:

$$\ln(K_{eq}) = -\frac{\Delta H}{R \cdot T} + \frac{\Delta S}{R} \quad (3)$$

If $\ln(K_{eq})$ is drawn versus $1/T$ (Van't Hoff diagram), multiplying the slope and intercept of the curve on the diagram with the universal gas constant (R), ΔH and ΔS are calculated. The equilibrium constant should be a dimensionless quantity in accordance with the dimensionality of equation (1).

By reviewing of the adequate literature, it was found that several different approaches [6,7] are used to determine K_{eq} . Thus, the use of b (dm³·mg⁻¹, Langmuir constant) as K_{eq} is found in numerous literatures. In addition to this, the use of constants from other models of the adsorption iso-

therms is practiced, i.e. *Freundlich*, *Frumkin*, *Flori - Huggins* model etc. [8].

Also, the equilibrium constant can be derived from the distribution coefficient (K_d), then the partition coefficient (K_p) or from some other theoretical approach. The main issue is that the equilibrium constant must be dimensionless.

K_d and K_e can be represented [6,7] in the following manner:

$$K_d = \frac{q_e}{C_e} \quad \text{and} \quad K_e = \frac{C_s}{C_e} \quad (4)$$

where in: C_s is the concentration of the dissolved substance adsorbed on the adsorbent at equilibrium in mg·dm⁻³, C_e is the concentration of adsorbates in the solution, in equilibrium in mg·dm⁻³.

The value of the distribution coefficient, K_d , is obtained by drawing the dependence $\ln(q_e/C_e)$ on C_e and extrapolating of C_e to zero. After fitting the experimental points (necessary high value of the coefficient of determination, R^2), a place where the line fitting crosses vertical axis gives the value of K_e .

Since the coefficient K_d has the dimension, in order to be used as the equilibrium constant of the thermodynamic equation, its translation into the non-dimensional quantity by multiplication with water density is done, or by multiplying by a factor of 55.5 and a molar mass of adsorbates [7,8], in the second case:

$$K_{eq}(\text{dimensionless}) = 1000(g \cdot dm^{-3}) \times K_d(dm^3 \cdot g^{-1}) \quad (5)$$

$$K_{eq}(\text{dimensionless}) = 55.5(mol \cdot dm^{-3}) \cdot M_w(g \cdot mol^{-1}) \cdot K_d(dm^3 \cdot g^{-1}) \quad (6)$$

$$\Delta G = -R \cdot T \cdot \ln(1000 \cdot K_d) \quad (7)$$

$$\Delta G = -R \cdot T \cdot \ln(55.5 \cdot M_w \cdot K_d) \quad (8)$$

where: factor 55.5 is the number of moles of pure water per liter; M_w is molar mass of the adsorbate (g·mol⁻¹).

The values of K_p can be obtained by drawing the diagram $\ln(C_s/C_e)$ towards C_e and extrapolating C_e to zero. In this case, the partition coefficient was in accordance with the equilibrium constant, so there was no need for its change into dimensionless quantity.

$$K_{eq}(\text{dimensionless}) = K_p \quad (9)$$

$$\Delta G = -R \cdot T \cdot \ln(K_p) \quad (10)$$

Thermodynamic equilibrium constant can be determined by calculation from the constant of *Langmuir* isotherm b (dm³·mg⁻¹) using the *Van't Hoff* thermodynamic equation [9].

In order to obtain the dimensionless parameter, the *Langmuir* constant is multiplied by 10⁶ (water density is 10⁶ mg·dm⁻³), because the testing was performed from an aqueous solution:

$$K_{eq}(\text{dimensionless}) = 10^6(mg \cdot dm^{-3}) \cdot b(dm^3 \cdot mg^{-1}) \quad (11)$$

$$\Delta G = -R \cdot T \cdot \ln(10^6 \cdot b) \dots \dots \dots (12)$$

ΔH and ΔS are obtained from the slope and the intercept on the diagram according to the equations 2 or 3. However, some authors [10] recommend that K_{eq} can be obtained as a non-dimensional parameter by multiplying b ($\text{dm}^3 \cdot \text{mg}^{-1}$) with a molar mass of the adsorbates (M_w , $\text{g} \cdot \text{mol}^{-1}$), and with 1000 and finally, with a factor of 55.5:

$$K_{eq} (\text{dimensionless}) = M_w (\text{g} \cdot \text{mol}^{-1}) \cdot 55.5 (\text{mol} \cdot \text{dm}^{-3}) \cdot 1000 (\text{mg} \cdot \text{g}^{-1}) \cdot b (\text{dm}^3 \cdot \text{mg}^{-1}) \dots \dots \dots (13)$$

$$\Delta G = -R \cdot T \cdot \ln(M_w \cdot 55.5 \cdot 1000 \cdot b) \dots \dots \dots (14)$$

The effect of temperature on the adsorption of the adsorbate can be clarified by the analysis of activation energy [6] of the modified equation of *Arrhenius* type:

$$S^* = (1 - \theta) \cdot \exp\left(\frac{-E_a}{R \cdot T}\right) \dots \dots \dots (15)$$

where S^* is probability of bonding, and depends on the temperature of the system, E_a is the activation energy ($\text{kJ} \cdot \text{mol}^{-1}$).

Area coverage (θ) is estimated [6] by the following equation:

$$\theta = \frac{1 - C_e}{C_0} \dots \dots \dots (16)$$

where C_0 is the initial dye concentration ($\text{mg} \cdot \text{dm}^{-3}$).

S^* and E_a are calculated from the graph $\ln(1 - \theta)$ versus $1/T$ via linear regression from the slope and intercept curve.

Results and Discussion

The results of thermodynamic parameters are given which are calculated on the basis of the equilibrium constant obtained in different ways, according to the recent literature: from the distribution and partition coefficient as well as from Langmuir constants [8].

Figure 1 shows the dependence of the variable parameters necessary for the calculation of the distribution and partition coefficient, K_d and K_p . After fitting the experimental points at different temperatures, K_d and K_p are calculated from the values on the ordinate, i.e. the place where the fitting line intersects the vertical axis. According to the appearance of a fitted straight line and position towards the experimental points, there is a noticeable high degree of functionality in both diagrams, which confirms that the results of calculating the thermodynamic parameters are acceptable and can be used to describe the dependency.

Figure 1. Diagram for the calculation of the distribution (a) and partition coefficient (b) for the process of dye sorption on polyester at different temperatures

Figure 2 shows two diagrams describing the change in free energy from temperature, depending on the method of determining ΔG , indirectly, via distribution or partition coefficient. The high functionality of fitted straight lines is noticeable which accompany experimental points. Based on these diagrams (slope and intercept), the basic thermodynamic parameters, enthalpy and entropy of the adsorption process were determined. Figure 3 also gives the same dependence, but for the calculation of free energy using *Langmuir* constant. Again, in this case, it is noticeable that fitted straight line perfectly covers the experimental points, so the results can be accepted and further analyzed. Generally, the value of ΔG can determine the character of electrostatic interactions between charged dye molecules and the charged substrate (polyester fibers), i.e. whether this is a chemical or physical sorption (physisorption or chemisorption). Also, there are cases that one type of sorption may be dominant, while the other is only with a slight contribution to the sorption mechanism.

Figure 2. A change in free energy depending on the temperature for the process of dye sorption on polyester (value ΔG is calculated on the basis of an equilibrium constant obtained from the distribution (a) and partition (b) coefficient, K_d and K_p , according to the equations 7, 8 and 10, respectively)

Figure 3. Thermodynamic diagram for sorption of dispersed dye on polyester fiber (value of ΔG is calculated on the basis of the Langmuir constant, b , according to the equations 12 and 14)

Tables 1 to 5 give numerical data for basic thermodynamic parameters obtained via the equilibrium constant, K_{eq} , calculated from the distribution coefficient, partition coefficient and Langmuir constant. Tables 1 and 2 refer to the calculation of thermodynamic parameters over K_d , which is converted into a dimensionless quantity based on the equations 5 and 6, using various factors, with annulled dimensionality of the distribution coefficient. Table 3 refers to the parameters obtained via the partition coefficient, whereas Tables 4 and 5 refer to the results in which the Langmuir constant b was used.

According to the values for the coefficient of determination, R^2 , in all Tables the high dependence of the regression models obtained by fitting the experimental points is confirmed; practically, in all cases this statistical parameter is higher than 0.99. Hereby, the validity of the results is completely accepted, i.e. the adequacy and applicability of the obtained values for the basic thermodynamic parameters.

The weakest result was shown by the application of the partition coefficient for calculating the equilibrium constant and then for other thermodynamic parameters, according to the result for R^2 , as well as to the numerous values of individual thermodynamic parameters.

Based on the results for ΔG in Tables 1-5, the negative values (-10 – -40 $\text{kJ}\cdot\text{mol}^{-1}$) indicate the justification and spontaneity of the nature of the process, i.e. the favoring nature of dye adsorption at 303, 333 and 368 K, with high potential. Thus, this confirms the feasibility of the process and the spontaneous nature of the adsorption at the applied temperatures, with a good degree of the dye molecules affinity for the adsorbent surface through physisorption.

In general, the change in free energy for physisorption is known to be between -20 and 0 $\text{kJ}\cdot\text{mol}^{-1}$, physisorption together with chemisorption is in the range of -20 and -80 $\text{kJ}\cdot\text{mol}^{-1}$, whereas chemisorption is in the range of -80 to -400 $\text{kJ}\cdot\text{mol}^{-1}$ [11].

The negative value of ΔG is reduced with increasing the temperature, suggesting that the process of sorption in the specific case is beneficial, naturally spontaneous and efficient at higher temperatures. Decreasing the value of ΔG with increasing the temperature favors the process of exhaustion of a disperse dye on the polyester fiber from the bath at high temperature. Since the values for ΔG in this specific case are in the range -10 to -40 $\text{kJ}\cdot\text{mol}^{-1}$, the physical character of sorption during dyeing of the polyester fiber by disperse dye is confirmed.

A positive value for enthalpy from all tables (1.1×10^{-2} – 2.9×10^{-1} $\text{kJ}\cdot\text{mol}^{-1}$) reflects the endothermic nature of the sorption process, and a lower value of the required amount of heat characterizes the phenomenon of physisorption [11]. This result can also be an indicator of the occurrence of a single-layer adsorption. Moreover, physisorption can be explained by the presence of many groups on the adsorbate and adsorbent that are capable of forming electrostatic cooperation, resulting in a low enthalpy due to weak coulomb interaction between protons and the

weak basic groups spread on the surface of the adsorbent [11].

The positive value of ΔS from the Tables (from 1.1×10^{-4} to $1.5 \times 10^{-1} \text{ kJ} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$), corresponds to the increased degree of freedom in the solid/liquid interface, as a result of dye molecules sorption. In this particular case, the values for the change in entropy are close to zero, i.e. to negative values, so it is confirmed that it is a physical sorption with a possible partial share of chemisorption, considering that the numerous values for ΔS are close to zero but not below zero.

Table 1. The results of thermodynamic parameters for dyeing of modified polyester obtained from the distribution coefficient (according to equation 5)

Temperature (K)	K_d ($\text{dm}^3 \cdot \text{g}^{-1}$)	R^2	ΔG ($\text{kJ} \cdot \text{mol}^{-1}$)	ΔH ($\text{kJ} \cdot \text{mol}^{-1}$)	ΔS ($\text{kJ} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$)	R^2
303	0.045	0.991	-9.60			
333	0.152	0.993	-13.92	29.15	0.13	0.996
368	0.356	0.998	-17.98			

Table 2. The results of thermodynamic parameters for dyeing of modified polyester obtained from the distribution coefficient (according to equation 6)

Temperature (K)	K_d ($\text{dm}^3 \cdot \text{g}^{-1}$)	R^2	ΔG ($\text{kJ} \cdot \text{mol}^{-1}$)	ΔH ($\text{kJ} \cdot \text{mol}^{-1}$)	ΔS ($\text{kJ} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$)	R^2
303	0.045	0.991	-17.21			
333	0.152	0.993	-22.28	29.18	0.15	0.997
368	0.356	0.998	-27.22			

Table 3. The results of thermodynamic parameters for dyeing of modified polyester obtained from the partition coefficient (according to equation 9)

Temperature (K)	K_p	R^2	ΔG ($\text{kJ} \cdot \text{mol}^{-1}$)	ΔH ($\text{kJ} \cdot \text{mol}^{-1}$)	ΔS ($\text{kJ} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$)	R^2
303	0.45	0.991	1.99			
333	1.52	0.993	-1.17	29.15	0.09	0.992
368	3.56	0.998	-3.89			

Table 4. Thermodynamic parameters for dyeing of modified polyester obtained from the *Langmuir* constants (according to equation 11)

Temperature (K)	b ($\text{dm}^3 \cdot \text{mg}^{-1}$)	R^2	ΔG ($\text{kJ} \cdot \text{mol}^{-1}$)	ΔH ($\text{kJ} \cdot \text{mol}^{-1}$)	ΔS ($\text{kJ} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$)	R^2
303	0.013	0.999	-23.80			
333	0.018	0.998	-27.14	0.011	0.00011	0.999
368	0.027	0.997	-31.25			

Table 5. Thermodynamic parameters for dyeing of modified polyester obtained from the *Langmuir* constant (equation 13)

Temperature (K)	b ($\text{dm}^3 \cdot \text{mg}^{-1}$)	R^2	ΔG ($\text{kJ} \cdot \text{mol}^{-1}$)	ΔH ($\text{kJ} \cdot \text{mol}^{-1}$)	ΔS ($\text{kJ} \cdot \text{mol}^{-1} \cdot \text{K}^{-1}$)	R^2
303	0.013	0.999	-31.41			
333	0.018	0.998	-35.51	0.011	0.00014	0.999
368	0.027	0.997	-40.49			

Figure 4 shows the dependence of the surface coverage with temperature for the highest initial dye concen-

tration, $120 \text{ mg} \cdot \text{dm}^{-3}$. The probability of adhesion and the activation energy were determined from both the intercept and the slope. The dependence of the variables is extremely high, $R^2 = 0.999$, which confirms the significant functionality and absolute acceptability of numerous values of the obtained parameters from the regression model.

The size of the activation energy gives an idea of a type of adsorption which is mostly physical or chemical. It is known that the activation energy corresponds to the physical mechanism of sorption in the range of 5 to 40 $\text{kJ} \cdot \text{mol}^{-1}$, while the range of 40 to 800 $\text{kJ} \cdot \text{mol}^{-1}$ suggests the mechanism of chemisorption [3,11].

The result obtained for the activation energy in this study was $3.5 \text{ kJ} \cdot \text{mol}^{-1}$, indicating that the dye sorption onto the fiber can confirm the physical sorption mechanism, which is in line with the previous results for other thermodynamic parameters. A smaller amount for E_a , assumes that the reactions are relatively fast and the adsorption process of a disperse yellow dye on polyester is endothermic. The positive value of E_a suggests that the increase in temperature favored the dye adsorption, which agrees with the accepted standard temperatures for dyeing polyester.

From the section of the diagram in Figure 4. the value for the parameter S^* was obtained. This parameter is defined as a function of the adsorption system which shows the potential of adsorbate to remain on the adsorbent. A value of S^* between 0 and 1 favors the sorption. In this particular case, the adhesion probability has a value less than one and higher than zero ($S^* = 0.45$), which means that this is a favorable bonding of the dye to the fiber or the ability of the adsorbate to remain on the adsorbent for a long time.

Figure 4. Variation of surface coverage with temperature to determine activation energy during dye sorption on polyester fiber

Conclusion

In all diagrams. for determining the parameters used to calculate the change in free energy, as well as in the diagrams for calculating the change in free enthalpy and

entropy, a very high functionality of linear regression of the curves was observed. Accordingly, the validity of the results is absolutely accepted, i.e. the adequacy and applicability of calculated values of basic thermodynamic parameters.

The change in free energy during polyester dyeing was negative, which corresponds to the spontaneous and dominant physical process of dye sorption on polyester. In other words, this confirms the feasibility and spontaneity of sorption at the applied temperatures, with good degree of affinity of dye molecules for the adsorbent surface through physisorption.

The positive value for the change in enthalpy during dyeing reflects the endothermic nature of the interactions, and a relatively lower value characterizes the phenomenon of physisorption, i.e. the formation of physical, mostly electrostatic interactions.

The positive value for the change in entropy confirms the physical sorption with a possible partial share of chemisorption, since the values are close to zero, but they are not negative.

Knowing the results of thermodynamic parameters of the dye sorption process on polyester, i.e. knowing the nature and character of interactions between the adsorbate and the adsorbent can imply the possibility of a different approach in the process of dyeing of the polyester with disperse dye, all in favor of a higher exhaustion, saving and environmental protection.

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Izvod

TERMODINAMIČKA RAZMATRANJA SORPCIJE DISPERZNE BOJE NA POLIESTARSKA VLAKNA

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U članku je predstavljena analiza termodinamike sorpcije tokom procesa bojenja sirovih poliestarskih vlakana disperznom bojom. Bojenje vlakana je izvršeno diskontinualno, šaržni proces, prema standardnoj proceduri uz variranje početne koncentracija boje i temperature bojenja. Korišćeno je nekoliko usvojenih modela za izračunavanje termodinamičkih parametara kojima se definiše i pojašnjava proces sorpcije disperzne boje na poliestarsko vlakno. Utvrđena je visoka funkcionalnost promenljivih za izračunavanje osnovnih termodinamičkih parametara, čime je potvrđena validnost rezultata. Zavisno od načina izračunavanja, promena slobodne energije tokom bojenja poliestra, bila je negativna (-10 - -40 kJ·mol⁻¹), što odgovara spontanom i uglavnom fizičkom procesu sorpcije. Pozitivna vrednost za promenu entalpije tokom bojenja (1.1×10^{-2} - 2.9×10^1 kJ·mol⁻¹), odražava endotermnu prirodu interakcija, a relativno manja brojčana vrednost karakteriše fenomen fizisorpcije. Pozitivna vrednost za promenu entropije (1.1×10^{-4} - 1.5×10^{-1} kJ·mol⁻¹·K⁻¹) potvrđuje se da je u pitanju fizička sorpcija.

Ključne reči: poliestar, modifikacija, bojenje, disperzna boja, termodinamički parametri.