

## **Characterization of Dye-ability and Diffusion Kinetics of Chemically Modified Cotton Fabrics towards Disperse and Direct Dyes**

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**Abstract:** *This article aims to study the effect of chemical modification of cotton fabrics on their dye-ability towards disperse and direct dyes. The cotton samples were modified with 2-cholor-2-dimethylaminoethyl hydrochloride with two different concentrations (10 and 30) %. The obtained synthesized dimethylamino ethyl (DMAE) cotton samples, had two different nitrogen contents (0.386, 0.795) N%. The dye-ability expressed as (K/S) values were measured for the (DMAE) cotton that dyed separately with disperse and direct dyes dye classes at different dyeing temperatures and different dyeing times. The obtained results verify the role of modification process in the production of modified cotton with excellent dye-ability towards disperse and direct dyes under neutral pH conditions in the absence of salt with high reactivity, giving high fixation efficiency and good dyeing quality. The work is extended to study dyeing diffusion kinetics on those sensitized and dyed cotton fabrics with direct dye and thermodynamic parameters including: % exhaustion, dyeing rate constant, half dyeing time, and diffusion coefficient and activation energy. The results of this part deduced that modification process improved the structural characteristics of the cotton fabrics via improving their behaviour for all of diffusion kinetic parameters.*

**Keywords:** *Synthesis- DMAE-cotton- Dye-ability- Diffusion kinetics- Thermodynamic parameters.*

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### **1. INTRODUCTION**

Cotton is a native cellulosic fiber can be regarded as a highly crystalline fiber, where amorphous regions are formed by the most extended chains. The molecules of a used dye cannot penetrate the highly ordered crystalline regions of native cellulose and their adsorption can only take place in the amorphous regions, which are less ordered [1,2]. Direct dye can bonded with cellulose fabric, this bonding is mainly dependent on the primary hydroxyl groups of cellulose and the resulted attraction will be weak, as the direct dye molecules are only attached to the cotton by hydrogen bonds and Van Der Waals forces, thus any aqueous solution will break these forces of attraction. Thus it is possible by modifying the ionization of the carboxyl groups in monocarboxyl cellulose, to improve its dye-ability by direct dyes [3]

Many article researches are directed to strength this attraction via one of the stated types of chemical modifications of cotton [4-6]. Disperse dyes are the only water-insoluble dyes and having the smallest dye molecules among all dyes. A disperse dye molecule is based on an azo benzene structure. Native cotton cannot be dyed with disperse dyes as this fabric does not have a cationic moiety for disperse dye attraction [7-10].

Thus the chemical composition of fibers determines, to some extent, the type of best dye to be applied. There are several types of dye-fiber interactions that are reported in the literature, as; electrostatic, Van Der Waals bonding and hydrophobic interactions. As the primary hydroxyles of cellulose elementary units are responsible for the solubility and dye-ability of cellulosic materials, thus the formation of dye-fiber hydrogen bonds is controlled and depended mainly on the presence of hydrogen bonding functional groups in the fiber and the dye[11] .

Chemical modification of cellulosic fabrics is generally achieved by blocking the functional groups (OH) that are already present in the native fiber or by introducing new active functional groups to increase the affinity and ability of cotton fibers to different dye classes. Other approaches are based on using cross-linking agents and a reactive additive that contains alcohol which enables cellulose to be grafted via the cross linking agent. The additive also contains another moiety to which the dye

molecule can have affinity because of its active charged characteristics, thus enables modified fabrics to be dyed with different dyes. On the other hand pre-quaternized cotton could be successfully dyed with disperse dyes. The main important aspects of this applied chemical modification is to obtain shorter dyeing time, lower temperature of dyeing than normal, salt or no salt to exhaust the dye bath , neutral pH fixation of modified fabrics thus saving both energy and water in dyeing industries and improving all dyeing characteristics[11].

This work aimed to evaluate the effect of amination process on the dye-ability of cotton fabrics where the resulting DMAE-cotton fabrics two different nitrogen contents (0.386, 0.795) were dyed separately with disperse and direct dye at different dyeing temperatures (30,50 and 70°C) each for different time intervals. The work was extended to study the effect of amination process on the diffusion kinetics and thermodynamic parameters of dyeing DMAE-cotton fabrics with direct dye- as an example of the applied dyes, these parameters including: % exhaustion, dyeing rate constant, half dyeing time, and diffusion coefficient and activation energy.

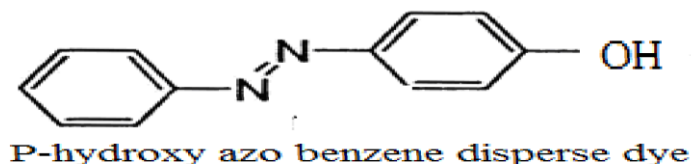
## 2. EXPERIMENTAL WORK

### 2.1. Materials and Chemicals

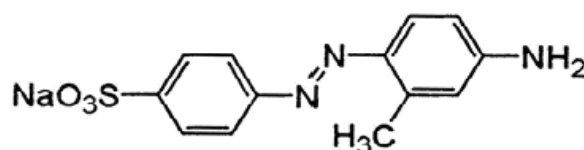
- 1) Mill desized scoured and bleached cotton fabric (Poplin) was kindly supplied by El-Shorbagy Company. The fabric was purified in the laboratory by scouring with a solution containing  $\approx 2.5$  gm/l of detergent, then thoroughly washed with tap water and dried at ambient conditions.
- 2) 2-Cholor-2-dimethylaminoethyl hydrochloride ( $\text{Cl-CH}_2\text{-CH}_2\text{-NH(CH}_3)_2\text{Cl}$ ) was kindly supplied by Fluka AG, Buch SC and used as aminated reagent in the modification process.

**Dye Stuffs:** Two types of dye classes were used:

- i) Disperse dye: P-hydroxy azo benzene disperse dye which was laboratory prepared according to the reported method [12] having the following basic chemical formula:

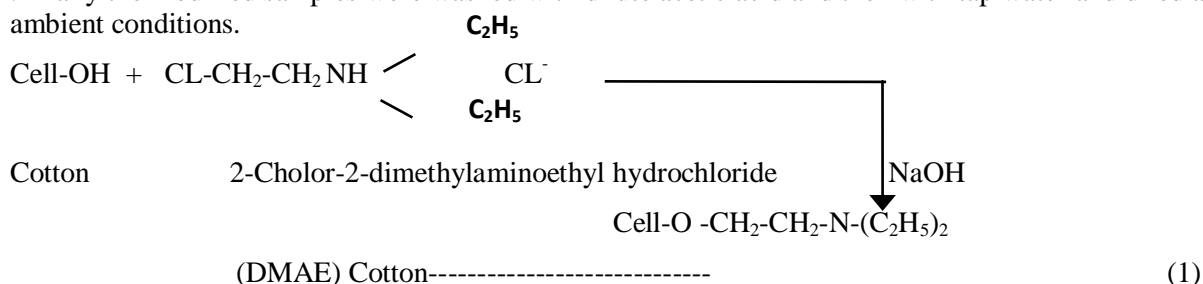


- ii) Direct dye: 2- Solophenyl Orange TGL, C.I. Direct Orange 34 supplied by Bayer Company



### Modification Process

Dimethylamino ethyl cotton (DMAE-Cotton) was prepared according to the reported Hartmann process [13] as following in equation (1). The fabrics were independently padded twice to about 100% pick up with (10 & 30%) aqueous solutions of- 2-cholor-2-dimethylaminoethyl hydrochloride , then immersed in( 4 & 12%) sodium hydroxide respectively for 10 minutes at 95°C, respectively .Finally the modified samples were washed with dilute acetic acid and then with tap water and dried at ambient conditions.



## **Characterization of the Synthetized DMAE Cotton**

The resultant DMAE cotton fabric samples were analyzed for determining the nitrogen content in their structure quantitatively using Kjeldahl Test Method [14]. The resultant DMAE cotton samples had two nitrogen contents (0.386, 0.795), were referred as samples (I&2) in the whole work respectively.

### **2.2. Dyeing Method**

#### *i) Dye-Ability*

This method of dyeing was carried out to characterize the effect of applied chemical modification on increasing the dye-ability of cotton with both disperse and direct dye. where the DMAE cotton fabrics with two different nitrogen contents (0.386, 0.795) those referred as samples (I& 2) respectively - were independently dyed according to the conventional exhaustion method in a laboratory dyeing apparatus using two dye classes namely: disperse and direct dyes using liquor ratio 1:50 and dye concentration is [5] g/l . The dyeing processes were performed at three different dyeing temperatures ;( 30, 50 and 70 °C) at different dyeing times: (1, 5, 8, 15, 20, 30 and 60) minutes each factor for each dye in a separate experiment. The dyeings were washed with a soap solution [2] g/l at 60°C for (15) minutes, rinsed with tap water and finally dried at ambient conditions.

#### *ii) Colour Strength (K/S)*

The colour strength (K/S) values for all of the examined dyed samples -based on the reflection spectra at the proper wave length – were determined [15] using the Kubelka Munk equation (2):

$$F(R) = (1-R)^2/2R = (K/S) \text{ -----} \quad (2)$$

Where, R = reflectance; K = absorption coefficient; S = scattering coefficient. The spectral reflection curves given throughout this investigation were measured using a Double Beam Recording Spectrophotometer attached by an integrating sphere (Shimadzu U.V. 300, Japan).

### **Dyeing Kinetics and Diffusion Studies**

#### *Dyes Diffusion*

This method of dyeing was applied for evaluating the role of the used amination process on enhancing the dyeing kinetics and diffusion thermodynamics of direct as an example of the used dyes. Where, DMAE cotton fabrics with two different nitrogen contents (0.386, 0.795) samples (I& 2) respectively - were dyed according to the conventional exhaustion method in a laboratory dyeing apparatus with direct dye using liquor ratio (1:50) and dye concentration was [0.5] g/l. The dyeing processes were performed at three different dyeing temperatures :( 30, 50 and 70 °C) at different dyeing times : (1,5,8,15,20, 30 and 60 ).After each time interval the dyed fabrics were removed from the dye-bath immediately and thoroughly washed two times each with 25ml cold distilled water which were then added to the remaining dyeing liquor solution.

### **2.3. Testing & Analysis**

The resultant liquors were measured spectrophotometrically to determine the concentration of the diffused dye via optical density measurements. From these data all of the diffusion kinetics parameters were calculated including: % exhaustion, dyeing rate constant, half dyeing time, diffusion coefficient and activation energy.

## **3. RESULTS AND DISCUSSION**

### **3.1. The Dye-Ability of the Examined Samples**

The dye-ability results expressed as (K/S) values, for the different examined dyed fabric samples those separately dyed with disperse and direct dyes at different dyeing temperatures and times, are shown in tables (1,2) and represented in figures (1,2) (a-c) .Higher (K/S) values obtained for the modified cotton fabrics samples (I , 2) in comparison with those of blank unmodified ones must be due to the presence of DMAE groups in the molecular structure of cotton cellulose. The extent of (K/S) increase was a function in nitrogen content of quaternized cotton samples (I , 2) having nitrogen contents (0.386, 0.795) respectively caused an increment in the ratio of the cationic site to the anionic dyes applied.

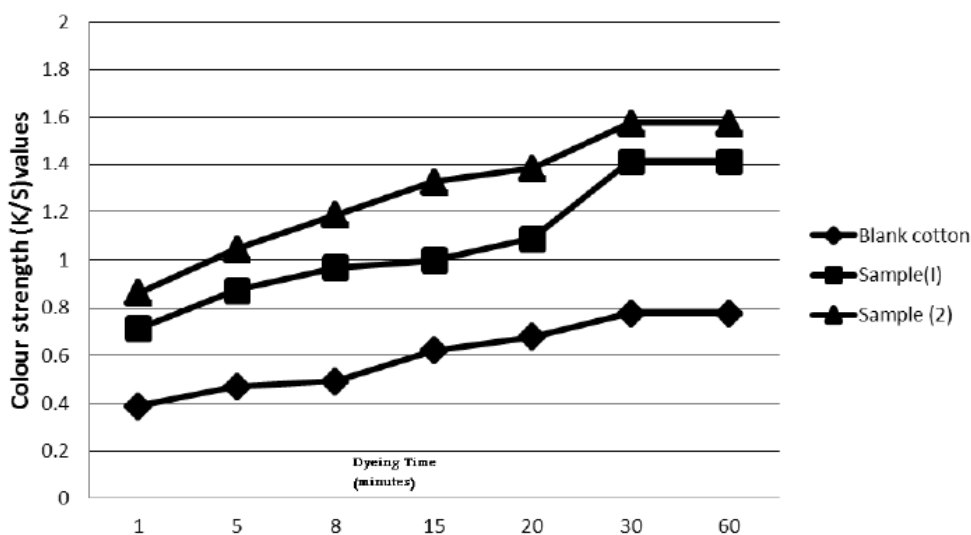
The introduction of (DMAE) groups in the molecular structure of cotton fabric leading to an increase in cellulose swellability by opening up its structure, thus enhancing the rate of dye adsorption, diffusion, and penetration into the modified fiber pores [16-18]. Moreover, DMAE groups act as a built-in catalyst in the molecular structure of cotton cellulose by creation of positive moieties those enable complete ionic attraction with these applied dyes.

In addition, the dye-ability of the different tested cotton samples increased by increasing both of dyeing time and dyeing temperature, regardless of the sample nature [19]. Where, high diffusion efficiency is attained when a maximum penetration of the dye into fibrous material is achieved at a shorter period of dyeing, this in turn depends on the chemical properties of the dye liquor, its temperature and the state of fibrous material ,i.e., its swelling properties .

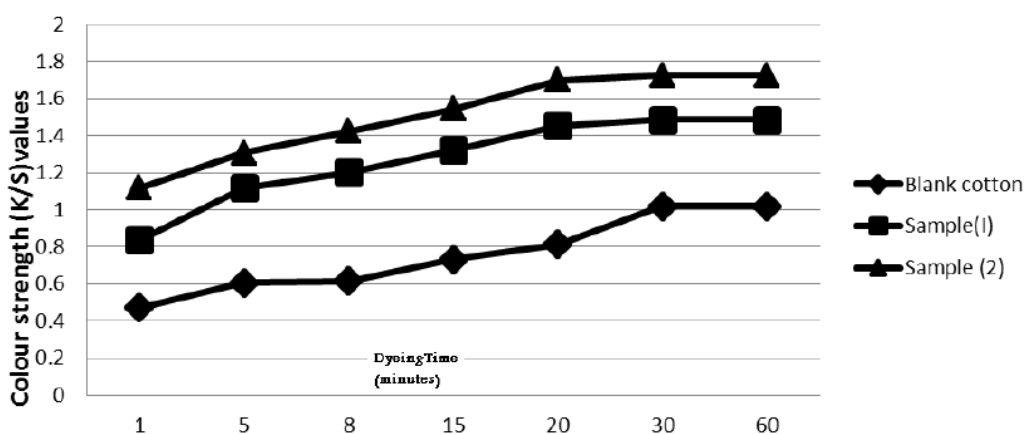
**Table1.** Colour strength (K/S) values of the examined samples dyed with direct dye at different dyeing times and temperatures.

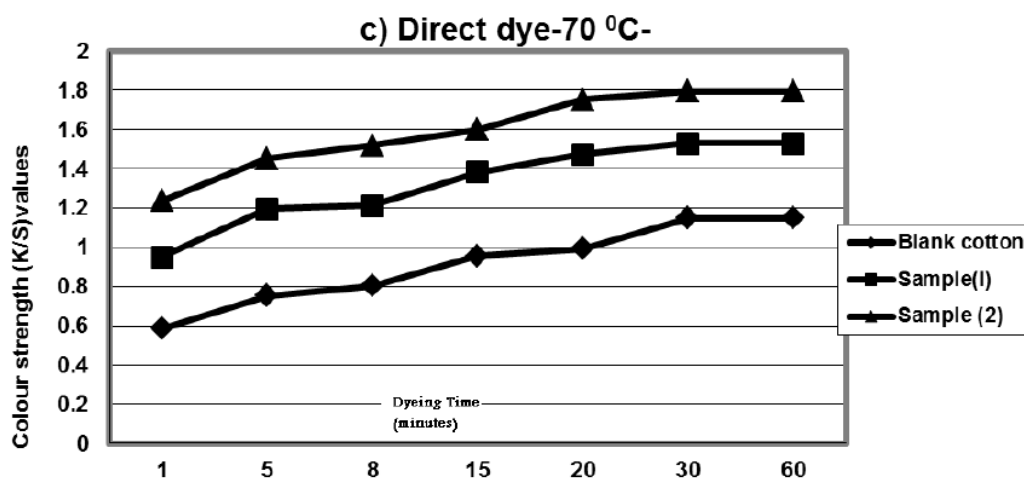
Dyeing Time (minutes)	Colour strength (K/S) values of:								
	Blank Samples at temp. °C=			Samples(1) with N% = 0.386 at temp. °C=			Samples (2) with N%=0.795 at temp. °C=		
	30	50	70	30	50	70	30	50	70
1.0	0.387	0.472	0.586	0.714	0.837	0.950	0.864	1.118	1.237
5.0	0.472	0.607	0.754	0.875	1.118	1.195	1.046	1.308	1.454
8.0	0.494	0.614	0.802	0.968	1.203	1.212	1.188	1.423	1.519
15.0	0.622	0.731	0.956	1.00	1.323	1.383	1.325	1.542	1.600
20.0	0.680	0.812	0.993	1.088	1.454	1.475	1.383	1.700	1.754
30.0	0.777	1.019	1.148	1.413	1.486	1.530	1.576	1.727	1.795
60.0	0.777	1.019	1.148	1.413	1.486	1.530	1.576	1.727	1.795

a) Direct dye- 30 ° C-



a) Direct dye- 50 ° C

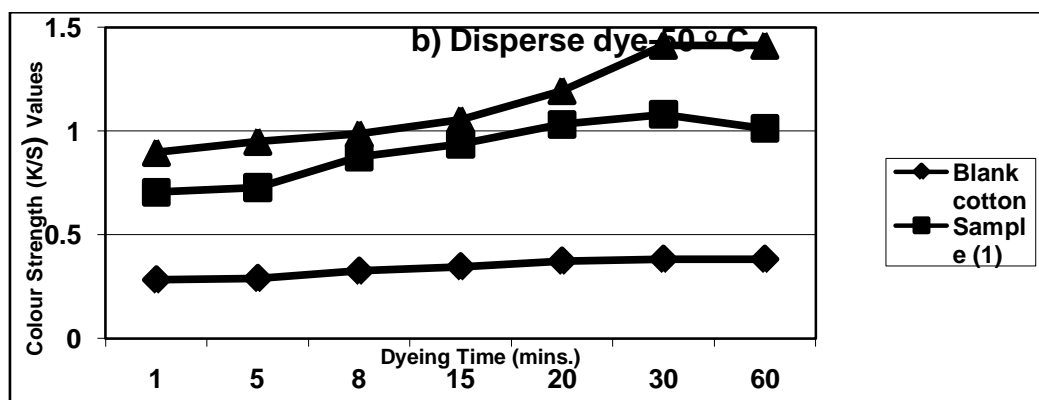
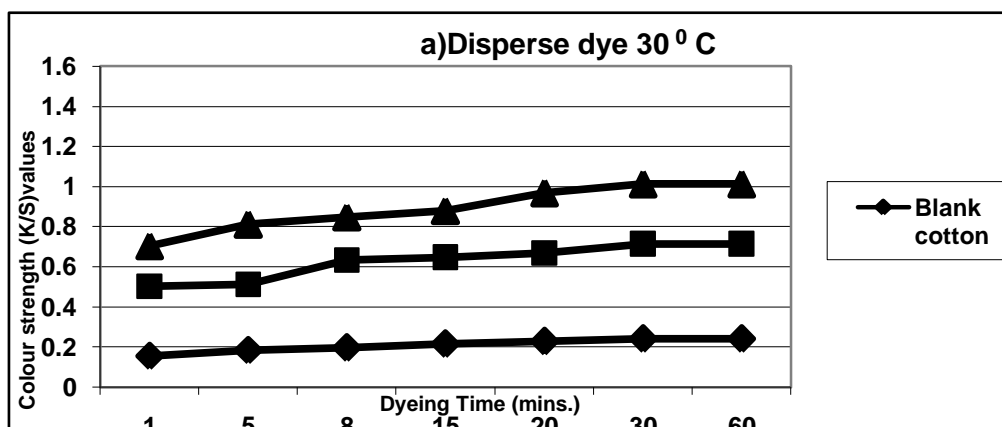


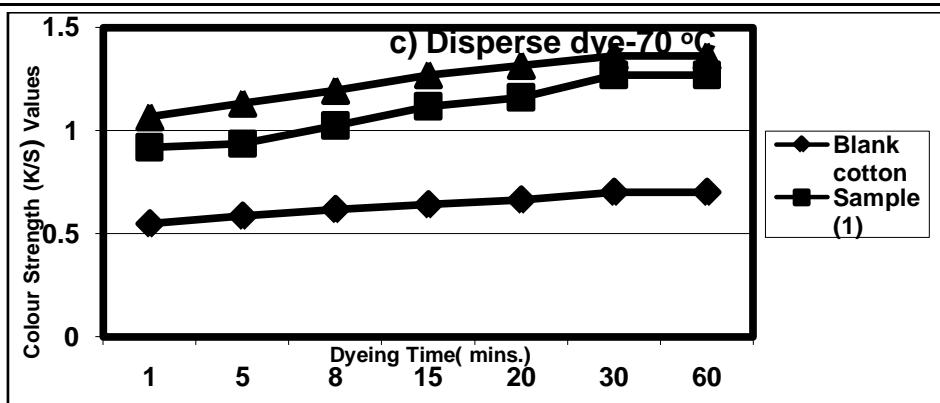


**Figure(1)a-c:** Colour strength (K/S) values of different examined samples dyed with direct dye at different dyeing times and temperatures

**Table2.** Colour strength (K/S) values of the examined samples dyed with disperse dye at different dyeing times and temperatures

Dyeing Time (minutes)	Colour strength (K/S) values of:								
	Blank Samples at temp. °C=			Samples (1) with N% = 0.386 at temp. °C=			Samples (2) with N%=0.795 at temp. °C=		
	30	50	70	30	50	70	30	50	70
1.0	0.156	0.283	0.549	0.503	0.705	0.920	0.705	0.897	1.067
5.0	0.185	0.290	0.586	0.512	0.727	0.938	0.812	0.950	1.133
8.0	0.196	0.325	0.618	0.633	0.875	1.026	0.848	0.987	1.195
15.0	0.215	0.346	0.641	0.648	0.938	1.118	0.880	1.00	1.271
20.0	0.227	0.373	0.664	0.668	1.195	1.163	0.968	1.033	1.316
30.0	0.240	0.382	0.701	0.714	1.413	1.271	1.013	1.081	1.363
60.0	0.240	0.382	0.832	0.714	1.413	3.026	1.013	1.013	1.521





Figure(2)a-c: Colour strength (K/S) values of different examined samples dyed with disperse dye at different dyeing times and temperatures.

### 3.2. Dyeing Kinetics and Diffusion Studies of Direct Dye on DMAE-Cotton

The dyeing kinetics investigation including: % exhaustion, time of half dyeing, diffusion coefficient, activation energy and dyeing rate constant. These parameters are investigated and calculated for the examined samples that dyed with direct dye class for different dyeing time intervals (1, 5, 8, 15, 20, 30 and 60 minutes), at different temperatures (30, 50 and 70°C) in order to evaluate the role of the used amination process on improving these diffusion thermodynamics parameters.

#### 3.2.1. Dye Exhaustion

The percent exhaustion values of all dyed samples were calculated by using equation (3) below while the data are given in table (3) and represented through figures (3)(a-c):

$$\% \text{ Exhaustion} = 100 \times \frac{C_i - C_t}{C_i} \text{ , -----} \tag{3}$$

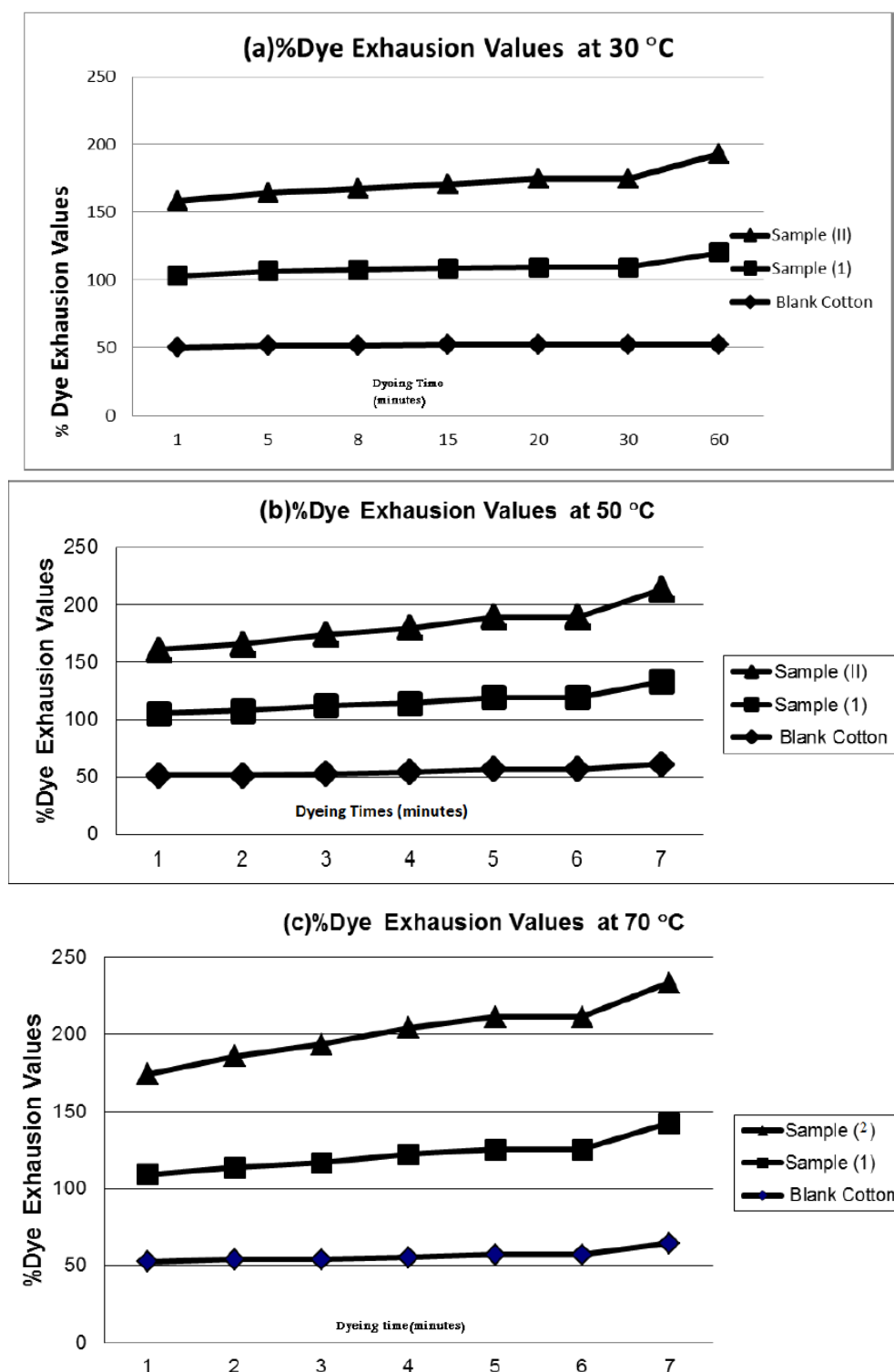
Where:  $C_i$  and  $C_t$  are the initial and final dye- bath concentrations at different dyeing times respectively.

Generally, it was clear that, there was a gradual increase in the percent exhaustion values by increasing both the dyeing time and dyeing temperature. In addition the percent increase in dye exhaustion for the examined samples after 60 minutes at 70°C was (22.6, 38.7 and 39.55%) for blank, sample (1) and sample (2) respectively with respect to their mates at the first dyeing time. Thus samples (2) with the highest nitrogen percent recorded the highest percent increase in dye exhaustion. These results can be interpreted by the time adsorption curves [figures 3 (a-c)] which provided a complete description of the rate of adsorption of the dye under the employed dyeing conditions depending on two factors [1,2]: equilibrium position and the rate at which equilibrium is attained. In addition to the facts that:

- a) The % exhaustion may be determined by the rate at which the dye can arrive at the fibre surface and not at all diffusion inside the fibre.
- b) Increasing the temperature of dyeing can facilitate: i) the transfer of the dye molecules from the dye liquor to the fibre surface, ii) the adsorption of the dye at the fibre surface, and iii) the diffusion of the adsorbed dye from the surface into the pores of the substrate . c) The presence of DMAE- groups in the molecular structure of cellulose increases the moisture regain, wettability and dye attraction and penetration, thus the solvated dye molecules can easily penetrate into the fibre pores

Table3. Percent dye exhaustion values at different dyeing times and temperatures of different examined samples dyed with direct dye.

Dyeing Time (minutes)	Percent dye Exhaustion Values of:								
	Blank Samples at temp. °C=			Samples(1) with N%= 0.386 at temp. °C=			Samples(2)with N%=0.795 at temp. °C=		
	30	50	70	30	50	70	30	50	70
1.0	50.00	52.0	52.60	53.10	54.00	56.30	55.30	55.70	65.20
5.0	51.30	53.76	53.90	54.90	56.60	59.60	57.60	57.60	72.30
8	51.30	55.1	53.90	56.10	59.50	62.90	59.60	61.80	76.80
15	52.50	56.41	55.30	56.10	60.10	66.90	62.10	65.40	81.90
20	52.50	59.05	57.20	56.70	62.70	68.20	65.60	69.90	85.80
30	52.50	56.90	57.20	56.70	62.70	68.20	65.60	69.90	85.80
60	52.50	61.10	64.50	67.70	72.20	78.10	72.50	80.30	90.97



Figure(3)a-c: Percent dye exhaustion values at different dyeing times and temperatures of different examined samples dyed with direct dye

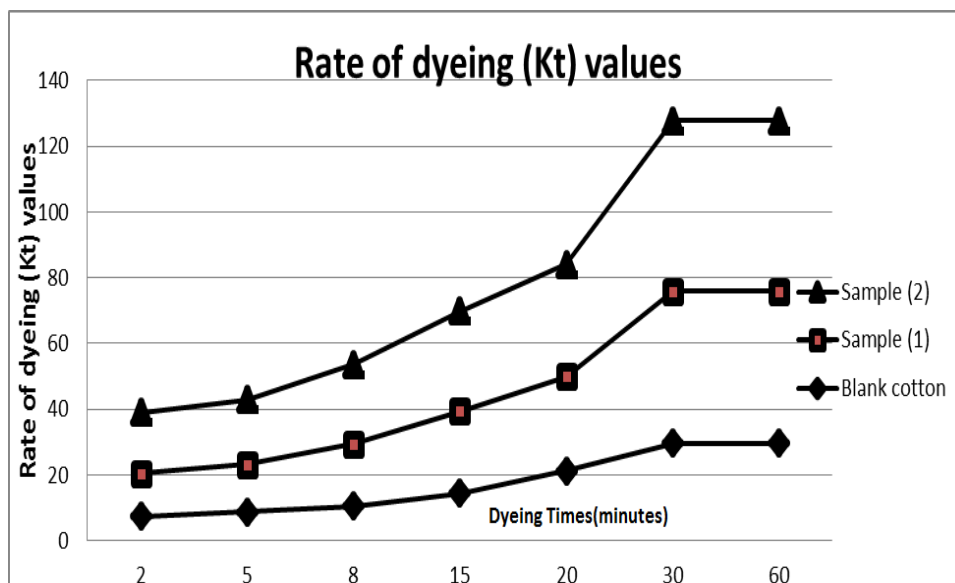
### 3.2.2. Rate of Dyeing (Kt)

Dyeing rate can be defined as the weight of dye adsorbed on the surface of the fabric per unit time at maximum dyeing temperature using equation (4), which often gives a fairly good fit to rate of dyeing curves [20]:

$$K_t = (1/ C_t - C_t) - (1/ C_i) \text{-----} \quad (4)$$

Where  $C_t$  = is the percentage of dye adsorbed on the fabric at time (t) and  $C_i$  = is the percentage of the initial dye-bath concentration and  $K_t$  is an arbitrary velocity constant.

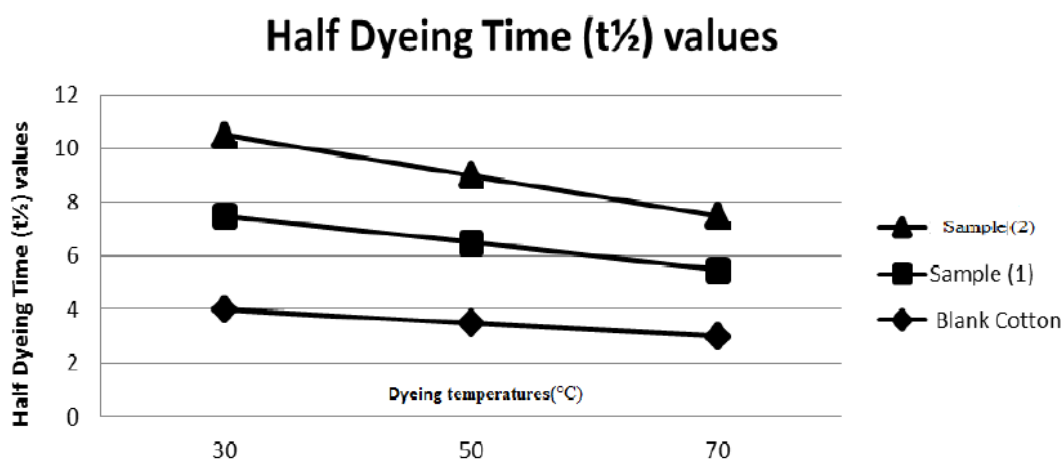
We compared time adsorption isotherm for the examined at maximum dyeing temperature (70°C) for different dyeing time intervals and the data as represented through figure (4). The results showed that, the dyeing rate increases with the progression of the dyeing time and that the modified cotton samples (2) showed the highest dyeing rate values and both results agreed well with the % exhaustion results discussed before.



**Figure(4):** Rate of dyeing (Kt) values of direct dye at different dyeing Times and maximum dyeing temperature (70 °C).

### 3.2.3. Determination of Half Dyeing Time ( $t_{1/2}$ )

The half-dyeing time ( $t_{1/2}$ ) is regarded as the time required for the fibre to adsorb half as much dye as it will adsorb in the equilibrium state [21]. Figure (5) showed the variation of ( $t_{1/2}$ ) values of the examined samples with dyeing temperatures. Generally, it is observed that, ( $t_{1/2}$ ) values decrease with rising in dyeing temperature. Also, the obtained results indicate that, the amination process lowered the time of half dyeing due to its effect explained before, and modified cotton samples (2) showed the lowest ( $t_{1/2}$ ) values. This result agreed well with the previously discussed dye exhaustion results.



**Figure(5):** Half Dyeing Time ( $t_{1/2}$ ) values of different examined samples dyed with direct dye at different dyeing temperatures

### 3.2.4. Diffusion Coefficient (D) within the Fibre

The diffusion coefficient (D) is numerically equal to the amount of the dye diffusing in unit time across unit area of the fibre under a unit concentration gradient and can be calculated from the following mathematical equation (5):

$$D = Ct / C_{\infty} \cdot d^2 \times 100 / t \text{-----} \tag{5}$$



## Characterization of Dye-Ability and Diffusion Kinetics of Chemically Modified Cotton Fabrics towards Disperse and Direct Dyes

Where  $C_{\infty}$  : is the percentage of dye adsorbed on the sample at equilibrium conditions between the sample and the dye-bath divided by the weight of the sample,  $C_t$  = is the percentage of dye adsorbed on the sample at time (t) and (d) is the fibre diameter.

The diameters of blank, sample (1) and sample (2) were: (0.4025, 0.4080 and 0.4500mm) respectively. Table (5) showed that the diffusion coefficient values decreased with the increase of the dyeing time, while these (D) values increased with the rising of dyeing temperature for all samples under test. Also, the modified cotton sample (2) had the highest (D) values than the other samples. These results can be interpreted in the facts that: i) the dissolved dyes are exhausted at the first few minutes of dyeing, supposing that the solute molecules remain vibrating about an equilibrium position until acquiring a certain critical energy, to be able to overcome the restrain of their surroundings and move or diffuse [4]. ii) The role of modification process in enhancing the diffusion coefficient, where the introduction of DMAE-groups into cellulose structure increases its amorphosity via increasing its moisture regain and wettability.

**Table5:** Diffusion coefficient (D) values in [ $Cm^2 \cdot Sec^{-1}$ ] of the dyed samples with direct dye at different dyeing times and temperatures.

Dyeing time (minutes)	Values of (D X 10-4) of :								
	Blank Samples at temp. °C=			Samples(1) with N% = 0.386 at temp. °C=			Samples (2) with N%=0.795 at temp. °C=		
	30	50	70	30	50	70	30	50	70
1.0	28.0	33.0	35.5	41	47	57	61	87	148
5.0	5.4	6.7	6.9	7.9	8.6	16.5	12.0	17	24
8	3.4	4.03	4.3	4.8	5.2	6.0	6.9	9.3	12
15	1.8	2.1	2.2	2.6	2.7	2.9	3.4	4.5	5.1
20	1.3	1.5	1.6	1.9	1.9	2.1	2.4	1.9	3.0
30	1.2	1.4	1.4	1.2	1.4	1.4	1.2	1.4	1.4
60	0.44	0.44	0.44	0.47	0.47	0.47	0.64	0.47	0.64

### 3.2.5. Activation Energy of Diffusion (ED)

The activation energy may be regarded as the energy which the dye molecule might acquire in order to be able to move within the fibre matrix, they are then in an activated state and are able to overcome, the restrain of their surroundings and move or diffuse [22]. Thus, it can be calculated using equation (6):

$$2.303 \log D_T = - E_D / RT + \text{constant} \text{-----} \quad (6)$$

Where  $D_T$  is the observed diffusion coefficient at a temperature T, R is the universal gas constant, and  $E_D$  is the activation energy.

Thus by plotting the logarithm of the observed diffusion coefficient at different temperatures against the reciprocal of the absolute temperature, the slope of the obtained curves gives the value E/R, from which the activation energy of diffusion was calculated.

The data of the calculated  $E_D$  values are given in table (6). These results indicated that, the activation energy values for the examined samples followed the order: samples (2) > samples (1) > blank samples, which agreed well with the order of diffusion coefficient discussed before.

**Table6:** Activation energy values ( $E_D$ ) in (KJ/ Mole/ K) of different examined samples dyed with direct dye

Samples Name.	(ED) in (KJ/ Mole/ K).
Blank Cotton	1.91
Samples(1) cotton with N% = 0.386	2.41
Samples (2) cotton with N%=0.795	2.80

## 4. CONCLUSION

The obtained results verified our search goals that were:

- 1) Improving dye-ability by chemical modification: the DMAE –cotton samples had noticeable dye-ability with both disperse and direct dyes, where the presence of DMAE groups in the molecular structure of cellulose act as a built-in catalyst, thereby creating a cationic site on the fabrics to increase the attraction of the anionic dye. Also, DMAE increases the swellability of the fibers

(i.e., increases pore size), thereby encouraging both diffusion and aggregation of dye particles inside the fiber pores, i.e., better dye-ability, especially at higher nitrogen content, and at shorter dyeing times.

- 2) Also, the dyeing kinetics and thermodynamic parameters of direct dye as an example of the used dyes were enhanced by chemical modification via improving the structural characteristics of cotton fabrics as a result of the quaternization process.

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