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Optimizing the Dyeing Process of Alkali-Treated Polyester Fabric with Dolu Natural Dye

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Abstract
An attempt has been made to optimize the process of dyeing polyester (PET) fabric with natural dyes. Polyester has been first treated with NaOH solution in order to study its impact on the dyeability to the natural dye. The required and used colour component was extracted from a natural plant, namely: Rhubarb; Rheum officinale. The chemical structure of the used colouring matter is observed to have all the characteristics of a typical disperse dye.
The colour strength of PET fabric was noticed to increase as a result of alkali treatment which may be attributed to the alteration in the hydrophobicity of polyester fibre. The most effective parameters that may affect the dye uptake of the natural dye on alkali-treated PET fabric and the final dyeing properties including dye bath pH, dyeing temperature and time were studied in details.
The behaviour of the selected and used natural dye was found to be similar to that of disperse dyes. Addition of salicylic acid to the dye bath accelerated the rate of dyeing and subsequently higher colour strength was attained. Non- ionic dispersing agent was also used to ensure better dyeing uniformity and higher dispersion stability of dye liquor.
It is obvious from this study that natural dyes can produce bright colour hues and colour fastness properties equivalent to those of synthetic disperse dyes. Thus, natural dyes may be considered as an eco- friendly alternative to the synthetic ones.
Key words: Polyester, natural dye, Rhubarb, dyeing, alkali- treated polyester.

I. Introduction

Synthetic fibres, in general, are dyed with disperse dyes. Disperse dyes, as most of the synthetic dyes, especially the azo dyes (-N=N-) have deleterious environmental consequences, and contribute to pollution and waste disposal problems. (1)

The dyes are produced from intermediates and chemicals which are toxic and hazardous to human health as well as to the environment. As a result, a great interest in realization of clean industry and using a safe and eco- friendly alternatives to synthetic dyes has been generated during the recent years. (2) In line with this trend, there is now an increasing interest for using natural dyes for colouring both natural and synthetic fibres. (3)

A number of investigations have been reported on the application of natural dyes on wool (4-7), silk (8-11), and on cotton fibres (12-16). However, limited studies are made on the application of natural dyes on synthetic fibres (17-22). On the other hand, intense studies were made for improving the physical properties of polyester fibres especially in the trend of increasing the water absorption capacity as well as improving the electric properties (minimizing static charges accumulation on fibre surface). Alkaline treatment of polyester with aqueous solutions of sodium hydroxide and/or amine compounds was found to enhance the physical properties of polyester and improve its appearance to be silk- like fibres. Therefore, many various studies were made to optimize the alkaline treatment conditions in order to realize the desired qualities (23-26).

In the present study, a natural dye (Rhubarb) is selected to study its behaviour during the dyeing of alkalized polyester fibres and optimizing the dyeing conditions.

II. Experimental

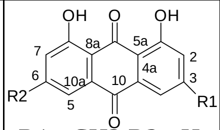
Materials:

Fabric

Scoured and bleached polyester fabric weighting 200 g/m^2 was used for this study.

Dyes

Chinese Rhubarb "Dolu" (*Rheum officinale*) was obtained from the Agricultural Seeds Medicinal and Medical Plant Company (Harraz), Cairo, Egypt.

English name	**Botanical name**	**Part used**	**Chemical structure**	**C.I No.**
Chinese Rhubarb	*Rheum officinale*	Roots	 R1= CH3,R2= H	Natural yellow 23

Chemicals

Sodium hydroxide for (alkali treatment of polyester), Tanasperse OH (dispersing agent) and Tanaterge FTD-N (anionic detergent) from SYBRON TANATEX Company and salicylic acid as (an accelerator) were used throughout this investigation.

Procedures:

Alkali Treatment

Polyester samples were treated with aqueous solution of NaOH (12%) at L.R. 1:50 at different degrees of temperatures (50-80° C) and for various durations (30-120 min.). The treated samples were, then, rinsed with hot and cold water.

Extraction of the dye

The dye was extracted from rhubarb plant regarding the following method:

-10 gm of plant (powder) + 100 ml water.

-Boiling for 1 hour then the filtration process takes place.

After filtration the extracted solution was placed in a cold place and also a little amount of alcohol was added to the extract in order to prevent the appearance of bacteria in the extracted solution until its usage.

Dyeing

The dyeing process was performed according to the following conditions:

-L.R 1:100
-50% of the dyeing solution is the dye extract.
-7 g/l salicylic acid.
-pH value was about 2.5 after acid addition.

The dyeing process started at about 50 ° C then the temperature was gradually raised to 95℃ during 10 minutes, after which the dyeing process continued for 75 min.

Washing

The dyed samples were washed in order to remove the excess dye from the fibre surface . The soaping solution contained 2 g/l detergent and the process was carried out using liquor ratio 1:100 at 60° C for 15 minutes. The samples were then rinsed with hot and cold water and finally air- dried.

Colour Measurement

The colour strength (K/S) of the dyed samples were measured using spectrophotometer, model I.C.S., made by Texicon Ltd., Kennetside Park, New bury, Berkshire, UK. The K/S values were obtained directly according to the equation:

$$K/S = (1-R)^2 / 2R$$

Where K and S are constants associated with the light absorption and scattering of the fabric respectively. R is the reflectance of the dyed fabric measured at the wavelength of maximum light absorption expressed in fractional form.

Fastness Properties Determination

The fastness properties of the dyed samples were determined by the following tests prescribed by ISO and AATCC standards:

Washing fastness **ISO 105 C01**
Light fastness **ISO 105 B04**
Rubbing fastness **AATCC Test Method No.8**
Perspiration fastness **AATCC Test Method No.15**

III. Results and Discussion

(i) Optimization of alkali treatment

The treatment of polyester fabric in alkali medium is a common modification process for producing a fabric with desirable qualities. Alkali treatment of polyester fabric could produce a fabric with better performance. The results show that alkali treatment with NaOH at the optimum temperature and time could hydrolyze the polyester fiber surface and the fiber changes to a soft cloth with draping, and may also improve some of the fabric properties, such as fabric regain, water absorbency, and fabric pilling. [27, 28]

The dyeing and resultant colour properties of alkali treated polyester would be expected to change as progressively more material is hydrolytically removed from the fiber surface. Earlier studies have shown that alkali etched polyester tends to be dyed more deeply and to slightly different shades than does untreated polyester. [29]

The present work tries to take advantage of this treatment to enhance dyeability of natural dyes towards polyester fabric, and factors affecting this treatment were investigated in order to achieve better colour strength of the dyed samples keeping the weight loss in the range of not affecting the tensile strength of the treated samples.

1- Effect of treatment temperature:

The temperature of the treatment bath plays a great role, and to determine the suitable temperature that achieve maximum colour strength of the after dyed samples, taking into consideration its influence on the rapidity of the treatment and eventually on the weight loss, polyester samples were treated in sodium hydroxide solution as mentioned before for 30 minutes at different temperatures. The K/S of the alkali treated and dyed PET samples are illustrated in table (1).

Temperature	Weight loss%	K/S	Increase in K/S (%)
50°C	~ 0.54	4.09	9.06
60°C	~ 3.00	4.13	10.13
70°C	~ 4.00	4.21	12.27
80°C	~ 8.00	4.42	17.87

Table (1): The relationship between the treatment temperature and the K/S of the treated polyester fabric dyed with dolu extract.

From table (1), it can be noticed that the colour strength increased as a result of increasing the temperature which reaches its maximum value (4.42) at 80° C. These results were expected as polyester only starts to saponify to a larger extent above the glass transition temperature which is between 80 and 90° C with the usual fiber types. [30] It must be mentioned that the K/S of the untreated PET samples (control) is (3.75).

2- Effect of treatment duration:

To study the importance of this factor and its impact on the alkali treatment and hence the weight loss of polyester fabric, the alkali treatment process was carried out for different durations i.e. (30,60,90 and 120 minutes) at 70° C. The alkali treated PET samples were then dyed with dolu extract under definite conditions, washed and air- dried. The K/S were then, measured and the received data are shown in table (2).

Time	Weight loss%	K/S	Increase in K/S (%)
30 min.	~ 4.00	4.21	12.27
60 min.	~ 9.40	4.32	15.20
90 min.	~ 14.13	4.53	20.80
120 min.	~ 20.30	4.76	26.93

Table (2): The relationship between the treatment time and the K/S of the treated polyester fabric dyed with dolu extract.

It is well observed from table(2) that the colour strength of the dyed samples increases by increasing the treatment time, and it reaches its maximum value when performing the alkali treatment for 120 minutes, and it is worth mentioning that the weight loss was kept within limits.

It may be concluded, from the previous results, that there is a relationship between degree of alkaline surface modification of PET fibres and their dyeability to the applied natural dye extract (dolu). Alkaline treatment of PET fibres results in a substantial surface modification with formation of such a number of holes as a result of alkaline hydrolysis of PET polymer chains at the fibre surface [31]. Increasing the porosity of fibre surface will thus accelerate the rate of dye diffusion (dolu) inside the fibres and as a result, higher colour strength is attained.

(ii) Optimization of dyeing conditions

The initial studies showed that it was indeed possible to dye nylon and polyester with natural dyes in deep and fast colours [32], and to insure good performance it was better to choose a dye that resembles synthetic disperse dyes. Dolu was the dye that extracted from the rhubarb plant and it was evident that the dyes present in materials such as henna, juglone, madder and rhubarb had molecules which had all the characteristics of a typical disperse dye. [32]

Dolu is one of the dyes based on a quinone molecule (hydroxy anthraquinone) [33] and the values of standard affinity quite high compared to those for most synthetic dyes indicating a high affinity of the dye for polyester. [32]

The main colour component of Rhubarb roots is the chrysophanic acid which is present as the glycoside chrysophanein, C_{12} H_{20} O_9, together with emodin -3- monomethylether and rhein. [34]

In the present study, emphasis has been given on optimization of conditions for dyeing alkalized PET fibres with dolu natural dye. All the factors that may affect the dyeing process were studied in details.

1- Effect of dispersing agent concentration:

In order to disclose the function of dispersing agent in the natural dyeing bath with dolu, the dispersing agent was added to the bath at different concentrations i.e. (0, 0.5, 1, 1.5, 2 g/l). The results of K/S measurements of dyed samples are shown in table (3).

Dispersing agent conc.	K/S
0.0 g/l	3.36
0.5 g/l	3.20
1.0 g/l	3.21
1.5 g/l	3.47
2.0 g/l	3.39

Table (3): The relationship between the dispersing agent concentration and the K/S of the treated polyester fabric dyed with dolu extract.

Dyeing conditions: 50% dye extract , temp. 100° C, time: 60 min., L.R 1:100

It is clear from table (3) that dispersing agent is not playing a significant role in increasing the colour strength since its essential role in the dyebath is to help maintain dispersion stability under dyeing conditions and to assist level dyeing.

2- Effect of dyeing accelerator concentration:

Employment of carriers is a familiar method for increasing the speed of diffusion of dyes in hydrophobic fibers. Their particular feature is that they provoke loosening of the fiber microstructure. [35]Salicylic acid was selected as an accelerator or as a carrier to be added to the dyeing bath to facilitate the dyeing process; especially that the dyeing process was carried out under the boiling point.

Table (4) indicates the effect of salicylic acid concentration on the colour strength obtained.

Salicylic acid conc.	K/S	Increase in K/S (%)
without	3.36	-
3.0 g/l	3.47	3.27
5.0 g/l	3.51	4.46
7.0 g/l	3.82	13.69
10 g/l	4.30	27.98

Table (4): The relationship between salicylic acid concentration and the K/S of the treated polyester fabric dyed with dolu extract.

Dyeing conditions: 50% dye extract, temp. 100°C, time: 60 min., L.R 1:100

It can be concluded from the data in table (4) that colour strength of the pretreated fabric increases by increasing salicylic acid concentration up to 10 g/l. The maximum colour strength (4.30) was obtained using 10 g/l of salicylic acid, since the colour strength is found to increase by about 28% compared to that sample dyed without using salicylic acid.

Dyeing assistants or carriers are playing an essential role in dyeing synthetic fibres especially when the dyeing process is carried out under atmospheric pressure. Salicylic acid acts as a carrier or plasticizing agent for PET fibres resulting in increasing the number and size of voids within which the molecules of dolu natural dye can diffuse easily and faster inside the molecular structure of PET fibres.

3- Effect of dyeing pH:

The effect of dyeing pH was investigated in order to achieve maximum colour strength of the dyed samples. The results are presented in table (5).

Dyeing pH value	K/S
4	3.54
5	3.27
6	2.93
7	2.62
8	2.34

Table (5): The relationship between dyeing pH value and the K/S of the treated polyester fabric dyed with dolu extract.

Dyeing conditions: 50% dye extract, 7 g/l salicylic acid, temp. 100°C, time: 60 min., L.R 1:100

It is obvious that the lower the pH value, the more colour strength attained. A gradual decrease in K/S is noticed as a result of increasing the pH of dyeing from pH 4 to pH 8, since the (%) reduction in K/S reached about 33.9%. This result may be attributed to the effect of pH of dyeing bath on the state of functional groups in both alkalized PET fibre and dolu natural dye.

Increasing the pH of the dyebath enhance the tendency of carboxyl groups in alkalized PET fibres and the aromatic hydroxyl groups in the dye molecules to ionization, resulting in increasing the electrical repulsion between fibre and dye. Thus, the rate of dyeing will be effectively reduced and the magnitude of decreasing in dye up take will depend to great extent on the degree of ionization of both –COOH and
-OH groups in fibre and dye respectively.

In the aforementioned factor, the pH of the dyeing bath reached
~ 2.5 by using 7 g/l salicylic acid and the K/S obtained was (3.82). When comparing this result with the results based on table (5), it is better to add salicylic acid to the dyeing bath without adjusting the pH value.

4- Effect of dyeing temperature:

To study the importance of this factor in the dyeing process, the pretreated samples were dyed at various degrees of temperature i .e. (60, 70, 80, 90, and 100°C), after which the K/S of the dyed samples was measured.

Table (6) illustrates the influence of the dyeing temperature on the colour strength of the dyed samples.

Dyeing Temperature	K/S
60˚C	0.93
70˚C	2.62
80˚C	2.86
90˚C	6.84
100°C	6.54

Table (6): The relationship between dyeing Temperature˚C and the K /S of the treated polyester fabric dyed with dolu extract.

Dyeing conditions: 50% dye extract, 7 g/l salicylic acid, time: 60 min., L.R 1:100

It is well observed from table (6) that the K/S of the pretreated samples dyed with dolu extract increases by raising the dyeing temperature, especially when raising the temperature from 80 to 90˚C since there was a remarkable increase in the colour strength. The (%) increase in K /S is found to reach about 139.2% as a result of raising dyeing temperature from 80 to 90˚C.

Referring to the action of temperature , below 90˚ C, on the colour strength of alkalized PET samples , one can easily notice its relatively slight effect since the (%) increase in K /S as a result of raising dyeing temperature from 70˚C up to 80˚C not exceed about 9%. This result confirms that dyeing of PET fibres is effectively performed when temperature exceed 80˚C to reach at least 90˚C. This behavior of dolu natural dye under the influence of dyeing temperature is similar to that of synthetic disperse dyes.

This could be due to the possibility of existence of dye molecules in an aggregated form at lower temperatures and the solubility will increase at higher temperatures of dyeing. [33] Besides, the carrier is capable of penetrating from boiling solutions inside the fibers. [35]

On the other hand, temperature plays a very effective role in determining the state of the molecular polymer chains, by raising the temperature the movement speed of segment polymer chains is increased resulting in such reduction in glass transition temp. (Tg) as well as dyeing transition temp. (Td). As a result, the number and volume of voids will be greater and the diffusion of dolu dye molecules will be easier and faster leading to higher amount of dyes inside the fibre and consequently higher colour strength is achieved.

These are expected results since the mechanism of dyeing, i.e. solid solution, is similar to that of dyeing hydrophobic fibers with anthraquinone -based disperse dyes, and it is well known that the rate of dye uptake, as well as the total dye uptake, increases with an increase in temperature [36], since the dyeing process is endothermic. [5]

5- Effect of dyeing time:

The dyeing time and its influence on the obtained colour strength was also investigated in order to achieve maximum colour strength.

The pretreated samples were dyed with dolu extract at boiling for different durations i.e. (30, 45, 60, 75 min.) and the data is presented in table (7).

Dyeing Time	**K/S**
30 min.	4.49
45 min.	5.38
60 min.	6.54
75 min.	7.09

Table (7): The relationship between dyeing time (min.) and the K/S of the treated polyester fabric dyed with dolu extract.

Dyeing conditions: 50% dye extract, temp. 100̊ C, pH=2.5, L.R 1:100

From table (7) one can notice that the colour strength increases with increasing the duration of the dyeing process, since maximum K/S is attained after 75 min. These are predictable results since prolonged dyeing time gives the dye the opportunity to penetrate and diffuse inside the PET fibres attaining maximum dye absorption capacity when reaching the dynamic dyeing equilibrium state.

(iii) Colour fastness determination:

The treated and untreated samples dyed with dolu extract, using optimum conditions were subjected to fastness tests such as rubbing, washing, perspiration, and light. All were evaluated by the visual assessment and the received results are illustrated in table (8).

N.B.: 1- (*) refers to staining on polyester fibres and (**) refers to staining on cotton fibres.

2- The samples were exposed to the artificial light (xenon) for 50 hours.

Table (8): Colour fastness properties of the treated and untreated samples dyed under the optimum dyeing conditions.

Fabric	**Rubbing**		**Washing**			**Perspiration**						**Light**
						Acid			**Alkali**			
	Dry	Wet	Alteration	Stain		Alteration	Stain		Alteration	Stain		
				*	**		*	**		*	**	
Untreated	**4/5**	**4/5**	**4/5**	**5**	**5**	**4**	**4**	**4/5**	**4/5**	**4**	**4/5**	**7/8**
Treated	**4/5**	**4/5**	**4/5**	**5**	**5**	**4**	**4/5**	**4/5**	**4**	**4**	**4**	**7/8**

The results in table (8) reveal that the dolu natural dye has very good colour fastness properties on both treated and untreated polyester fibres, and this proves that some natural dyes could be good alternatives to those harmful synthetic ones.

IV. Conclusion

This research paper is aiming to make use of the double effect of the alkali treatment of polyester fabric with NaOH along with the use of carrier (salicylic acid) to enhance the colour strength of the dyed materials to reach its maximum value.

Dolu natural dye extracted from Rhubarb plant can be used for dyeing polyester producing a yellow shade, given acceptable fastness properties (without using mordants) which are good enough comparing with those of synthetic disperse dyes.

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