

## *Optimization of Dyeing of Cationized Cotton Fibers with Safflower Extracts*

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### ***Abstract:***

**The** behaviour and dyeing properties of natural dyes extracted from safflower plant; *Carthamus tinctorius* were studied in details. Cotton fabric was cationized with cationic agent (Tanafix<sup>®</sup> SR) to enhance its dyeability to the used natural dye.

Three different methods were used for extracting the various colouring matters from safflower florets, viz., acid, neutral and alkaline methods. Acid method was used to extract the yellow colour species, i.e. safflomin A, B and C, whereas the alkaline method aimed to extract the red colour component, i.e. carthamin quinone along with the neutral extracting method.

The obtained extracts were used for dyeing the cationized cotton fabric and the various parameters that may affect the dyeing process were investigated. Maximum colour strength (K/S) was found to be realized by dyeing at pH=7 and 60-70° C for 90 min. for the three used extracts. Addition of sodium chloride to the dye bath was observed to have a negative effect on the colour strength for the three different safflower extracts.

The effect of dyeing pH and temperature on the colour hue was also determined by measuring the coordinate dyeing properties (L\*, a\*, b\*) of the obtained dyeings. The colour fastness properties to washing, perspiration, rubbing and light were also assessed.

**Key words:** Cationized cotton, natural dye, safflower extracts, colouring matters, colour hue, dyeing.

## Introduction

In recent years there is a great international awareness of environmental and ecological consideration. The health hazards associated with the synthesis and application of synthetic dyes <sup>(1, 2)</sup> has led to the revival of natural dyes <sup>(3)</sup>. Natural dyes appear to be the most convenient choice as eco- friendly colourants since they are less or non-toxic, non- pollutant and less hazardous compared with synthetic dyes <sup>(4, 5)</sup>

Therefore, a lot of studies were made on extraction, characterization and application of natural dyes. Many different investigations were carried out for optimizing the dyeing conditions of natural dyes on cotton <sup>(6-10)</sup>, silk <sup>(11-14)</sup>, wool <sup>(15-19)</sup>, nylon <sup>(20, 21)</sup>, polyester <sup>(22)</sup> and acrylic fibres <sup>(23)</sup>. Most of the natural dyes have no affinity towards cotton fibres; therefore, there were some trials to increase the dye ability of cotton to natural dyes by cationization treatment <sup>(24)</sup>.

Safflower florets were the source of different colours, yellow and red shades. Yellow shades may be referred to one or more of yellow colouring matters in safflower, safflomin A (Carthamidin, C<sub>27</sub> H<sub>31</sub> O<sub>15</sub>), safflomin B (Anthocyanin, C<sub>48</sub> H<sub>57</sub> O<sub>27</sub>) and safflomin C (Precarthamin, C<sub>44</sub> H<sub>44</sub> O<sub>27</sub>). Red shade may attribute to carthamin (glucoside of 2, 3, 4, 6-tetrahydro- chalcone) which was similar to yellow precarthamin in chemical structure but with different schematic figure and absence of carboxylic group – COOH <sup>(25, 26)</sup>.

## Experimental

### Materials:

#### *Fabric*

Mill desized, scoured and bleached cotton fabric was used throughout this investigation. The fabric was kindly received from Misr/ Helwan for Spinning and weaving Co., Egypt. The weight of square meter is 165 gm.

#### *Natural Dye*

The used natural colouring matters were extracted from safflower plant using different methods.

**English name:** Safflower

**Scientific name:** *Carthamus-tinctorius* L.

**Used organ:** Dried florets

## ***Chemicals***

Sodium carbonate, sodium chloride, formic acid (85%) and ethyl alcohol of laboratory grade were used.

## ***Auxiliaries***

Cationic agent (Tanafix SR) supplied by Sybron Chemie – Nederland and non-ionic detergent (Hostapal CV-ET) supplied by Hoechst were also used throughout this investigation.

## **Methodology:**

### ***Preparation of Plant Materials***

The plant materials were dried in dry air at 35° C, for 24 hours till they become crisp. Then, they were ground in a laboratory hammer, mill to pass through a 40 mesh (British Standard Screen) and sieve to be very fine powder.

### ***Extraction of the Dyes***

#### **• *Extraction of Bright Yellow Safflower Dye (acid extraction)***

The bright yellow safflower dye was extracted by immersing the corollas [100 g] for two days in acidic cold water [one liter] at pH 6 using diluted formic acid, which made the most of colouring matters dissolve. Then, the solution was filtered. Kept with some drops of ethyl alcohol and stored under refrigerator to be used.

#### **• *Extraction of Red Safflower Dye (alkaline extraction)***

After the slightly acidic extraction of the yellow colour, the red safflower dye was extracted by washing the rest of the rest of yellow colour out until the water runs clear. The washed petals [600 g] were placed in fresh water [one liter] and sodium carbonate was added to create a basic solution, pH 11, after one hour formic acid was added to adjust the pH at 7. Immediately, the colour of the bath turned from ruddy brown to bright pink. Then, the solution was filtered, kept with some drops of ethyl alcohol and stored under refrigerator to be used.

#### **• *Extraction of Dark Yellow Safflower Dye (neutral extraction)***

The same extraction method of bright yellow safflower dye without immersing time was used with tap water instead of acidic one. The extraction was performed for one hour at neutral medium and at room temperature.

### ***Cationization of Cotton***

Cotton fabrics were treated with 20% Tanafix SR (owf) as a cationic agent, at pH 6, 70°C and for 30 minutes. This treatment was done to enhance the cotton fabrics dye ability to the used natural dyes.

### ***Dyeing Process***

The cationized cotton samples were introduced into the dyebath which contains the required amount of dye extract. The pH of the dyebath was adjusted at the suitable level and dyeing process was carried out at 100°C, for 60 min. and at liquor ratio 1:100. The dyed samples were then rinsed with cold water.

The dyed samples were mordanted using 4 g/l alum at pH 5, at room temperature, for 60 min. and at L.R. 1:50. After mordanting the samples were rinsed, soaped with 2 g/l Hostapal CV-ET at 60°C, for 20 min., then rinsed with hot and cold water and air dried.

## **Measurements:**

### ***Colour Strength***

The colour strength of the dyed samples was evaluated by light reflectance technique using **ICS- TEXICON** Computerized Spectrophotometer, model M 520220 (produced by **ICS- TEXICON** Limited Co.,) England. The colour strength expressed as K/S value and was assessed directly by the Spectrophotometer according to the Kubelka-Munk 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.

### ***Colour Coordinates***

The colour coordinates (a\* and b\*) of the dyed samples were also measured to determine the effect of some parameters on the obtained colour hue.

### ***Colour Fastness***

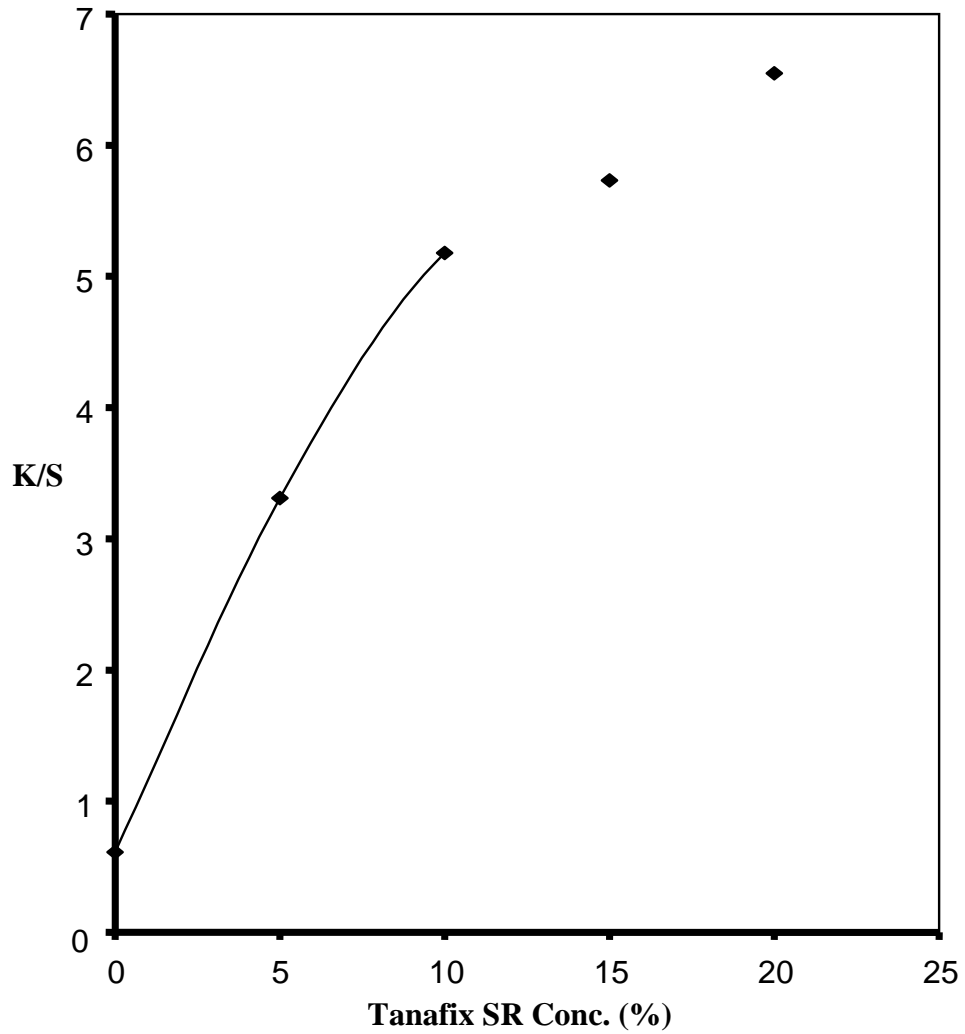
Colour fastness to washing, rubbing, perspiration were determined according to AATCC test method: (68- 1993) and fastness to light according to test method: (16A-1971).

## **Results and Discussion:**

### **(i) Cationization of cotton fabric**

Cotton fabric was found to have no or slight dye ability towards most of natural dyes on contrast of protein fibres. Therefore, cotton fabric was cationized with Tanafix SR to impart higher dye ability towards the used dye extracts. Cotton fabrics were pretreated with different concentrations of Tanafix SR ranging from 5-20% at constant conditions. The cationized cotton samples were then dyed with a fixed concentration of safflower extract and the K/S of the dyed samples were measured.

The results were shown in figure (1), from which it is observed that with increasing the concentration of cationic agent, more dye-adsorption sites are formed on the fibre surface to increase the K/S values of the dyed fabrics.



**Fig. (1): Effect of Tanafix SR Conc. on the Colour Strength of Cotton Fibres.**

Cationization Conditions:

Tanafix SR (X%), L.R. 1:50, pH (6), temp. 70° C and time 30 min.

Dyeing Conditions:

Neutral safflower extract conc.: 25 % (v/v), L.R.1:100, pH (6), 100° C and 60 min.

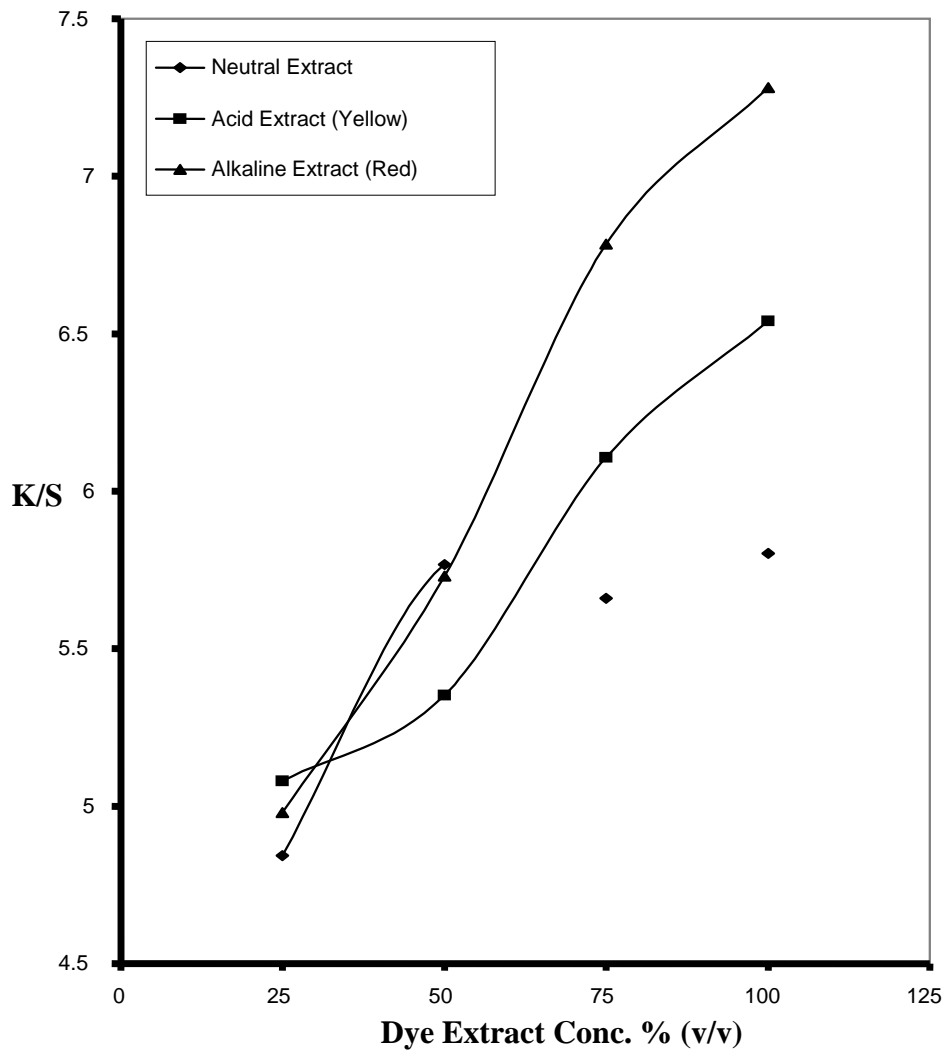
Maximum colour yield was obtained in case of cotton treated with 20% Tanafix SR. Pretreatment of cotton fabric with cationizing agent resulted in creation of cationic dye-sites capable of attracting anionic dyes via ionic attraction forces. Thus the amount of adsorbed anionic dye species i.e, natural colouring matters, in this case, will be depended on the number of cationic dye-sites (degree of cationization) on the fibre surface. By increasing the degree of cationization, the dye ability of cotton fibre was greatly increased allowing cotton to be dyed with safflower extract in a good and deep colour shade. The colour strength will be affected with the magnitude of ionic attraction between cationized cotton and anionic dye species.

Natural dyes have aromatic hydroxyl groups in their molecular structures which enable them to ionize in water giving dye anions. Thus, the number of positive charges on cotton and the degree of dye ionization will be a decisive factor in determining the magnitude of ionic attraction between dye anions and fibre cations and subsequently the obtained colour strength.

## **(ii) Optimization of dyeing conditions**

### **1- Effect of dye extract concentration:**

In order to establish a relation between safflower extract concentration and colour strength, different concentrations of safflower extract were added into the dye baths, i.e. 25, 50, 75 and 100% with respect to volume of dye bath. The cationized cotton samples were, then, dyed under fixed conditions of pH, temperature, time and liquor ratio. The colour strength of the dyed specimens was then measured and the results are plotted in figure (2).



**Fig. (2): Effect of Dye Extract Conc. on the K/S of Cationized Cotton Fabrics Dyed with Safflower Extracts. ( $\lambda=400$ )**

Dyeing Conditions:

Safflower extracts: (x%), L.R. 1:100, pH (7), temp. 100° C and time 60 min.



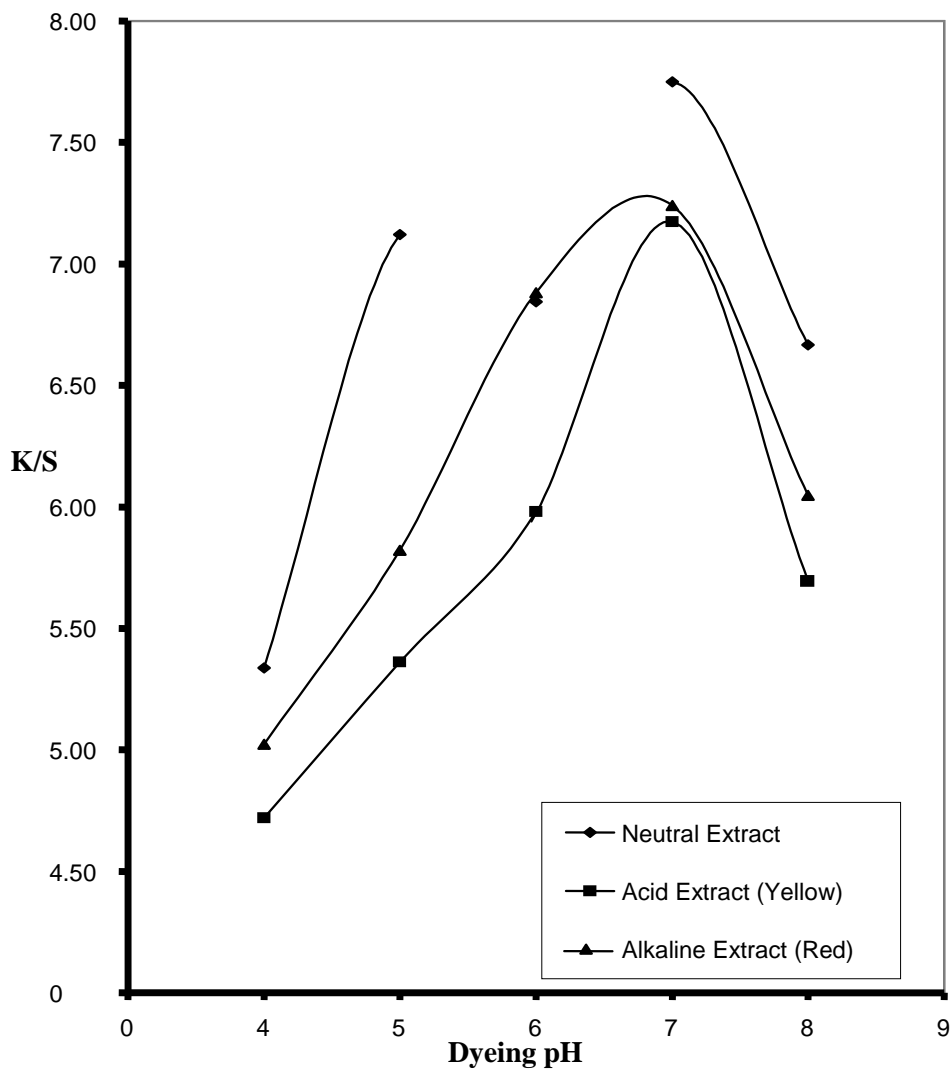
A great similarity in dyeing behavior of acid and alkaline safflower extracts was clearly noticed from figure (2). The colour strength of the two extracts was gradually increased with increasing the extract concentration. Neutral safflower extract was found to have a different behavior since maximum colour strength was achieved at 50% extract concentration beyond which no further increase in K/S was observed. The method of dye extraction determines greatly the nature of extract composition. The main colouring matters in acid extract is yellow safflower dye which readily soluble in water. Alkaline extract of safflower contains mainly the red carthamin dye which is less soluble in water and has a relatively high substantivity to cotton fibre.

For neutral extract, traces of red colour may be obtained with the yellow colouring matters which may affect their nature in aqueous solution. The behavior of these different extracts towards the cationized cotton fabrics will be affected with their relative affinities, solubilities, degree of dye aggregation in aqueous solutions as well as the ionic attraction magnitude between fibre cations and dye anions. In case of neutral extract, it may be supposed that the dyes admixture may enhance their aggregation in water especially at higher extract concentration, i.e. over 50%.

## **2- Effect of dyeing pH:**

The physico-chemicals properties of the dyeing process such as the dyeing equilibrium, degree of exhaustion and rate of dyeing are pH-dependent especially in case of ionic dyeing systems. In order to optimize the pH of the dye bath, the cationized cotton fabrics were dyed using a constant concentration of safflower extract under fixed dyeing conditions except the pH value, which were; 4, 5, 6, 7 and 8. The colour strengths of the dyed samples were measured and the data are plotted in figure (3). From the figure, one can notice the similarity in behavior, during the dyeing processes, of the three applied safflower extracts.

Maximum dye absorption was observed at neutral medium, i.e. pH=7, for the three extracts. The colour strength was gradually increased with increasing the dye bath pH value from 4 up to 7 to reach maximum percent increase in K/S of about 51.9, 45.1 and 44.2% for acid, neutral and alkaline extracts respectively, then started to decrease beyond pH=7.



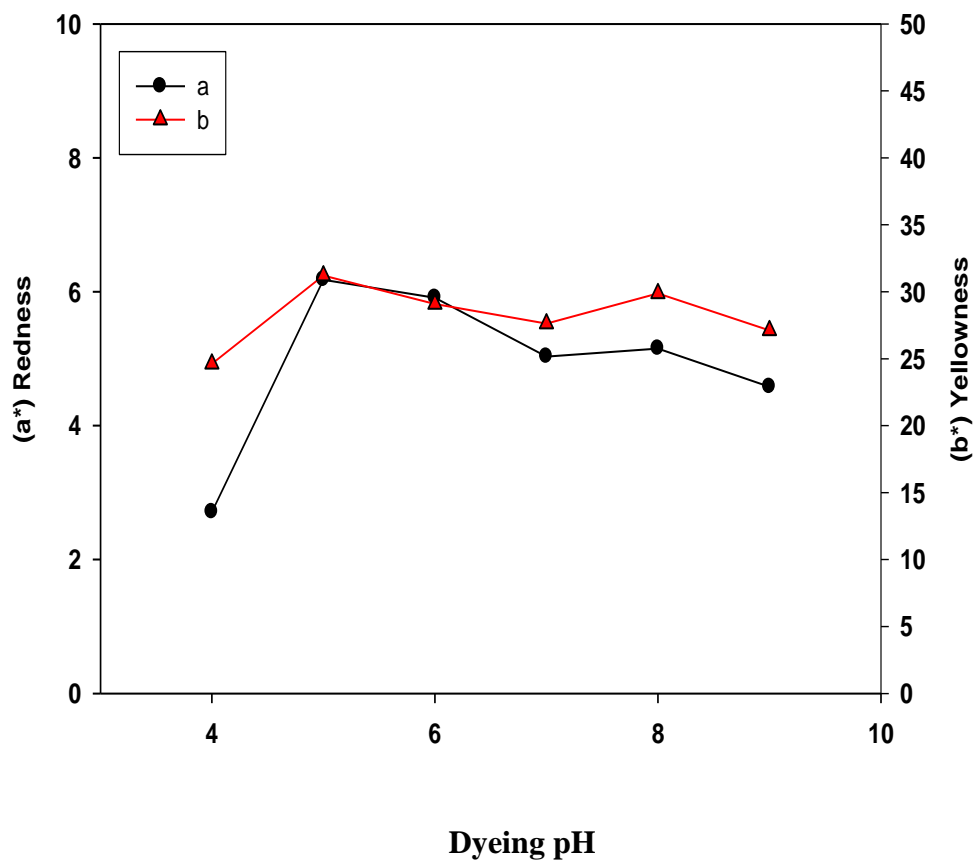
**Fig. (3): Effect of Dyeing pH on the Dye Uptake on Cationized Cotton Fabric Dyed with Safflower Extracts. ( $\lambda=400$ )**

Dyeing Conditions:

Safflower extracts conc.: 75 % (v/v), L.R.1:100 temp. 100°C and time 60 min.

Exhaustion and fixation of natural dyes on the cationized cotton fabrics depend essentially on the ionic attraction between dye anions and fibre cations. Therefore, the colour strength will be affected with the magnitude of ionic attraction which may be influenced with the pH of the medium. The cationic nature of the used cationizing agent (Tanafix SR) will acquire cotton fabrics positive charges capable of attracting anionic colour species. The relative stability of the cationic agent on cotton may be lowered under alkaline medium with decreasing its cationic nature. On the other hand, natural dyes depend on their solubility in aqueous solutions on the phenolic hydroxyl groups present in their molecular structure. The ionization of hydroxyl groups are greatly enhanced at alkaline medium, which may explain the increased colour strength of the different safflower extracts on cationized cotton by elevating the pH of the dye bath from 4 to 7.

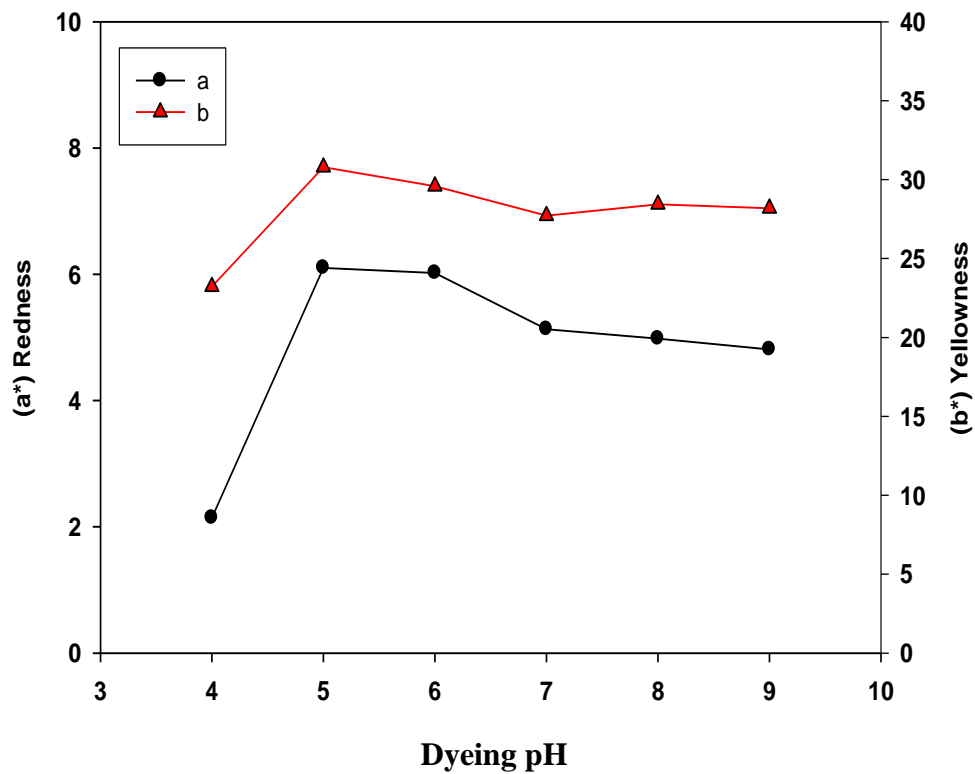
Increasing dye solubility means higher ionic attraction between dye anions and the positively charged cotton fibres leading to higher colour strength. The dyed samples were subjected to measuring their colour coordinates;  $a^*$  and  $b^*$  and figures (4-6) illustrate the influence dye bath pH on redness ( $a^*$ ) and yellowness ( $b^*$ ) of cationized cotton fabrics dyed with acid, neutral and alkaline safflower extracts respectively. Increasing the pH of the dye bath from 4 to 7 is accompanied with an increase in redness of samples. Maximum redness is achieved at pH 5 using acid extract while high readings of ( $a^*$ ) were obtained at pH 5-6 and 5-7 in case of neutral and alkaline safflower extracts respectively. It may be concluded, from the previous discussion, that pH of dyeing is a decisive factor in optimization of the dyeing results either for colour depth or for color hue.



**Fig. (4): Effect of Dyeing pH on the Colour Hue of Cationized Cotton Fabrics Dyed with Acid Safflower Extract. ( $\lambda=400$ )**

*Dyeing Conditions:*

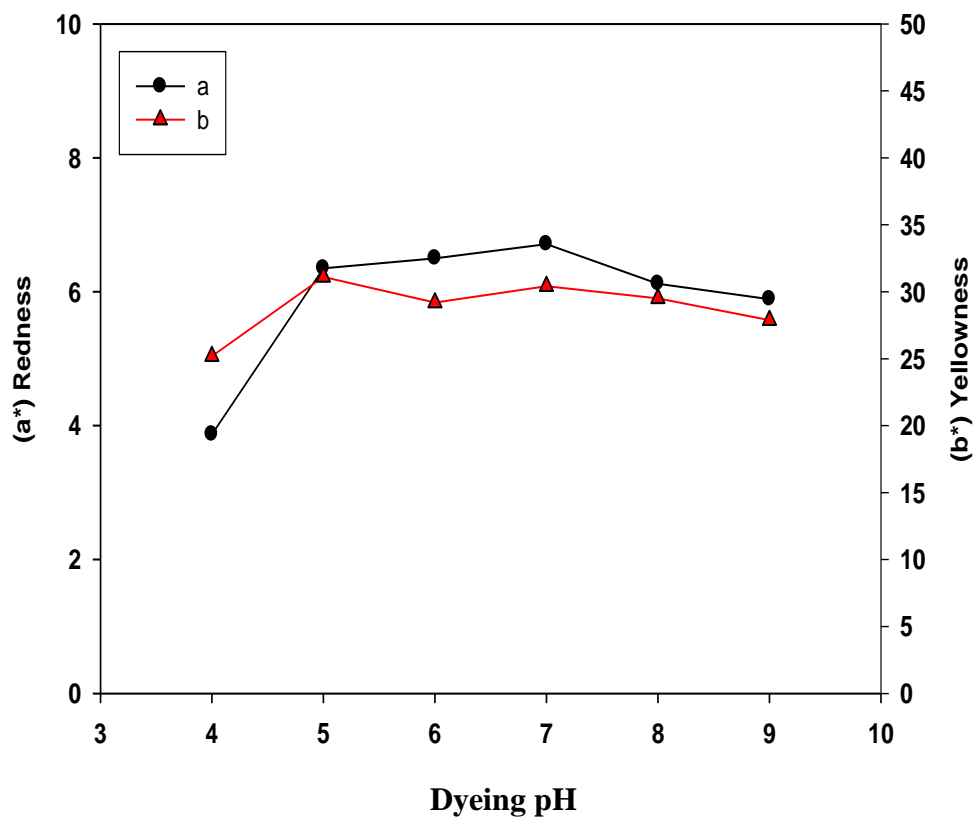
Acid Safflower extract conc.: 75 % (v/v), L.R. 1:100 temp. 100°C and time 60 min.



**Fig. (5): Effect of Dyeing pH on the Colour Hue of Cationized Cotton Fabrics Dyed with Neutral Safflower Extract. ( $\lambda=400$ )**

Dyeing Conditions:

Neutral Safflower extract conc.:75 % (v/v), L.R.1:100 temp. 100 °C and time 60 min.



**Fig. (6): Effect of Dyeing pH on the Colour Hue of Cationized Cotton Fabrics Dyed with Alkaline Safflower Extract. ( $\lambda=400$ )**

Dyeing Conditions:

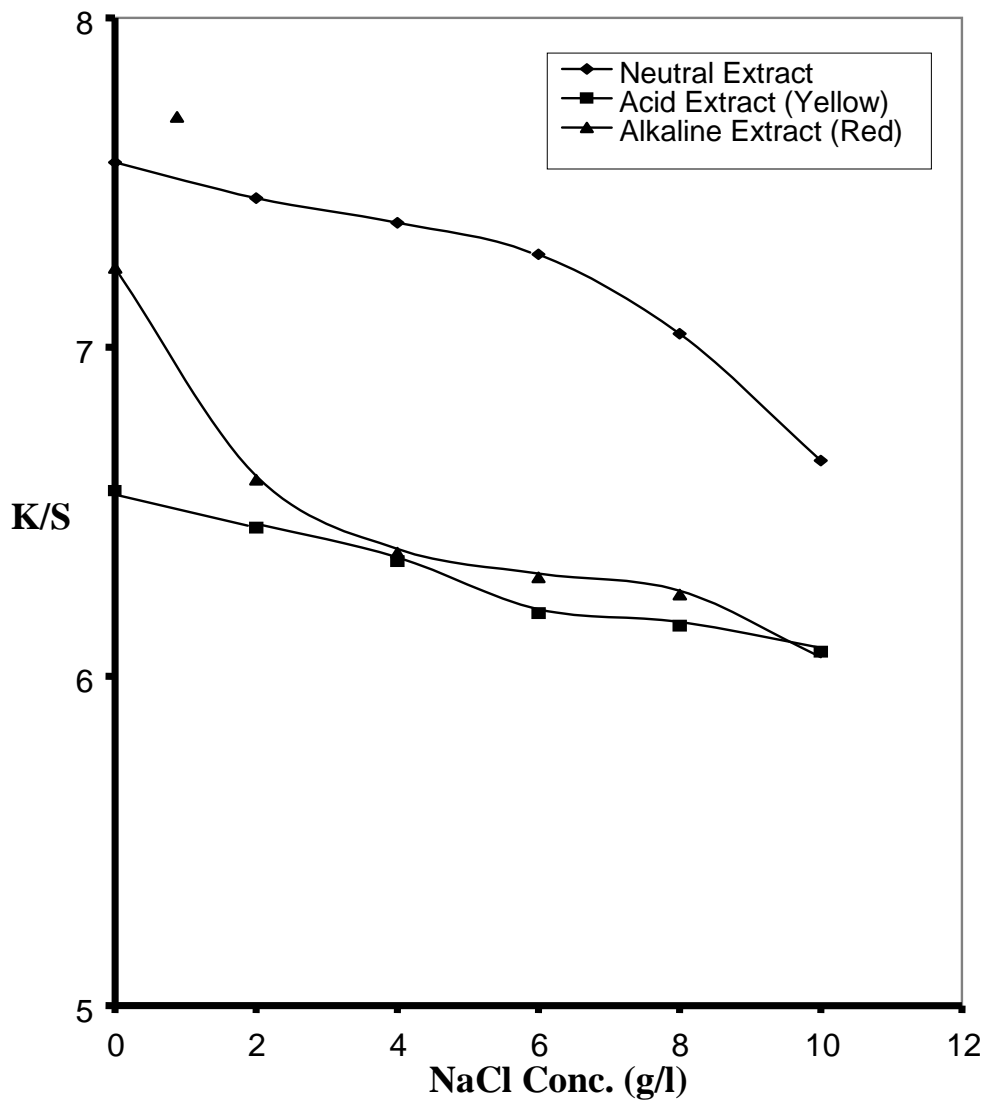
Alkaline Safflower extract conc.: 75 % (v/v), L.R.1:100, temp.100°C and time 60 min.

### **3- Effect of electrolytes:**

Neutral electrolytes play a great role in the dyeing of textile fibres. Their action depends essentially upon the type of both substrate and dyes. Electrolytes may be considered as exhausting agent in case of dyeing cellulosic fibres with anionic dyes, i.e. direct and reactive.

It is well known that cellulosic fibre acquire negative charges on its surface on immersing in aqueous solution, therefore, an electrical repulsion will be formed between the fibre and any anionic species present in the system. For controlling the ionic system and minimizing this repulsion, sodium chloride may be added at a suitable concentration depending upon the magnitude of the repulsion. The action of electrolyte in case of dyeing cationized cotton fibre with safflower extracts will be different owing to alteration of zeta potential of cellulose and for this reason its action on dyeing results was studied.

Cationized cotton samples were dyed with the three- safflower extracts at pH 7 using different sodium chloride concentrations, i.e. zero, 2, 4, 6, 8 and 10 g/l. The K/S of the samples are plotted in figure (7) from which, one can notice the decrease of colour strength by addition of sodium chloride, aggregation is increased by addition of electrolytes such as sodium chloride which may cause a reduction of dye solubility and if sufficient amount of sodium chloride is added, the dye will be precipitated. Addition of 2 g/l sodium chloride resulted in reduction in K/S reached about 1.53, 1.45 and 8.73% for acid, neutral and alkaline safflower extracts respectively with respect to samples dyed without sodium chloride.



**Fig. (7): Effect of Salt Concentration on the K/S of Cationized Cotton Fabric Dyed with Safflower Extracts. ( $\lambda=400$ )**

Dyeing Conditions:

Safflower extracts conc.: 75% (v/v), L.R.1:100, pH7, temp.100°C and time 60min.



Red safflower extract was found to be the most sensitive colour species to addition of electrolytes owing to its relatively low solubility. The percent decrease in K/S is illustrated in table (1). The difference in the chemical structure of the colouring matters contained in safflower extracts and the number of the hydroxyl groups present in their molecular structures may affect greatly the sensitivity of these dyes to addition of electrolytes. On the other hand, aggregation of natural dyes in aqueous solution will be accelerated by addition of electrolytes owing to the increased physical forces attraction between the dye molecules.

Table (1): Action of addition of NaCl on K/S of cationized cotton fabrics dyed with safflower extracts.

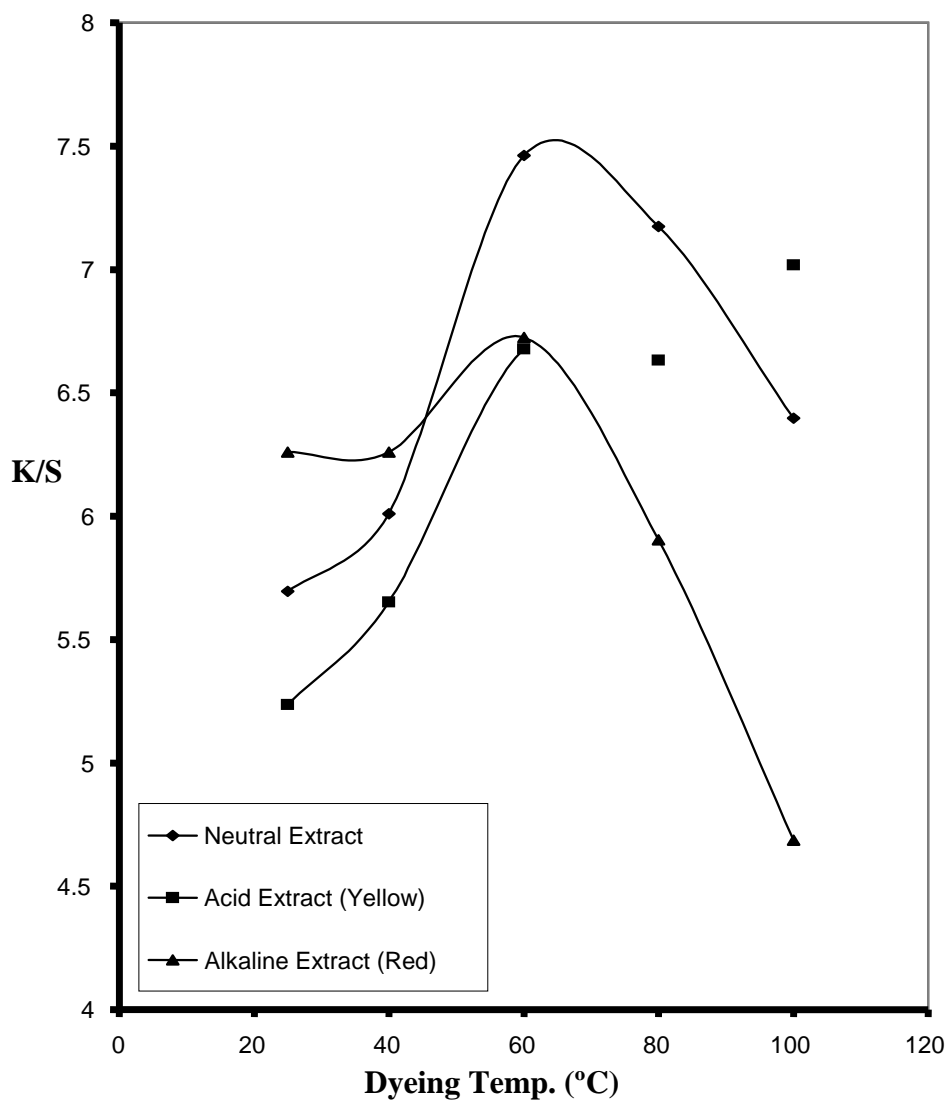
Na CL Conc. (g/l)	Acid Extract		Alkaline Extract		Neutral Extract	
	K/S	(%) Decrease	K/S	(%) Decrease	K/S	(%) Decrease
0	6.56	-	7.24	-	7.57	-
2	6.46	1.53%	6.61	8.73%	7.46	1.45%
4	6.36	3.05%	6.39	11.74%	7.38	2.51%
6	6.20	5.49%	6.31	12.85%	7.29	3.70%
8	6.17	5.95%	6.26	13.54%	7.04	7.00%
10	6.09	7.16%	6.06	16.30%	6.66	12.02%

#### 4- Effect of dyeing temperature:

Temperature of dyeing, in fact, has its vital role in all dyeing processes. By raising dyeing temperature, many different aspects may be changed, some of them are related to the substrate and some others are reflected on the form of the dye molecules in the aqueous solution. On the other hand, the dyeing equilibrium may also be altered under the action of temperature.

The action of dyeing temperature on the absorption of safflower extracts on cationized cotton fabric was investigated and the results are formulated in figure (8).

A general behavior was noticed for the three safflower extracts under the action of dyeing temperature since the colour strength increases with raising the temperature and reaching its maximum at 60°C, beyond which an appreciable decrease was carried out.



**Fig. (8): Effect of Dyeing Temp. on the K/S of Cationized Cotton Fabric Dyed with Safflower Extracts. ( $\lambda=400$ )**

Dyeing Conditions:

Safflower extracts conc.: 75 % (v/v), L.R.1:100, pH (7), and time 60min.

Red safflower colouring matter was found to be the most sensitive one to dyeing temperature since it gave the highest K/S at 60°C, and the lowest K/S at 100°C, compared with the other two dye extracts. Raising temperature may function as a disaggregating factor for natural dyes, enhancing their solubilities and accelerating their rates of diffusion and adsorption on the fibre surfaces.

Therefore, the overall result is increasing the colour yield and hence the final K/S for the three safflower extracts. The effect of temperature is controlled within a definite range between 25°C and 60°C. Above 60°C, the situation was varied since the stability of the cationizing agent on cotton fabric will be negatively influenced at high temperature.

It must be mentioned that the most convenient temperature for cationization process is about 60-70°C, for fixing maximum amount of cationic agent on cotton. Therefore, it may be suggested that under higher temperature some of the cationic agent may desorb into the bath leading to less dyeable cotton fabric.

On the other hand, the attraction forces between natural dyes and cotton will be decreased by elevating temperature which may be attributed to the low natural substantivity of these dyes to the fibre.

The influence of dyeing temperature on colour hue of cationized cotton dyed with safflower extracts was also studied and the results are illustrated in figures (9-11). In figure (9), in case of acid safflower extract, one can notice that bright yellow colour could be realized at lower temperature <60°C, whereas at higher temperature, the received yellow was dull and reddish.

It was noticed; from figure (10), that by raising the dyeing temperature from room temperature up to 100°C, in case of neutral safflower extract, the colour hue was altered owing to the slight increase of redness ( $a^*$ ) and reduction of yellowness ( $b^*$ ). It may be concluded that the colour hue of the dyed samples tends to be more reddish by raising dyeing temperature. As for alkaline extract, figure (11), maximum redness was noticed to achieve at 40-60°C.

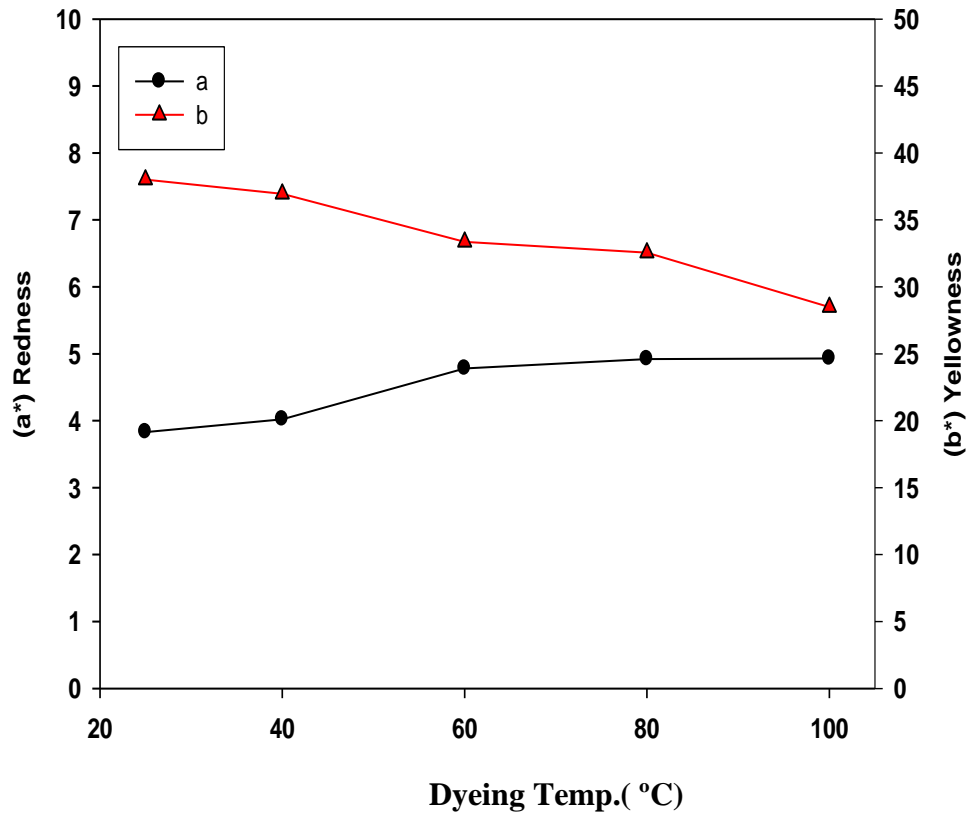
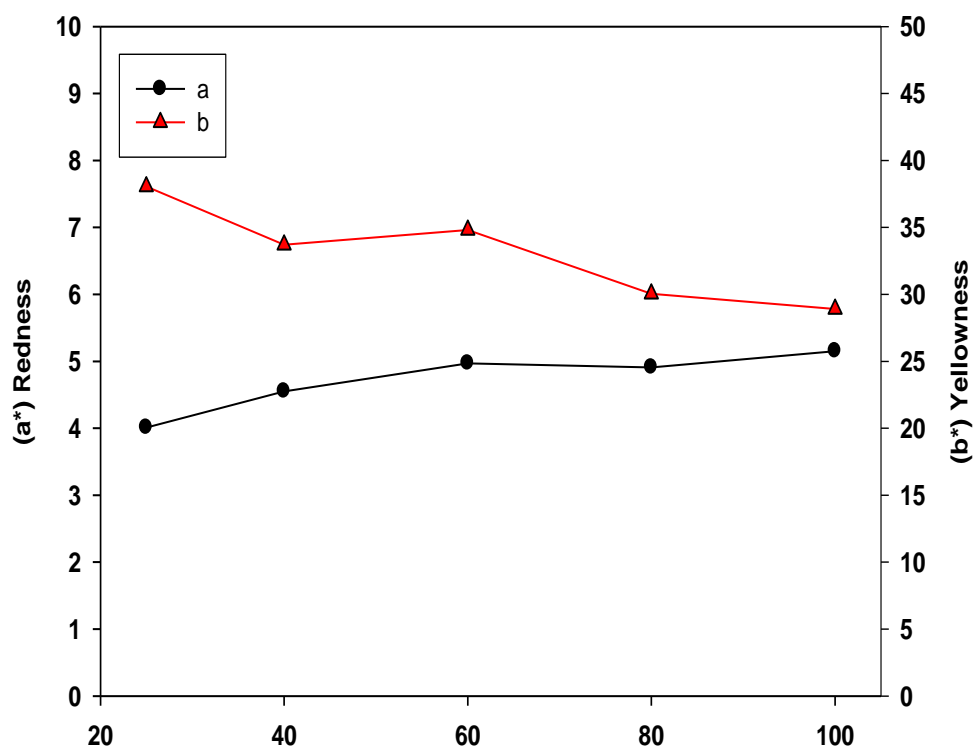


Fig. (9): Effect of Dyeing Temp. on the Colour Hue of Cationized Cotton Fabrics Dyed with Acid Safflower Extract. ( $\lambda=400$ )

Dyeing Conditions:

Acid safflower extract conc.: 100 % (v/v), L.R.1:50, pH (7), and time 60min.

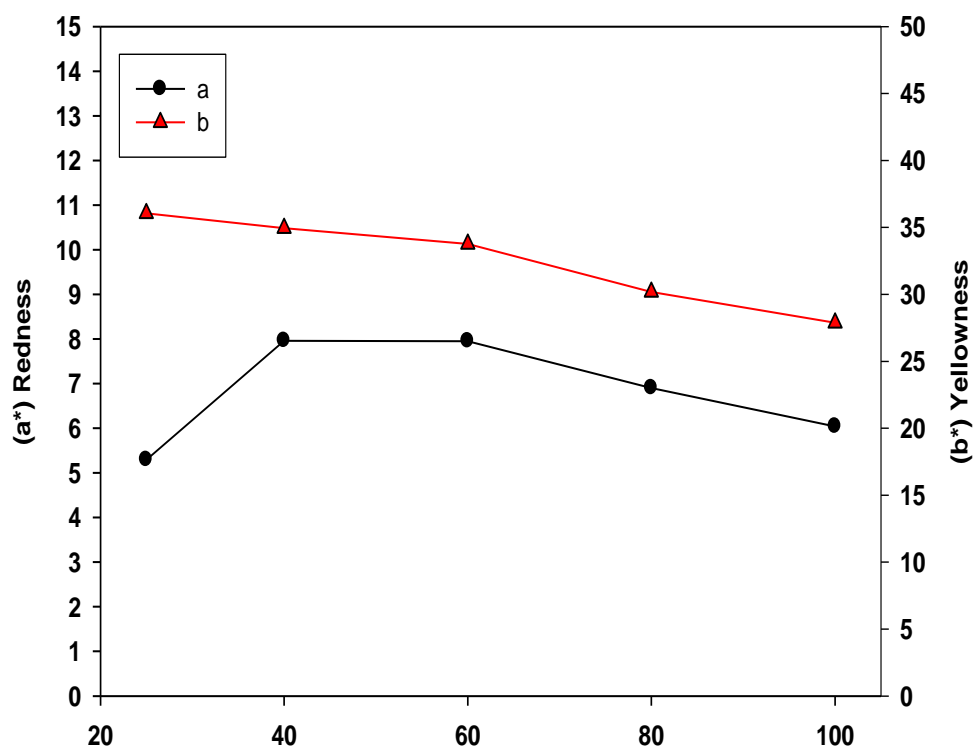


Dyeing Temp. (°C.)

**Fig. (10): Effect of Dyeing Temp. on the Colour Hue of Cationized Cotton Fabrics Dyed with Neutral Safflower Extract. ( $\lambda=400$ )**

Dyeing Conditions:

Neutral safflower extract conc.: 75 % (v/v), L.R.1:100, pH (7), and time 60min.



Dyeing Temp.(°C.)

**Fig. (11): Effect of Dyeing Temp. on the Colour Hue of Cationized Cotton Fabrics Dyed with Alkaline Safflower Extract. ( $\lambda=400$ )**

Dyeing Conditions:

Alkaline safflower extract conc.: 75 % (v/v), L.R.1:100, pH (7), and time 60 min.

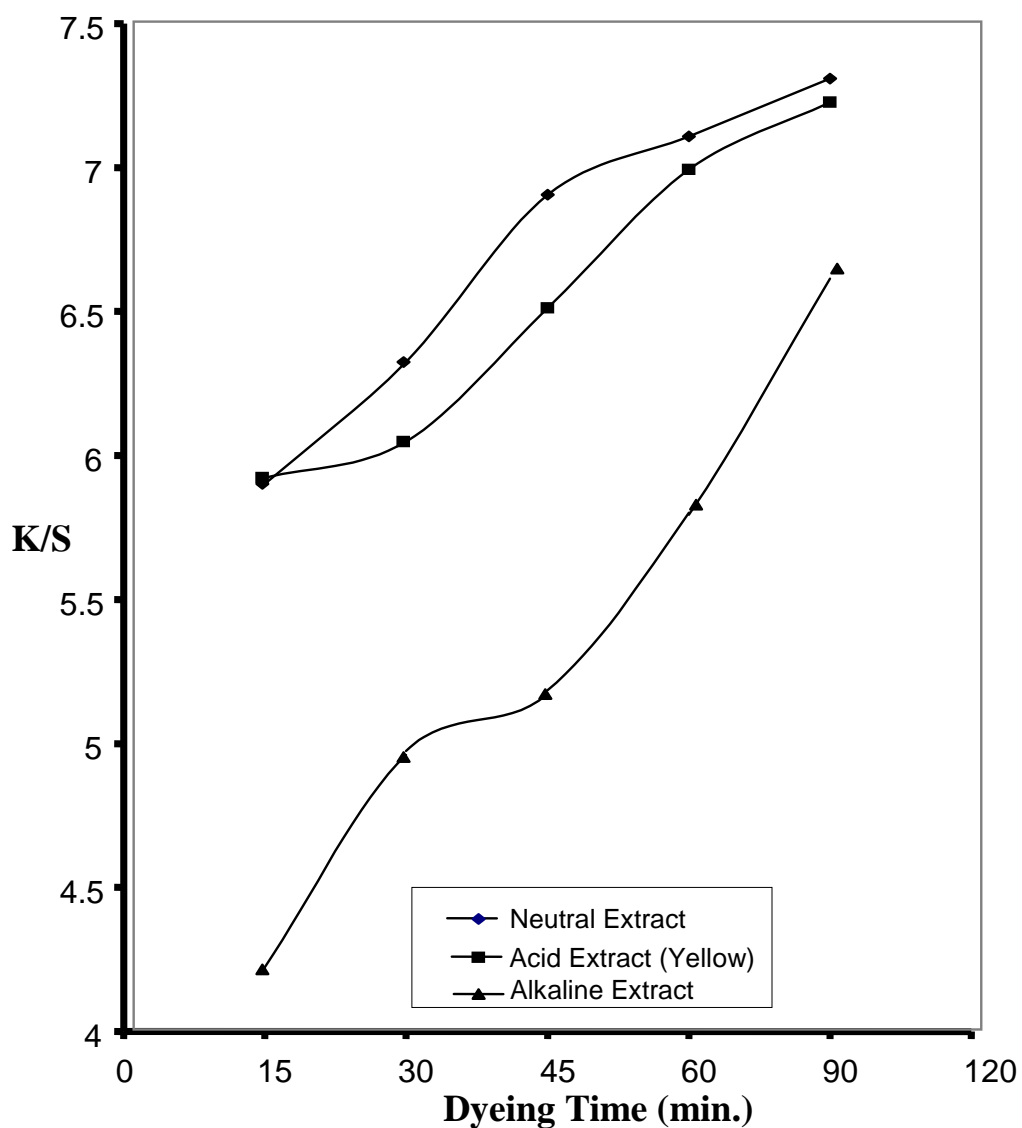
## **5- Effect of dyeing time:**

Time of dyeing was also studied for determining the most suitable duration required for realizing maximum dye absorption and highest colour strength. Different cationized cotton samples were subjected to dyeing under constant conditions of extract concentration; dye bath pH, temperature and liquor ratio, and the dyeing processes were carried out for different durations (15-120 minutes).

Figure (12) illustrates the action of dyeing time on the received colour strength for the used dye extracts. By lasting the time of dyeing, the K/Ss for the three extracts are gradually increased to reach their maximum values after 90 minutes.

The dyeing time is related to the rate of dyeing which greatly influenced by many various factors such as:

- 1- The molecular size of the colouring species.
- 2- The rate of diffusion either in aqueous solution or within the fibre itself.
- 3- Degree of dye aggregation in aqueous solution.
- 4- The substantivity of the colour species.
- 5- Dyeing temperature.
- 6- The pH of the dye bath.



**Fig. (12): Effect of Dyeing Time on the K/S of Cationized Cotton Fabric Dyed with Safflower Extracts. ( $\lambda=400$ )**

Dyeing Conditions:

Safflower extracts conc.: 75 % (v/v), L.R.1:100, pH (7) and temp. 100°C.



### (iii) Colour fastness properties

The colour fastness properties of cotton fabrics dyed with neutral and alkaline safflower extracts were evaluated in terms of fastness to washing, rubbing, perspiration and light according to the AATCC recommendations as shown in table (2). The obtained dyeing results are found to realize an overall fastness properties ranging from good (3) to very good (4-5). The colour fastness properties of alkaline safflower extract is observed to be relatively lower than that of neutral extract due to the higher sensitivity of the red colour especially to alkaline treatment (perspiration).

Table (2): Colour fastness properties of cotton fabrics dyed with Neutral and alkaline safflower extracts.

Cotton dyed with safflower	Washing			Rubbing		Perspiration					Light		
	Alt.	stain		Dry	Wet	Acid			Alkali				
		*	**			Alt.	stain		Alt.	stain			
							*	**		*		**	
Neutral extract	3-4	4-5	3-4	4-5	2-3	4	4-5	4	4	4	4	4	5
Alkaline extract	3	4-5	4	4-5	4-5	3-4	4-5	4	2-3	4	3-4	3	3

**N.B.:** (\*) refers to staining on cotton fibres and (\*\*) refers to staining on wool fibres.

### Conclusion:

This research work is aiming to separate the different colouring matters existing in safflower plant by using three different extracting methods. Also this paper is aiming to make use of the cationization treatment of cotton to enhance its dye ability towards natural dyes. The three extracts dyed cationized cotton successfully and to reach their maximum colour strength values, there was a detailed study to all the parameters affecting the dyeing process.

Addition of electrolytes in the dyeing bath tend to have no significant role, on the contrary it has a negative effect. Also we took into consideration the role of the pH value of the dye bath and the dyeing temperature on the colour hue of the samples dyed with the three safflower extracts.

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