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Application of Natural Amino Acid Extracted from Soybean Seed Waste on Egyptian Cotton

Shereen O. Bahlool and Salah M. Saleh*
Department of Cotton and Textile Fibers Chemistry, Cotton Research Institute, Agricultural Research Centre, Giza, Egypt.

THE aim of this paper was to research how coating of Egyptian cotton fabric with renewable source of natural amino acids extracted from soybean seed waste would influence the textile to be coated and how it affects the penetration of reactive dyestuff. Cationization have been studied on scoured and mercerized cotton fabrics for achieving electropositive charge and better adsorption and absorption properties and the optimum conditions were determined. The changes in surface morphology of fabrics after cationization were identified by FTIR spectroscopy analysis. The results revealed obtained that the color measurements, tensile strength, elongation and fastness properties of cationized cotton fabrics has improved compared with the untreated fabrics. An important advantage of cationic treatment is in the economical and environmental impact by using the waste of soybean and minimize the environmental problems occurred in reactive dyeing by making salt-free reactive dyeing.

Keywords: Soybean, Egyptian cotton, Cationization, Amino acid extraction, Reactive dye.

Introduction

Egyptian cotton is known for its high quality, and thus the fabrics made from it have distinct properties. Egyptian-grown cotton is known for its ability to create extra-long staples, or fibers. These fibers can produce thread or yarns that are thinner than other cottons because of their longer length. This yarn is smaller in diameter yet stronger than other cottons. Fabrics made of Egyptian cotton are softer, finer and last longer than any other cotton so are worth putting the slight extra bit of money into. Since finer yarns mean a higher thread count, the weave of the fabric is significantly stronger and lasts a lot longer than regular cotton [1]. Whereas, cotton as one of the natural cellulosic fiber which builds up negative charges on its surface when immersed in water, lead to an inverse effect on exhaustion of anionic dyes [2]. The slightly negative charge on the fibers repels anionic dyestuff and hence the efficiency of dye exhaustion and fixation on cellulosic fiber is generally low.

Reactive dyes considered as the most popular dyes for dyeing cotton fabrics because of their color shades and good color fastness properties due to it chemically bonding between the reactive dye and the cellulosic fibers which is being dyed [3-5]. But this application requires a very high concentration of electrolytes (such as: NaCl or Na_2SO_4), because of the fact that both cotton fibers and reactive dyes are anionic, that cotton fabrics acquires negative charge in aqueous medium and thus repels negatively charged dye anion during dyeing and the salt suppresses negative charge build-up at the fiber surface and promotes increased dye uptake, even with required using large quantity of salt in dye bath which can vary up to 100g/l depending on the depth of color required, the structure of the dyes and the dyeing recipe, even so, only 65–70% of reactive dyes are exhausted, remaining 25–30% of dyes are removed as a colored effluent after dyeing [6,7]. Many attempts have aimed to improve dye bonding and dye adsorption on materials through chemical modification [8].

*Corresponding author e-mail: salah.mansour@arc.sci.eg
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Cationization is one of the most important modifications for cellulose. Cationic modification is the method that has been employed in order to change the surface charge of cellulosic fibers and improve the cationic activities of cellulose fibers [9]. Cationization may enhance dye absorption and can not only increase the dyeing color strength but also improve wash fastness and to reduce the pollution [10]. Many researches worked to using variety of cationic compounds in order to improve different properties of cotton fabrics [11]. Cationic sites can be introduced either by aminization or cationization. Our previous work deals with dyeing nylon/cotton blends to a solid shade in one bath using one dye. The fabric treated with chloroacetic acid (CAA), to form the anionic form followed by the cationization form using the inorganic salt of magnesium chloride [12]. According to our previous work, cationization of cotton fabric for achieving electropositive charge and better adsorption properties was carried out with cationic compound produced from extractable solution of chicken feather and dyeing of modified cotton with alkaline extraction of acacia bark [13]. Utilization of feather waste to improve the properties of some Egyptian cotton fabrics were studied [14]. Initially waste feather protein was used to prepare a reactive cationic crosslinking modified agent, WLS, as a starting material which was used to prepare a quaternary ammonium type cationic environment-friendly fixing agent (named WLSPR) for dyeing cotton fabric with reactive dyes after solid color processing [15]. Antibacterial activity of cationically modified cotton fabric with a water-soluble carboxymethyl chitosan was studied by Amira et al. [16]. Modification of cotton fabric was done during cationization process by the means of cationizing agent, namely Quat-188 for improving the physical properties in addition to impart antimicrobial properties to cotton fabric and modified cotton fabric by using herbal in the presence of softeners, [17]. An excellent mordant dyeing can be achieved. by a novel approach for enhancement of mordanting dyeing using natural dyes using UV/O_3 irradiated-chitosan treated fabrics, [18].

Soybean hull was found to be effective for cationization in salt-free dyeing of cotton which presented the possibility of salt-free reactive dyeing of cotton by modifying the surface of cotton fabric using soya bean. The cationization of cotton was carried out with natural amino acid extract obtained by acid hydrolysis from soybean and cationized in the pad-dry-curing process to investigate its dye-ability with reactive dye in both a conventional alkaline dye bath and salt-free acidic dye bath [6]. Protein was extracted from soybean flakes and meals by ionic-strength of lime as alkali treatment [19]. Protein extraction and purification by lime treatment and ultrafiltration on soybean flakes and meals is an environmentally friendly process that promises a novel alternative to conventional chemical treatment methods. Enhancing the extraction of protein from soybean and other health promoting factors were identified and evaluated for implementation, including pilot-scale studies [20]. Protein extracts from un-germinated and/or germinated local Egyptian soybean and lupin flours were hydrolyzed using the enzyme papain, [21]. It was observed that amino acid content in soya bean waste contains a total of 17 types of amino acids, out of which glutamic acid and arginine contents are the highest [22].

This study is an attempt to modify cotton fabrics by using natural amino acid extracted from soybean seed waste. In addition, salt-free reactive dyeing of cationized cotton fabrics was carried out with the consideration of the environment. The present work aims to eliminate salt usage in the reactive dyeing of cellulosic fabric by modifying the cotton fiber to increase dye-fiber interaction is therefore the best way to overcome the environmental problems occurred in dyeing cotton with reactive dyes making salt-free reactive dyeing.

Materials and Methods

Materials

Egyptian cotton fabric Giza 90 was purchased from Misr- Mehalla Spinning and Weaving Company, Egypt. The cotton samples had the following specifications: Plain wave 1/1, weight of square meter 130 gm, number of threads per cm of warp 38, number of threads per cm of weft 30, and count number of yarn 40.

Chemicals

All chemicals used were of analytical grade using doubly distilled water (18.5 MΏ.cm-1). Hydrochloric acid (HCl), sodium hydroxide (NaOH), sodium chloride (NaCl), sodium carbonate (Na_2CO_3), acetic acid (CH_3COOH), triton x-100, Magnesium Chloride ($MgCl_2$) and detergent. Soybean seed waste as a source of cationizing agent was obtained from oil and fat unit, food technology research institute, agricultural research center, Egypt.

Dyestuffs

Reactive Orange ME-2RN was used for dyeing process. Chemical structure of the reactive dye was represented in Figure (1).

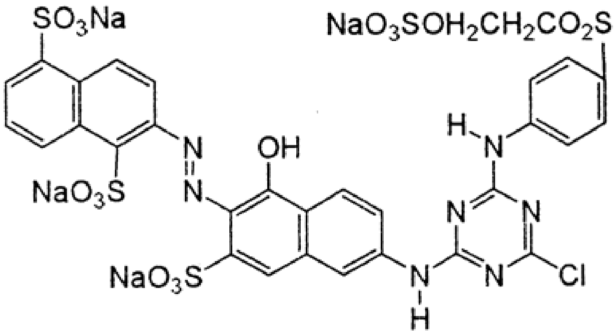

Fig.1. Reactive Orange ME-2RN, C.I: Reactive Orange 122.

Methods

Scouring

In order to improve the adhesion of the dye to cotton fibers, an alkaline pre-treatment in water solution containing sodium hydroxide 4 gm/l. NaOH (liquor ratio, 30:1), Wetting agent (triton X-100) using liquor ratio 1:50 at boiling for 90 minutes. Then rinsing with hot and cold water then air dried at room temperature

Mercerizing

The scoured fibers were immersed in 20% aqueous of sodium hydroxide for two minutes at room temperature. Treatment was carried out in slack state of fabrics, then fabrics washed with tap water, and then neutralized with aqueous solution containing 0.1% acetic acid followed by washing with hot water to ensure removal of residual chemicals. Finally, Samples were air dried

Amino Acids extraction

Extraction of amino acids from soybean seed waste was carried out using the methodology by Dessiea and Govindanb [6], with some adaptation. Soybean seed waste has been treated with continuous stirring with 5M HCl for 24 hours at room temperature (27 ± 3˚C) at 1:10 material to liquor ratio. The solution is then filtered and the pH adjusted 5.5 to 6.0 with (NaOH 10%) solution using digital pH meter. The filtrate was then taken as extracted amino acid. Total amino acid analysis is used to determine the amino acid content of amino acid-peptide- and protein-containing samples. The first step of amino acid analysis involves hydrolyzing these peptide bonds. The liberated amino acids are then separated, detected, and quantified. The method was carried out using amino acid analyzer Model Biochrom 31+ Protein Hydrolysate System by HCl acid hydrolysis technique. The results obtained showed that the total amino acids were 19% by weight.

Optimization of cationization parameters

The application of amino acid solution extracted from soybean seed waste on scoured and mercerized cotton fabric was done using three cationization baths by padding- dry- cure technique with 100% wet pickup as follows:

Cotton fabric samples were padded with aqueous extraction of soybean seed waste in presence of specified concentration of $MgCl_2$ (1/5th the content of amino acid) according to Nallathambi et al. [23].

Cotton fabric samples were padded with known concentration (0.2M) of $MgCl_2$ solution. All treated cotton fabrics were dried at a temperature of 105-110°C in hot air oven and further cured at 150°C for 3 minutes.

Dyeing

Reactive dye bath in which the treated and untreated cotton fabrics were dyed with 3% (Reactive Orange) at liquor ratio 1:20, following the standard procedure as shown in Figure (2). The dyed fabrics were then rinsed with hot water followed by rinsing with cold water and finally air dried.

Evaluation tests

All the fabric samples were conditioned for 48hrs at 65% (±5%) relative humidity and 27°C (±20C) according to IS:6359-1971 standard method .

Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) of Cotton fabric crushed samples were examined using FTIR 6300 instrument from Jasco Inc., Japan, equipped with a Specac Golden Gate ATR which is fitted with a diamond crystal and an interaction angle of 45°. All the spectra recorded in the range 4000–400 cm^{-1} were averaged over 32 scans at a resolution of 4 cm^{-1}. All the recorded data was processed with Spectra Manager II software from Jasco Inc., Japan.

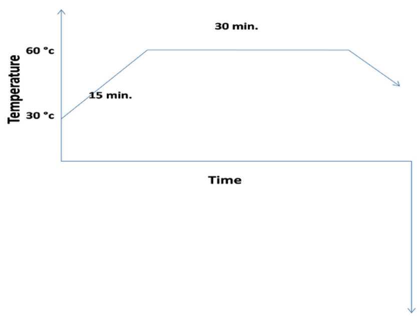

Fig. 2. Reactive Dyeing curve.

Measurement of color strength (K/S)

The color strength (K/S) of all specimens was recorded on the surface of cotton samples before and after treatment with a Spectrophotometer Perkin Elmer Model Lambda 35 with integrated sphere. Measurements were made using a D65 illuminant and 10-degree standard observer. Percentage of reflectance, collected at 10nm intervals over the visible spectrum (from 400 to 700 nm) was converted into the CIELAB color system. K/S was calculated according to the Kabulka-Munk equation:

$$\frac{K}{S} = \frac{(1-R)^2}{2R}$$

Where:
R: Decimal fraction of the reflectance of dyed samples.
K: Absorption coefficient.
S: scattering coefficient

Measurement of tensile strength (Kg/f) and elongation (%)

The tensile strength (N) and elongation (%) were measured using Shimadzu. The testing was carried out according to ASTM D-1682-1924

Fastness properties

Color Fastens to Washing: ISO 105 C10:2015 “Textiles - Tests for Color Fastness - Part C10: Color Fastness to washing with Soap or Soap and Soda”

Light fastens: ISO 105 B02:2014 “Textiles - Tests for Color Fastness - Part B02: Color fastness to artificial light: Xenon Arc Fading Lamp Test”

ISO 105-X12:2016 Textiles – Tests for color fastness- part x12: color fastness to Rubbing

Color fastness to perspiration: ISO 105 E04:2013 Textiles - “Tests for Color Fastness - Part E04: Color fastness to perspiration”

Results and Discussions

Confirmation of cationization: Infrared analysis

Cationization was confirmed using FTIR by checking presence of additional functional groups. It has been noted that Carbon, Nitrogen and Oxygen are major elements in soybean seed waste. FTIR spectroscopic analysis is used to determine characteristic peaks in functional group (4,000^{-1} - 400 cm^{-1}) and fingerprint (400 –1,400 cm^{-1}) region [24]. From Figure (3-1), the broader absorption band between 3600 and 3100 cm^{-1} is attributed to OH stretching vibration forming hydrogen bonds in the cellulose molecule. This common absorption band is composed of two small vibrations located at 3296 cm^{-1} (attributed to intermolecular hydrogen bonds) and 3341 cm^{-1} (attributed to intra-molecular hydrogen bonds). The peaks observed at 2895 – 2908 cm^{-1}, which is attributed to $–CH_2$ asymmetric vibrations. Other common absorption bands appearing in different intensities in the spectra were 1424 cm^{-1} which is the characteristics of $–CH_2–$symmetrical bending. One new notable absorption bands appearing in spectrum (B) in strong to medium intensities at 1648 cm^{-1} clearly depicts the presence of quaternary nitrogen group ($-N^+$-3R) in which the ‘R’ groups are substituted with amino groups. The amide I region lies in the range between 1600 and 1700 cm^{-1}, while amide II and amide III absorption bands comes at around 1530 and 1220 cm^{-1}. Amide I refers to the secondary structure of protein and arises mainly from C=O stretching, with a minor contribution from C-N stretching while the amide II band originates from N-H bending and C-H stretching vibrations. The other new absorption band was observed at peak 2345 cm^{-1} which is the characteristics of $–CH_2$-stretching. Such absorption bands, however, are practically nonexistent in the spectrum of untreated cotton (A). From Figure (3-2), apart from this, an FTIR spectrum (C) showed a larger intensity, which is probably due to additional formation of ester-COO- linkage. Another broader peak at 1000 to 1100 cm^{-1} found only in treated cotton is attributed to –CO-C- deformation and stretching which is absent in spectra (A) for control fabric. Overall, From Figure (3-3), there were no significant changes in the spectra of untreated cotton (A), and modified cotton sample with $MgCl_2$ (D), but a slight difference in the peak intensities of untreated and modified keratin fibers can be observed.

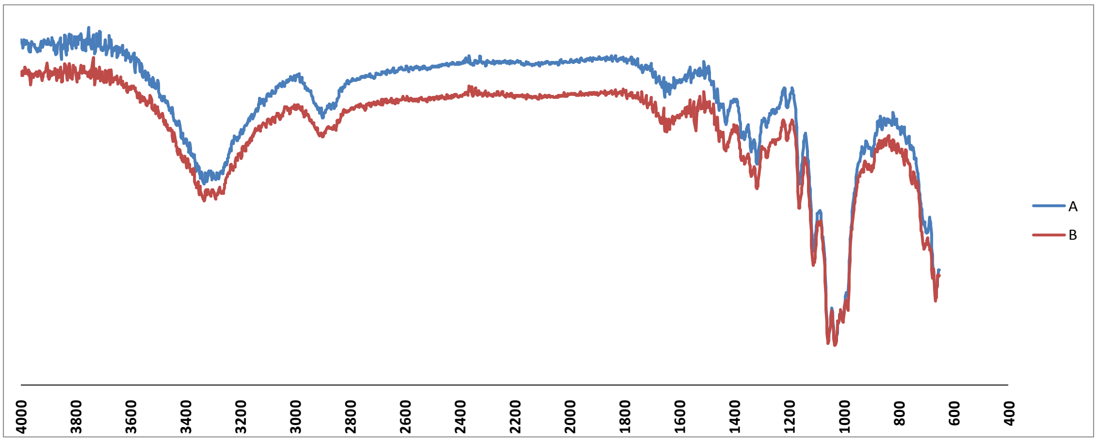

Fig. (3-1) FTIR spectrogram of (a) control fabric and (b) Fabric treated with soybean seed waste.

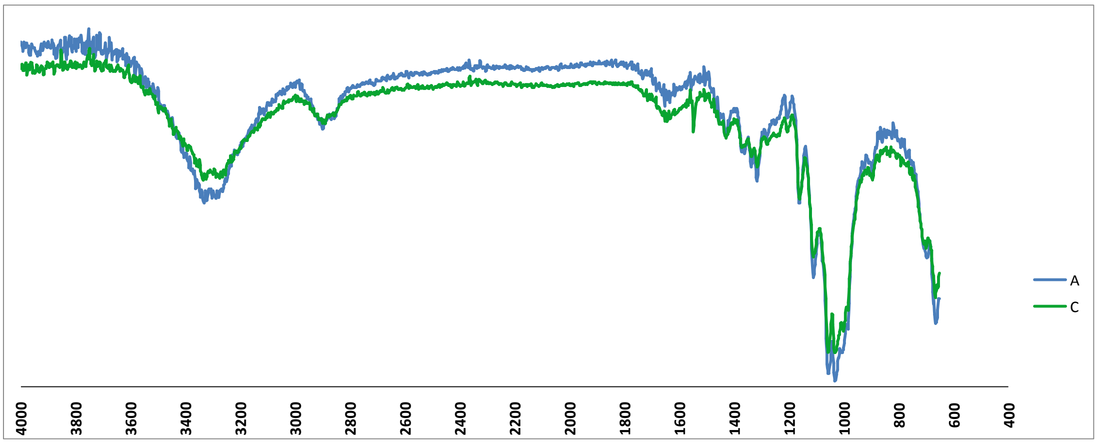

Fig. (3-2) FTIR spectrogram of (a) control fabric and (c) Fabric treated with soybean seed waste + $MgCl_2$.

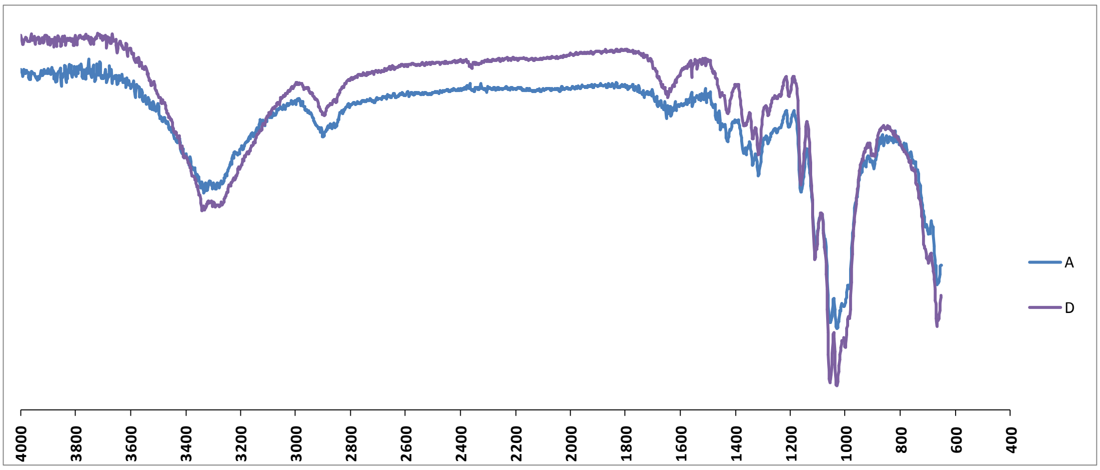

Fig.(3-3) FTIR spectrogram of (a) control fabric and (D) Fabric treated with $MgCl_2$.

Color strength measurements

When we focused on color strength (K/S) we found that there was drastic change of the K/S for cotton fabric samples which represented in Figure (4). It has been noted from the results obtained that the K/S changed with different treatment of cotton samples. Figure (4) clearly indicates that color strength of reactive dyes on cationized cotton fabrics is much higher than untreated cotton fabrics in the absence of salt. The results given in Figure (4) indicate that K/S value of mercerized cotton samples dyed with reactive Orange dye was higher than desized and bleached samples. These results indicate that mercerization treatment has an influence on K/S values due to that cotton fabrics can be considerably modified during mercerization in terms of crystallinity, orientation of crystallites, as well as orientation of macromolecular chains resulting in increased amorphous and less crystalline but with improved orientation of fiber's micro and macro units. Mercerization process involve partial destruction of inter molecular bonds. The fibrous transformation form cellulose I to cellulose II occurs during mercerization, which consists of a swelling of the initial fibers in alkali, followed by recrystallization during subsequent washing and subsequently increase the chemical activity due to the free hydroxyl groups (amorphous cellulose) which react with the dyes more than the un-mercerized samples [25].

Figure (4) clearly indicates that color strength of reactive dyes on cationized cotton fabrics is much higher than untreated cotton fabrics in the absence of salt. The maximum value of K/S was for cotton samples cationized with a mixture of soybean extract and $MgCl_2$. This result may be due to that $MgCl_2$ acts as mild acidic catalyst. Also, our previous work [26], pointed out that a possible interaction between Mg^{2+} and the oxygen atoms of cellulose has been indicated as shown in the Figure (5).

It has been noted that K/S values of cationized cotton fabrics with soybean extract, and cationized cotton fabrics with $Mgcl_2$ was nearly the same. On the other hand, the K/S of cationized cotton samples with a mixture of soybean seed waste extract and $Mgcl_2$ dyed improved and had the highest value followed by cationized cotton fabrics with soybean extract, and the lowest value for cationized cotton fabrics with $MgCl_2$.

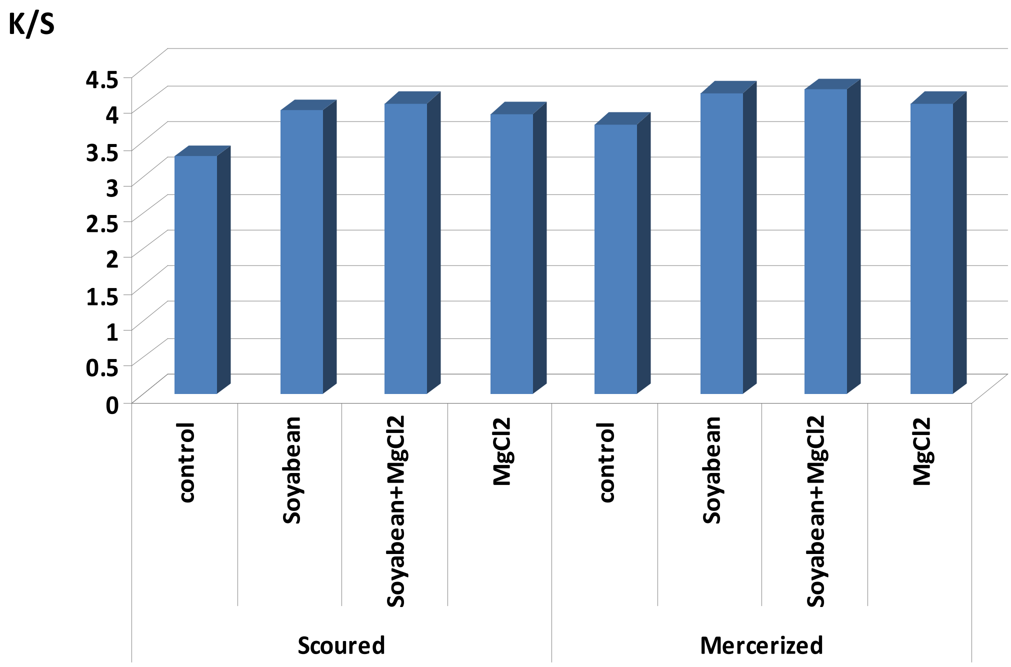

Fig.4. Color strength K/S values of cationized and un-cationized cotton dyed with reactive dye.

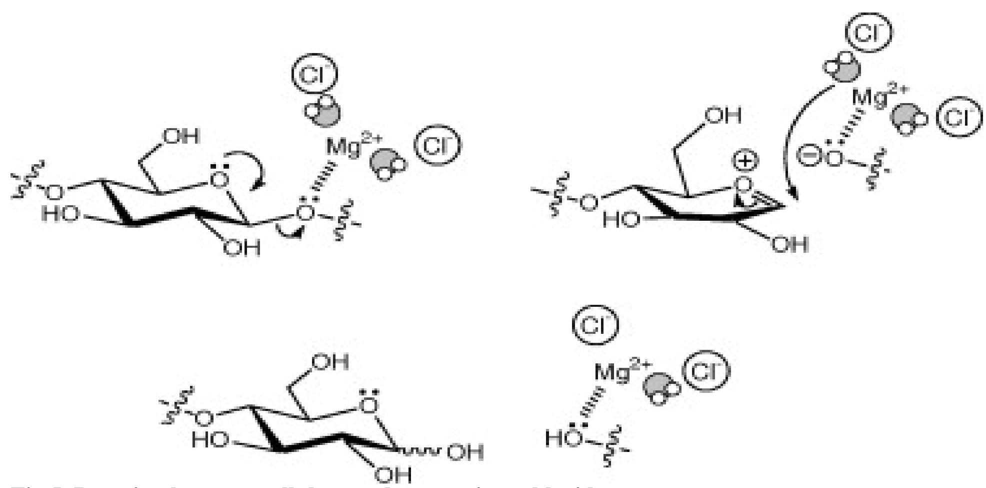

Fig.5. Reaction between cellulose and magnesium chloride.

Effect of cationization on mechanical properties; Tensile strength (Kg/f) and Elongation (%)

Tensile strengths (Kg/f) and elongation of different cotton fabrics were given in table (1). Scoured cotton fabric has higher tensile strength than mercerized samples. The result shows that a small loss that doesn't exceed (3-5%), in tensile strength was observed which might be due to curing effected on degradation of cotton. These results were agreement with the results obtained by Asaye, and Nalankilli [27]. Another assumption is that concentrated sodium hydroxide penetrates inside the fibers and reacts with the hydroxyl groups inside the macromolecule in such a way that it either produces sodium cellulosate or it links to the molecules through the pulling forces increase the amorphous region which lead to decrease the tensile strength. Effect of cationization on mechanical properties is also dependent on physical properties of the treated or cationized cotton fabrics. The tensile strength of the cationized sample showed little improvements due to the fact that cationization occurs mainly at methyl hydroxyl groups of cotton cellulose i.e. at carbon number six (C6) and without breaking the intermolecular hydrogen bonds was very well proved by the fact that the strength is not affected due to modification with amino acid. The improvement was due to the fewer tendencies of the reduction in the number of intermolecular hydrogen bonds by cross-linking of molecule by the cationic reactants. These results were with agreements with the results obtained by Dessiea, and Govindanb [6]. On the other hand, the results in Table (1) showed that the highest value of elongation % was of mercerized cotton samples cationized with Soybean+$MgCl_2$ compared with other treated cotton samples.

Effect of cationization on the color fastness

Cationized fabrics contain positive groups which increase reactive dye exhaustion due to ionic attraction. Good fastness level can be explained by that the amount of fixed reactive dye on cationized cotton fabrics is higher than on untreated fabrics because dye exhaustion is higher on cationized fabrics. That's shown in tables (2) and (3).

Color Fastness to Washing

Color fastness to washing of the cationized fabric was compared with un-cationized dyed cotton fabric shown in Table (2). Wash fastness of the cationized cotton fabric were similar to the conventional dyed cotton fabric. All samples were very good fastness level 4 or 4-5.

Color Fastness to Rubbing

The result in Table (2) showed that the rubbing fastness of all samples were very good grade.4-5. This may be explained by the formation of strong ionic bond between the anionic dye molecule of the reactive dye and the cationic surface of the cotton cationized fabric.

Color Fastness to Light

The light fastness of both un-cationized and cationized fabric dyed salt free was compared. The result in Table 2 showed that the light fastness of the all samples were excellent 6

Color Fastness to perspiration

The staining properties of both the un-cationized and cationized fabric is almost similar in both acidic and alkaline perspiration

TABLE 1. Effect of cationization on mechanical properties of cotton fabrics .

Treatment		Tensile Strength (Kg/f)	Elongation (%)
Scoured	control	22.50	8.85
	Soybean	21.72	9
	Soybean+$MgCl_2$	22.01	9
	$MgCl_2$	21. 2	9.2
Mercerized	control	21.07	10.5
	Soybean	21.15	10
	Soybean+$MgCl_2$	21.00	10.55
	$MgCl_2$	20.20	10.3

Where S = Scoured fabric, and M= Mercerized fabric

TABLE 2. Effect of cationization on the color Fastness (washing, rubbing, and light) of the cotton fabrics .

Treatment		Color Fastness to Washing						Color Fastness to rubbing		Color Fastness to Light
		C. change	Staining							
			dilacerate	cotton	polyamide	polyester	acrylic	dry	wet	
S	control	4	4	4	4-5	4-5	4-5	4-5	4-5	6
	Soybean	4	4	4	4-5	4-5	4-5	4-5	4-5	6
	Soybean+$MgCl_2$	4	4	4	4-5	4-5	4-5	4-5	4-5	6
	$MgCl_2$	4	4	4	4-5	4-5	4-5	4-5	4-5	6
M	control	4	4	4	4-5	4-5	4-5	4-5	4-5	6
	Soybean	4	4	4	4-5	4-5	4-5	4-5	4-5	6
	Soybean+$MgCl_2$	4	4	4	4-5	4-5	4-5	4-5	4-5	6
	$MgCl_2$	4	4	4	4-5	4-5	4-5	4-5	4-5	6

S and M as mentioned in Table (1).

TABLE 3. Effect of cationization on the color fastness to perspiration of the cotton fabrics .

Treatment		Color Fastness to perspiration (alkaline)						Color Fastness to perspiration (acidic)					
		C. change	Staining					C. change	Staining				
			diacetate	cotton	polyamide	polyester	acrylic		diacetate	cotton	polyamide	polyester	acrylic
S	Control	4	3-4	4-5	4-5	4-5	4-5	4	3-4	3-4	4-5	4-5	4-5
	Soybean	4	4	3-4	4-5	4-5	4-5	4	3-4	3-4	4-5	4-5	4-5
	Soybean+$MgCl_2$	4	3-4	3-4	4-5	4-5	4-5	4	3-4	4	4-5	4-5	4-5
	$MgCl_2$	4	3-4	3	4-5	4-5	4-5	4	4	4	4-5	4-5	4-5
M	control	4	4	4	4-5	4-5	4-5	4	3	3-4	4-5	4-5	4-5
	Soybean	4	4-5	3-4	4-5	4-5	4-5	4	4	4	4-5	4-5	4-5
	Soybean+$MgCl_2$	4	4	3-4	4-5	4-5	4-5	4	3-4	4	4-5	4-5	4-5
	$MgCl_2$	4	4	3-4	4-5	4-5	4-5	4	3-4	4	4-5	4-5	4-5

S and M as mentioned in Table (1).

Conclusion

The present investigation provided a method of salt free dyeing of cotton fabric with reactive dyes by modifying cotton with soybean seed waste as a source of amino acid. A comparison of reactive dyed cationized and un-cationized cotton using no salt technique was done. The infrared analysis (FTIR) before and after cationization of cotton confirmed that there was a change in chemical composition confirmed that the amino group was formed in cotton fabrics. An important advantage of cationic treatment is in the economic and environmental impact by using the waste of soybean and minimize the environmental problems occurred in reactive dyeing by making salt-free reactive dyeing.

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