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Preparation and Utilization of Fixing Agents for Dyed Cotton Fabrics

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PREPARATION of new cationic fixing agents and their utilization after dyeing cotton fabrics using reactive and direct dyes are studied. Both stock solution, which used in preparation of fixing agents and prepared fixing agent are analyzed to detect the percent of nitrogen, carbon and hydrogen atoms. The application of the prepared fixing agents after dyeing cotton fabrics either using reactive or direct dyes are investigated. The newly prepared fixing agents could be successfully used for fixing different types of dyes and in general, their points possess color strength value comparable or even higher to those obtained upon using selected commercial fix.

Keywords: Anionic dyes, Direct dyes, Fixing agents, Reactive dyes, Preparation.

Introduction

Cotton is the most widely used fiber in the world, due to good comfort and absorbency. Abundantly up to more than 50%, consumption of textile is made up of cellulose [1]. This cotton fabric is dyed with different types of dyes, and these dyes are organic colored compounds. Today customer needs good quality of cloth with good fastness properties and according to end use such as curtain should have good fastness to light, car seat cover should have good rubbing fastness**.** The anionic dyes are defined as a dye which colored ion is anionic in character containing sulphonic or carboxylic groups. These dyes used for dyeing natural fibers such as cotton, and wool [2]. Among the anionic dyes, direct and reactive dyes are the most popular dyes classes used for the dyeing of cellulosic fibers. Direct dyes very suitable for cotton, this is due to the linear and planar structure of the dye molecules which enable close with chains of cellulose molecules resulting in significant hydrogen bonding, but the wet fastness properties of direct dyes are not good for end uses and so these dyes are replaced by reactive dyes which have better wet fastness and brightness in many hues but the prime advantages of direct dyes are ease of application. Wide range of shade and economy compared with dyes of higher fastness (reactive, sulphur or vat). Reactive dyes are widely used for the dyeing of cotton (cellulosic fibers) because of their wide shade range and excellent wet fastness, which arises from a covalent bond formed between dye and the fiber. Generally, the dyed cellulosic fibers have a fastness to washing that does not meet the requirements of today's consumers. This is not only for many direct dyes but also for reactive dyes [3].

Practically, for every dyeing or printing process the use of auxiliaries is very important. Different auxiliaries improve the application, characterization and performance of penetration, leveling, fixation and shade deepening to provide brilliant intense dyeing. The class of auxiliaries which is used to improve the dye fastness is called fixing agents these fixing agents function by forming a dye – fixing agent complex of large molecular size and reduced aqueous solubility and therefore higher wet fastness[4]. Cationic fixing agents are most widely used for pretreatment and after treatment.

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Cationic fixing agents were initially applied as after treatments to the dyed textile fibers to improve their wet fastness properties [5, 6]. Later on, this limitation of mono functional cationic fixing agents has been overcome by the development of forming poly functional cross-linking fixing agents, which carry reactant groups capable of forming bonds that are more permanent with other suitable groups in the dye or fiber [7, 8]. For enhancing the dye ability of cellulosic fibers with reactive or direct dyes by pretreatment with a great variety of cationic products usually based on nitrogen. This modification of cellulosic fibers with cationic agents resulted in increased substantivity of anionic dyes for cellulosic fibers by introducing new cationic sites. Lewis and Lei reviewed numerous chemicals that can be used to provide cationic changes to cotton fibers [9]. Pretreatment of cellulosic fibers with cationic agents has been reported to enhance the uptake of anionic dyes and facilitate the fixation of reactive dyes in the absence of either salt or alkali [10, 11]. Amines, quaternary ammonium, phosphonium and tertiary sulphonium compounds can be used as dye fixing agents [12-15]. The aim of this work was to prepared new cationic fixing agents for overcome on, the wet fastness properties of anionic dyes.

Materials and Methods

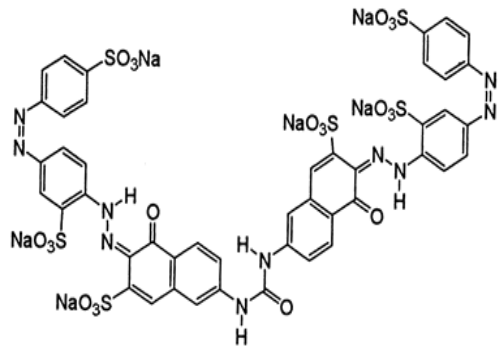
Materials

Fabrics

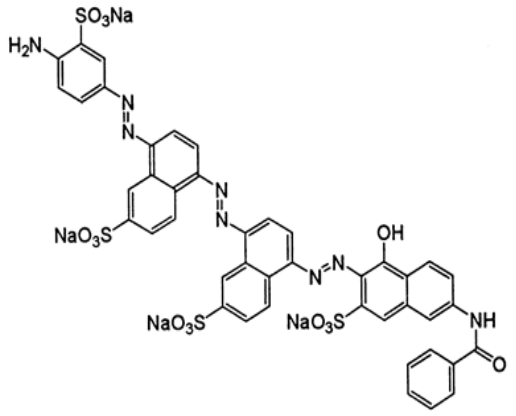
Mill scoured, bleached and mercerized plain weave cotton fabric (135 g/m^2) kindly supplied by Miser Co., El-Mahalla Elcobra.

Dyestuffs

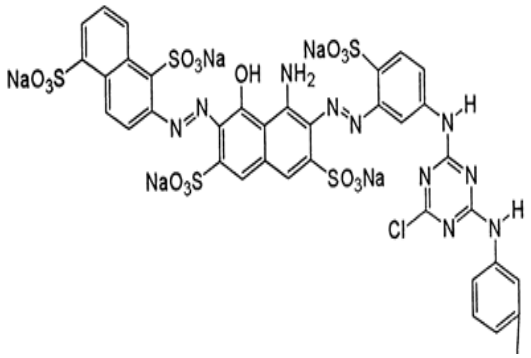
Direct Red (C.IRed80) , Direct blue(C.I. blue 85), and Reactive Red (C.I. Red 195) , Reactive blue(C.I. Blue 222)



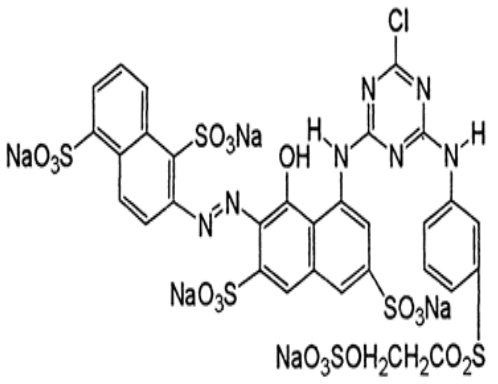
Direct Red (C.I direct 80)



Direct Blue (C.I direct 85)



Reactive Blue (C.I. reactive 222)



Reactive Red(C.I. reactive 195)

Chemicals

Di - ammonium phosphate, ammonium chloride, phosphoric acid, sequestering agent, buffer solution, commercial fixing agent were of laboratory grade chemicals were used.

Methods

Preparation of new fixing agents

The first step is to prepare a stock solution of di-ammonium phosphate with ammonium chloride in the presence of phosphoric acid. The second step is preparing different types of fixing agents from the stock by using different concentrations as follows:

Fix1: prepared using 150g of Stock, and150g Sequestering agent, and 50g Buffer soln., and 650g water to get a finally 1000 g.

Fix2: prepared using 250g of Stock, and 150g Sequestering agent, and 50g Buffer soln., and 550g water to get a finally 1000 g.

Fix3: prepared using 500g of Stock, and150g Sequestering agent, and 50g Buffer soln., and 300g water to get a finally 1000 g.

Dyeing Procedure

Dying cotton fabrics with reactive dyes

The samples of cotton fabrics weighing 5g, dyeing in 200 ml beakers with the two dyes, C.I.Blue222 and C.I. Red 195, at the liquor: ratio, 1: 30 using different concentrations for each dyes. Dyeing was carried out by adding 150ml water, 1g sequestering agent, 1g leveling agent, the dye either C.I.blue222 and /or C.I. Red 195 as required concentration (1%, 2%, 3%) O.W.F Then stay the temperature at 35ºc for 10 min., the salt was added according to the required shade from 30 to 80 g/L, then raise the temp. , gradually to 60°c and then stay for 30 min., then added the soda ash gradually according to the required shade from 5 to 20 g /L ,then adjust the PH from 10.4 to 10.6 , and then stay the temp. at 60 °c for 45 min. then rains by cold water, neutralization using 1g/l acetic acid at 40°c for 5 min. , rains to get ride from acetic acid then soaping(1g/L) soap at 90°c then rains by hot water then cold water, the dyed samples dried at room temperature.

Dying cotton fabrics with direct dyes

The samples of cotton fabrics weighing 5g, dyeing in 200 ml beakers with the two dyes, direct blue (C.I. Blue 85) and/ or direct red (C.I. Red 80), at the liquor: ratio, 1: 30 using different concentrations for each dyes. Dyeing was carried out by adding 150ml water, 1g sequestering agent, 1g leveling agent, the dye either direct blue or direct red as required concentration (1%, 2%, 3%) O.W.F, then stays the temperature at 35ºc for 10 min., and then raises the temp. Gradually to 100°c, the salt was added according to the required shade 30 to 80 g/L and then stays the temp. for 30 min. then rains by cold water, rains again by hot water at 70°c .

Treatment of dyed fabrics using fixing agent

Different concentrations (5 , 10, 15 and 20 g/L) of the three prepared fixing agent fix.1, fix.2, fix.3 and commercial fixing agent were used for treatment dyed cotton fabrics, in the presence of 0.5 g/L formic acid and water (liquor ratio 1:25), then heat to 60°c for 15 min.

Measurements and Analysis

Elemental analyses

Elemental analyses were performed on Carlo Erba 1108 Elemental Analyzer (Heraeus, Hanau, Germany). Analyses were carried out by the Micro analytical Research Center, Faculty of Science, Cairo University [16].

Color strength

The relative color strength of the prints expressed as K/S value [17] of the colored samples was determined by reflection measurements using data color international model SF 500, USA.

Fastness properties

Fastness to washing, rubbing, and perspiration was assessed according to the standard methods [18, 19]

Results and Discussion

Reactive dyes are extensively used for exhaustion dyeing of cotton fiber; it provides wide range of shades of good light and washing fastness on cellulosic fibers. These dyes suffer the disadvantage that dye fiber reaction is not 100% efficient. Exhaustion of the dye is incomplete due to the dyes are not react only with the fiber nucleophile reactive sites but also with nucleophiles present in the dye bath, to dye hydrolysis [20]. To improve the fastness properties of reactive dyes cotton fabric we should use cationic fixing agents [21]. Reactive dyes are the most important class of dyes used for cellulosic substrate because their high wet fastness, brilliance and range of hues [22, 23]. The most attractive feature of the use of these dyes is the simple dyeing process. The improvements in the wet fastness properties of these reactive (anionic) dyes can be brought about by pre-treatment or after treatment of textile

fibers. Various pre-treatment and after treatments system have been developed but at the moment most widely used are formaldehyde based cationic dye fixing agents.

Analyses of new fixing agents prepared

Table 1. Show the % of C, H, and N elements in the prepared fixing agent, (stock fix, fix.1, fix.2, fix.3), and Commerciale fix. From the Table we noticed that, the stock fix has the highest percent of nitrogen, the percent of N, is 6, 2.6, 3.36, 4.46, and 4.39 in case of stock fix, fix.1, fix.2, fix.3, and Commerciale fix respectively.

Effect of fixing concentration on color strength (K/S) Values.

Dyeing of cotton fabrics with red reactive dye

Figures 1-3 show the effects of fixing agents concentration on the color strength (K/S) of the dyed cotton fabrics upon using red reactive dye at different concentration 1, 2, and 3% respectively. From the Figures, we noticed that increase the fixing agent concentration from 5 to 20 g/l, this is lead to slightly increase in the K/S, this is true irrespective of either the type of fixing agent or the concentration of the dye used. At 1&2% concentration of dye used the uses of fixing1 and 2 give higher color strength but at 3 % concentration fixing 3 give slightly increase compared with fixing 1 and 2 , but in all cases the prepared fixing give color strength better than the commercial fixing used, this is may be due to the increase in the % of nitrogen as show in Table 3.1 and the presence of ammonia group in the prepared fixing agent which make swelling in the yarn of the cotton fabrics and increase in the dye uptake. The new fixing agents used are based on "Di- ammonium phosphate and ammonium chloride mixture" they increase their fixing power by mixing with hexa meta phosphate and buffering the solution by citric acid and potassium citrate .These increasing the dye uptake in cellulosic fabrics and improve the wet fastness properties of anionic dyes according the following equations:

$(NH_4)_2HPo_4 \rightarrow NH_3 + NH_4H_2Po_4$

$NH_3+H_3Po_4 \rightarrow NH_4H_2Po_4$

$NH_4H_2Po_4 \rightarrow NH_4^+ + H_2Po_4^-$

$NH_4Cl \rightarrow NH_4^+ + Cl^-$

TABLE 1. The % of C, H, and N elements for, the stock fix. fix.1, fix.2, fix.3, and Commerciale fix.

Elemental analyses	Stock Fix.	FIX. 1	FIX. 2	FIX. 3	Commerciale Fix.
C %	0.46	0.4	0.38	0.30	0.70
H %	8.33	8.51	9.29	9.89	11.5
N %	6	2.6	3.36	4.46	4.39

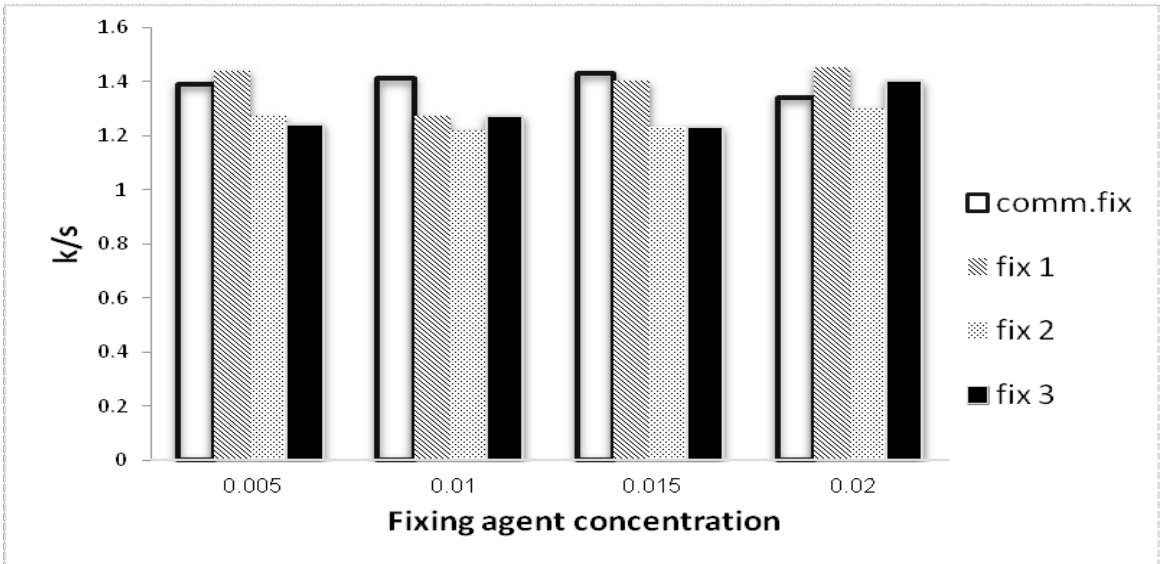


Fig.1. Effect of fixing agents concentration on the color strength of dyed cotton fabric using red reactive dye 1% shade.

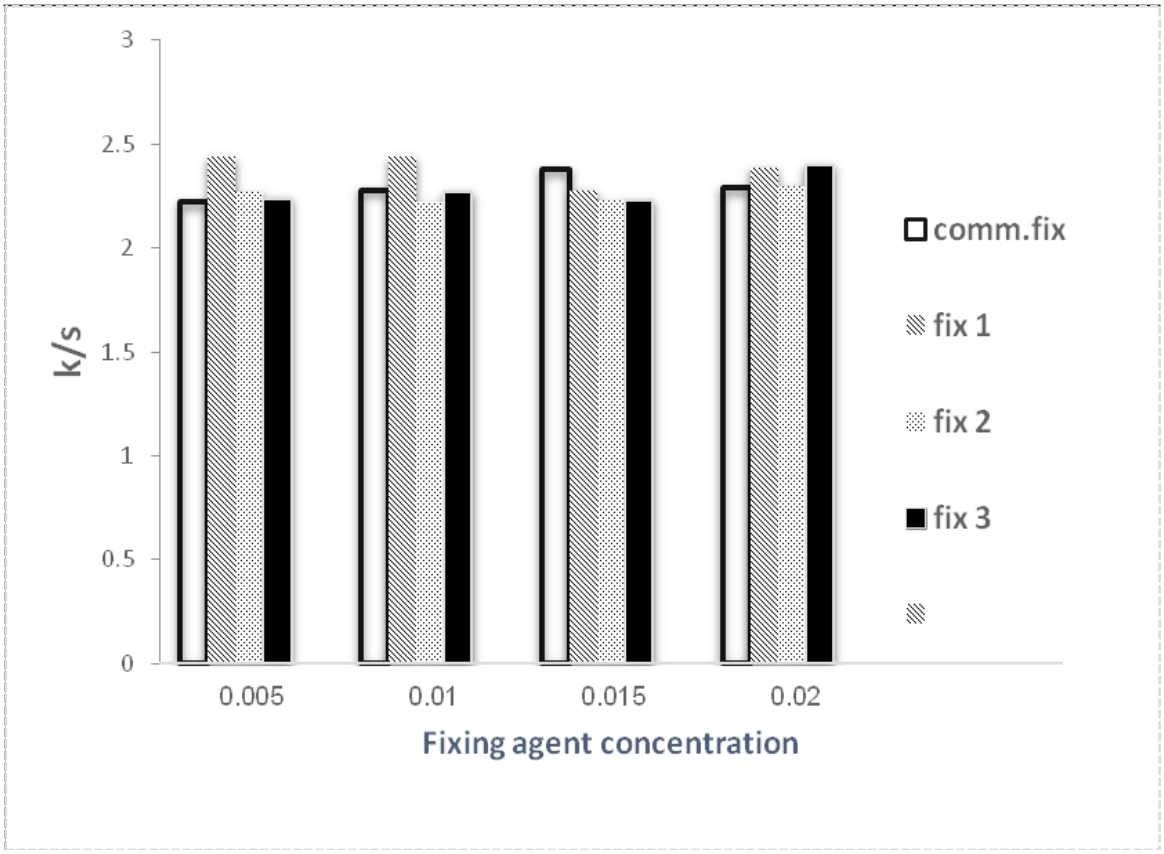


Fig.2. Effect of fixing agents concentration on the color strength of dyed cotton fabric using red reactive dye 2% shade.

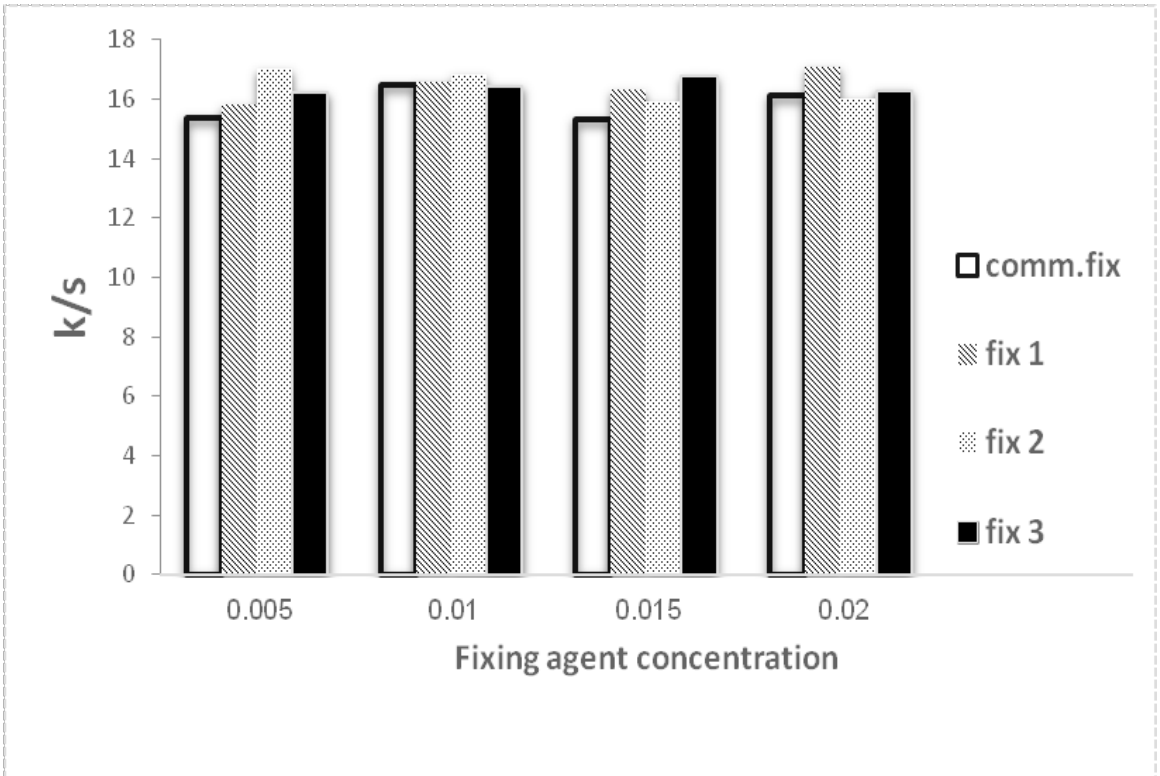


Fig.3. Effect of fixing agents concentration on the color strength of dyed cotton fabric using red reactive dye 3% shade.

It could be concluded that the uses of 10 g/L of prepared fixing in treatment of the dyed cotton fabric could be considered as optimum conditions in this case. The increase the fixing agent concentration more than 10 g/L this is lead to either constant or slightly increase in the color strength. From Figures 1-3, we noticed that increases the dye conc. this is lead to increases in the K/S, for example increase the dye conc., from 1, 2 and 3%, using 10 g/L of commercial fixing and/or fixing1 and/or fixing2 and/or fixing3 the K/S increase from 1.41, 2.28 and16.49and/or 1.27, 2.44 and 16.6and/or 1.22, 2.22 and 16.83 and/or 1.27, 2.27 and 16.44 respectively**.**

Dyeing of cotton fabrics with blue reactive dye

Figures 4-.6 show the effects of fixing agents concentration on the color strength (K/S) of the dyed cotton fabrics upon using blue reactive dye at different concentration 1, 2, and 3% respectively. From the Figures, we noticed that increase the fixing agent concentration from 5 to 20 g/l, this is lead to slightly increase in the K/S, this is true irrespective of either the type of fixing agent or the concentration of the dye used. Similar pattern was obtained upon using blue reactive dye like that upon using red reactive dye.

Dyeing of cotton fabrics with red direct dye.

Direct dyes represent an extensive range of colorants that are easy to apply and also are very economical. Direct dyes have an affinity for a wide variety of fibers such as cotton, viscose, silk jute, linen …etc. They do not make any permanent chemical bond with the cellulosic fibers but are attached to it via very week hydrogen bonding as well as Vander Waals forces. Their flat shape and their length enable them to lie along-side cellulose fibers and maximize the Vander Waals, dipole and hydrogen bonds. Direct dyes are usually applied with the addition of electrolyte at or near the boil in the machines capable of running at atmospheric pressure. When cellulose is immersed in a solution of a direct dye it absorbs dye from the solution until equilibrium is attained, and at this stage most of the dye is taken up by the fiber. The rate of absorption and equilibrium exhaustion varies from dye to dye. The substantivity of the dye for cellulose is the proportion of the dye absorbed by the fiber compared with that remaining in the dye bath. Direct dyes vary widely in their fastness properties, and staining effects on various fibers. Most direct dyes, however, have limited wet fastness in medium to full shades unless they are after-treated. The fastness of selected direct dyes can be improved in several ways, such as, by treatment with fixing agents.

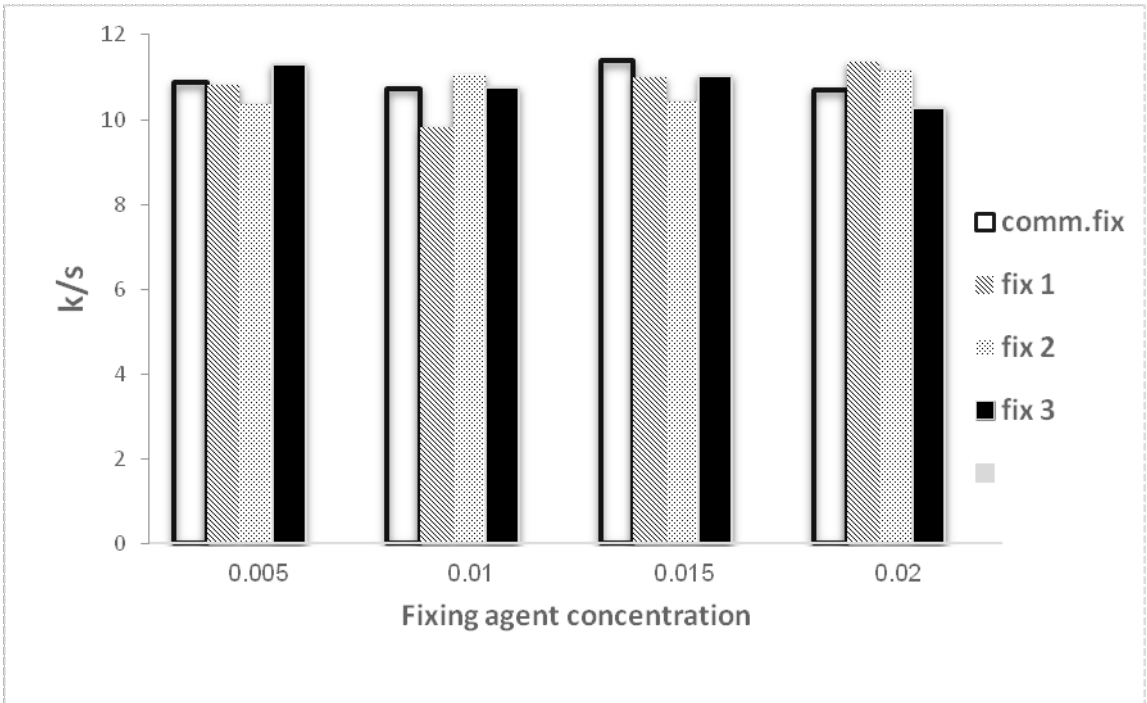


Fig.4. Effect of fixing agents concentration on the color strength of dyed cotton fabric using blue reactive dye 1% shade.

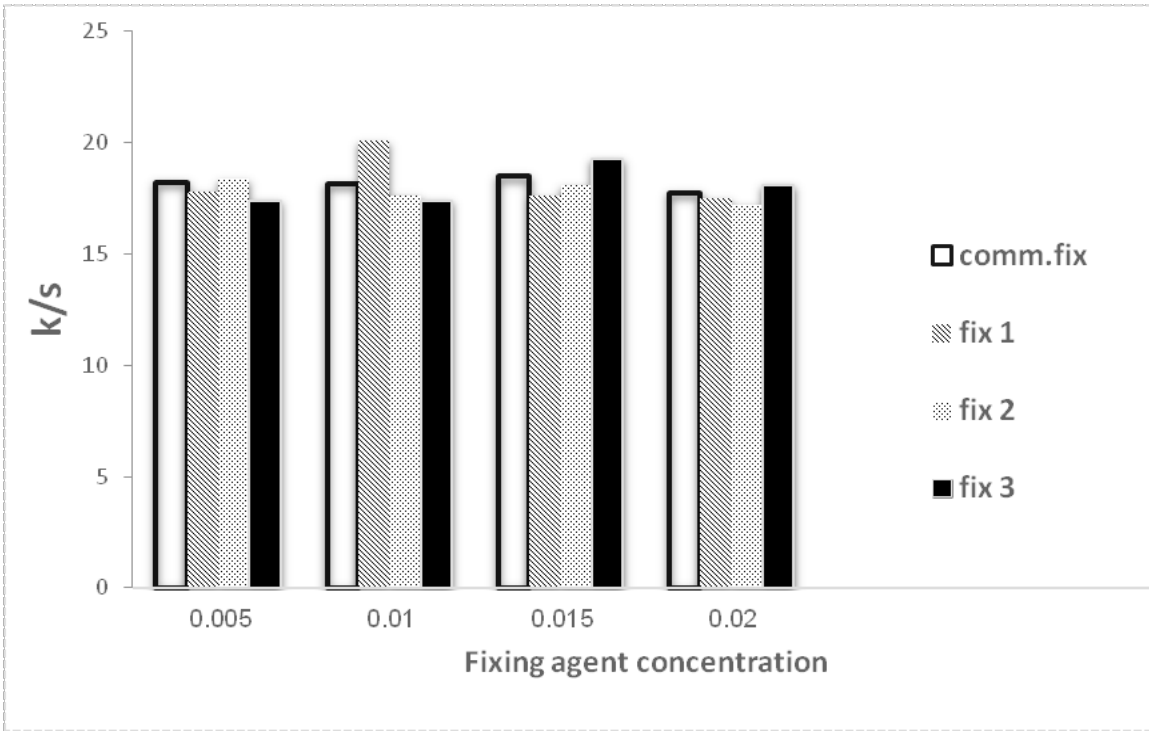


Fig. 5. Effect of fixing agents concentration on the color strength of dyed cotton fabric using blue reactive dye 2% shade.

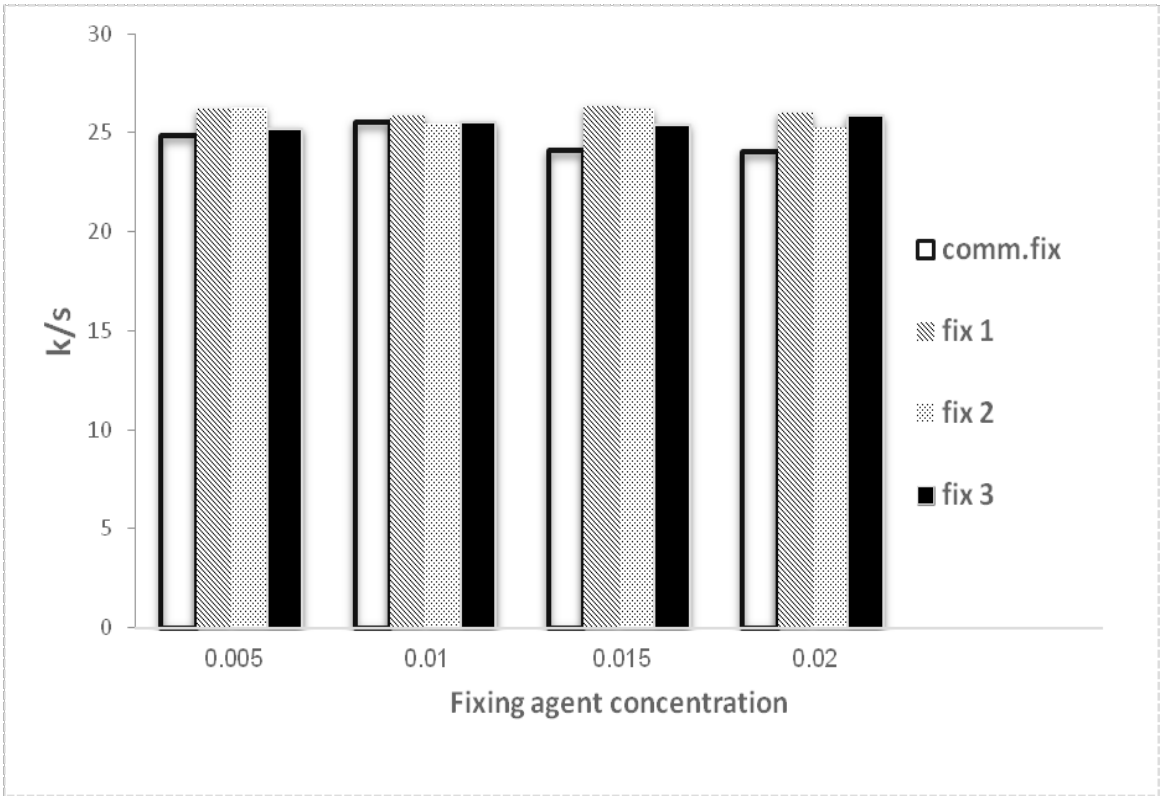


Fig. 6. Effect of fixing agents concentration on the color strength of dyed cotton fabric using blue reactive dye 3% shade.

Figures 7-9 show the effects of fixing agents concentration on the color strength (K/S) of the dyed cotton fabrics upon using red direct dye at different concentration 1, 2, and 3% respectively From the Figures7-9, we noticed that increase the fixing agent concentration from 5 to 20 g/l, this is lead to slightly increase or decrease in the K/S, this is true irrespective of either the type of fixing agent or the concentration of the dye used. At 1% concentration of dye used the uses of fixing2 give higher color strength but at 2 and 3 % dye concentration fixing 3 give slightly increase compared with fixing 1 and 2, but in all cases the prepared fixing give color strength equal or higher than the commercial fixing used, this is may be due to the presence of ammonia group in the prepared fixing agent which make swelling in the yarn of the cotton fabrics and increase in the dye uptake.

It could be concluded that the uses of 10 g/L of prepared fixing in treatment of the dyed cotton fabric with red direct dye could be considered as optimum conditions in this case. The increase the fixing agent concentration more than 10 g/L this is lead to either constant or slightly increase in the color strength. From Figures 7-9, we noticed that increases the dye conc. this is lead to increases in the K/S, for example increase the dye conc., from 1, 2 and 3%, using 10 g/L of commercial fixing and/or fixing1 and/or fixing2 and/or fixing3 the K/S increase from 12.33, 15.11 and18.53and/or 11.62, 13.28 and 18.42and/or 12.52, 14.1 and 18.12 and/or 11.03, 14.85 and 18.65 respectively**.**

Dyeing of cotton fabrics with blue direct dye.

Figures 10-12 show the effects of fixing agents concentration on the color strength (K/S) of the dyed cotton fabrics upon using blue direct dye at different concentration 1, 2, and 3% respectively. From the Figures, we noticed that increase the fixing agent concentration from 5 to 20 g/l, this is lead to slightly increase in the K/S, this is true irrespective of either the type of fixing agent or the concentration of the dye used. Similar pattern was obtained upon using blue direct dye like that upon using red direct dye. The increase the dye conc., from 1, 2 and 3%, using 10 g/L of commercial fixing and/or fixing1 and/or fixing2 and/or fixing3, the K/S increase from 10.23, 16.48 and 22.58and/or 10.08, 16.52 and 21.86 and/or 10.4, 16.81 and 23.14 and/or 10.25, 15.28 and 22.8 respectively**.**

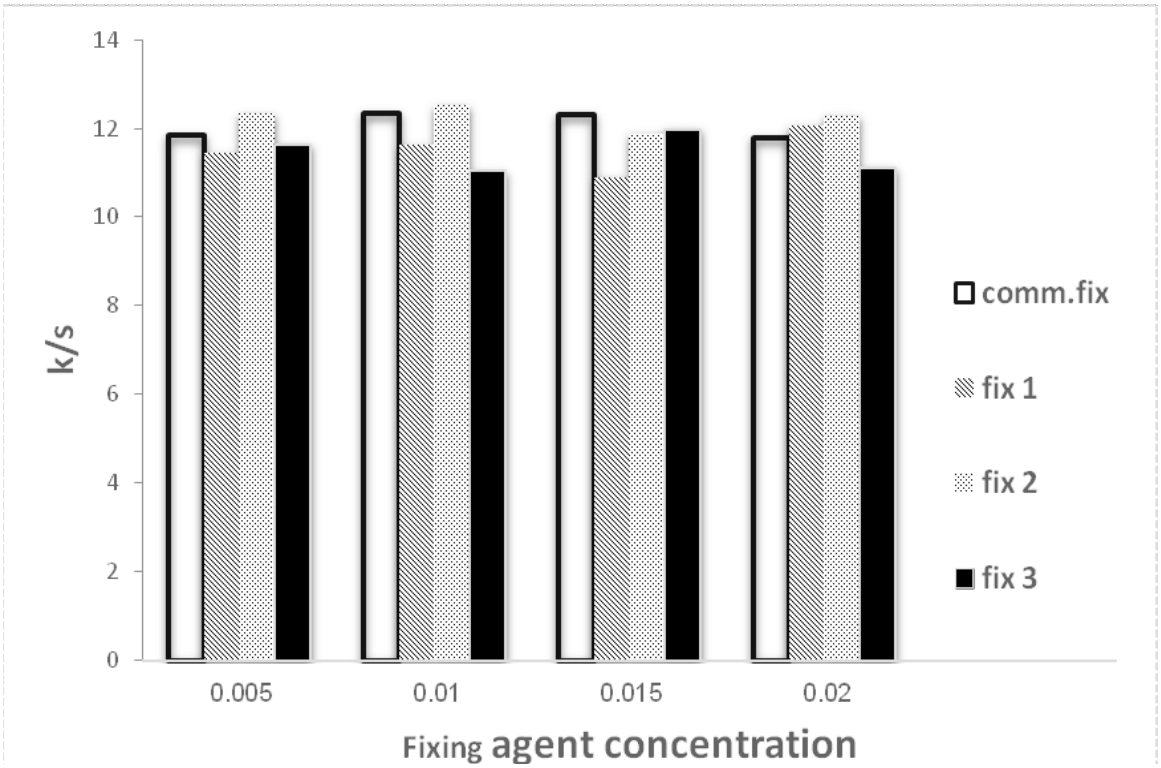


Fig. 7. Effect of fixing agents concentration on the color strength of dyed cotton fabric using red direct dye 1% shade.

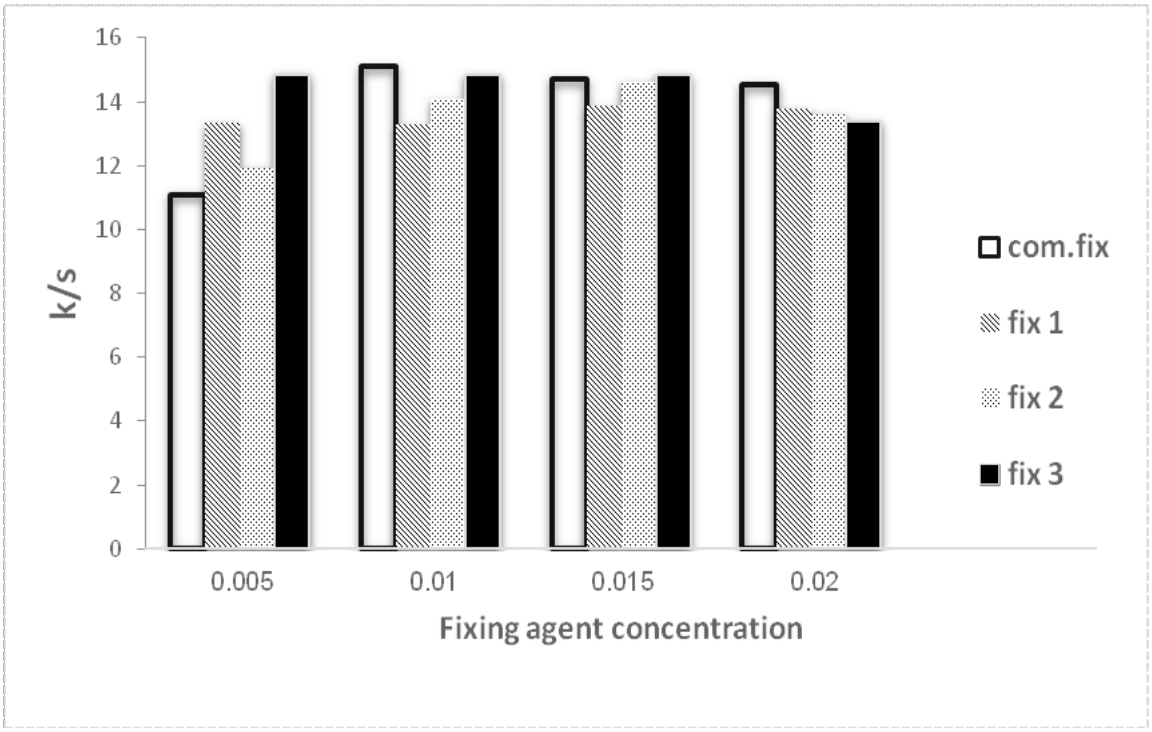


Fig.8. Effect of fixing agents concentration on the color strength of dyed cotton fabric using red direct dye 2% shade.

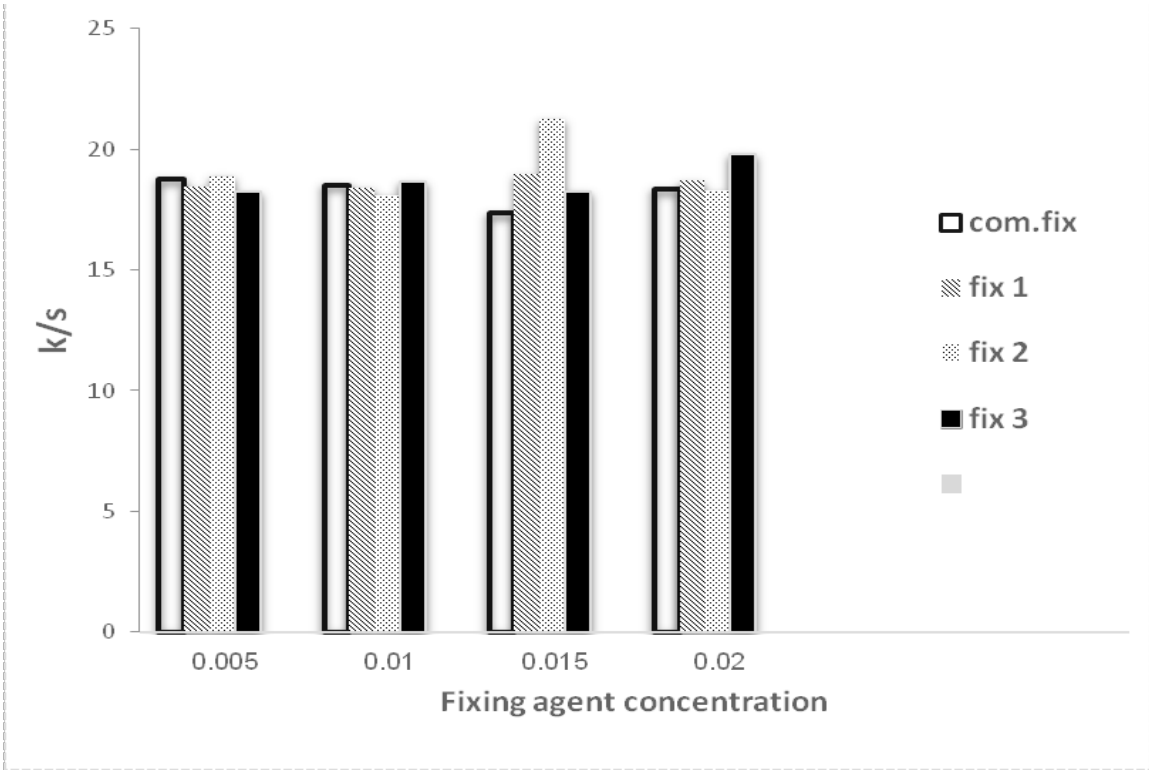


Fig. 9. Effect of fixing agents concentration on the color strength of dyed cotton fabric using red direct dye 3% shade.

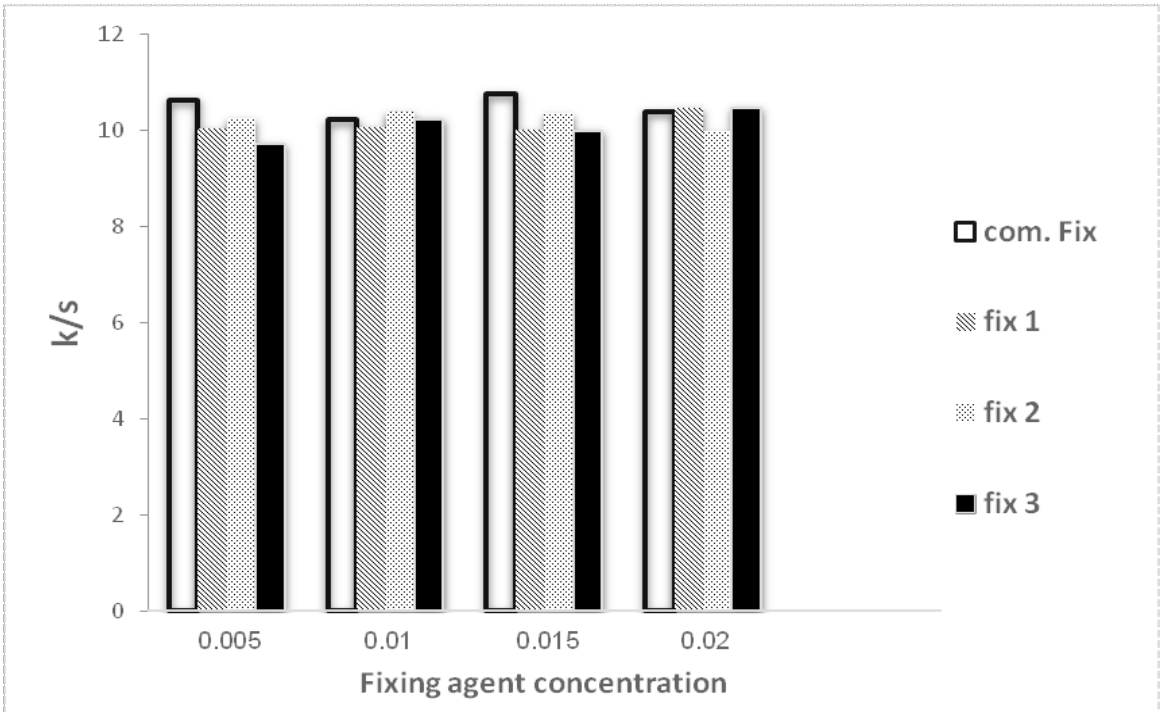


Fig. 10. Effect of fixing agents concentration on the color strength of dyed cotton fabric using blue direct dye 1% shade.

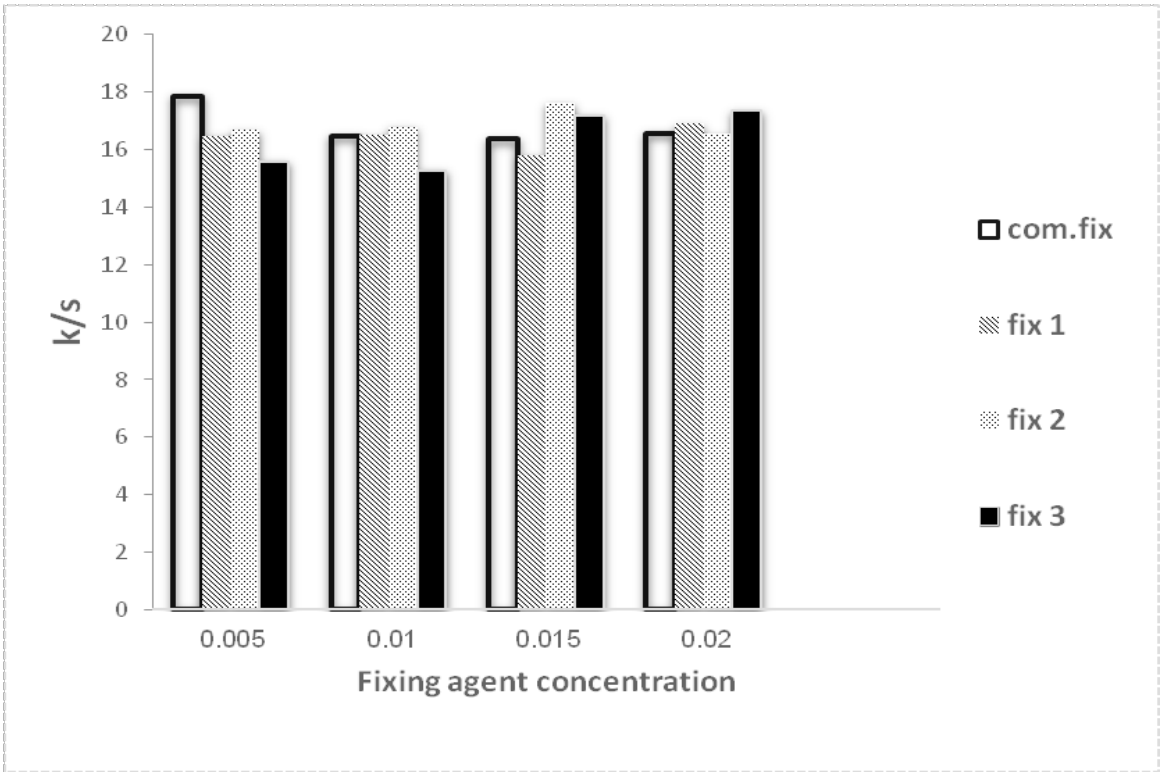


Fig.11. Effect of fixing agents concentration on the color strength of dyed cotton fabric using blue direct dye 2% shade.

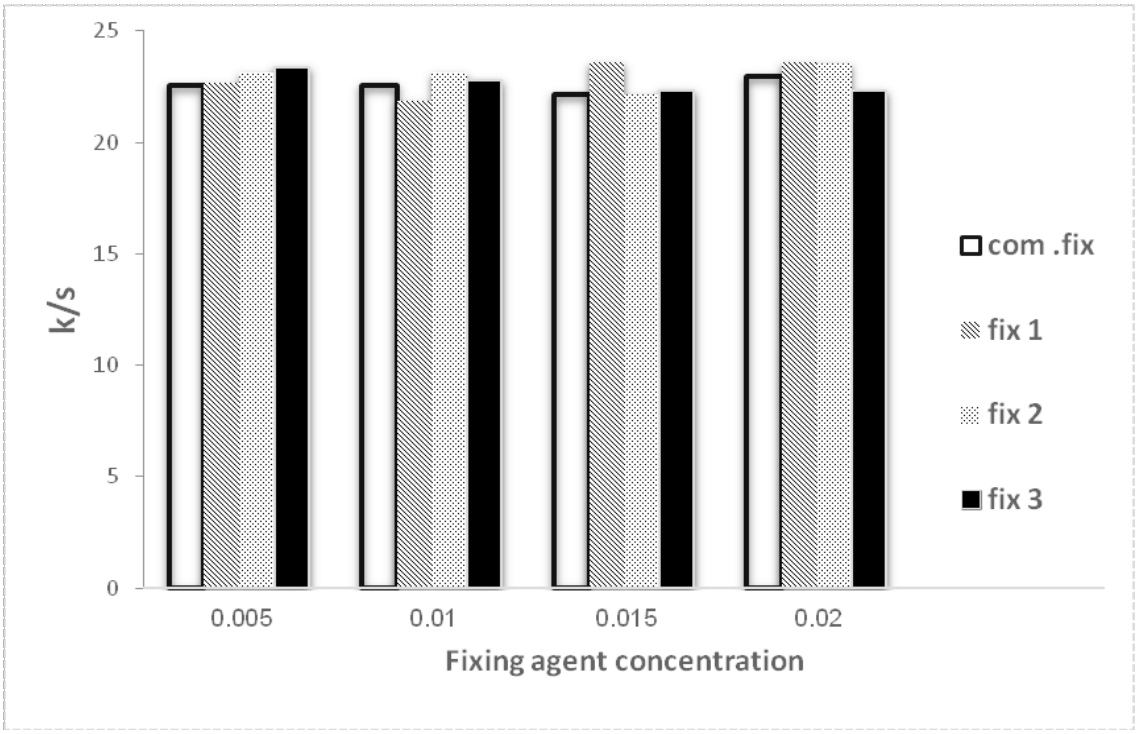


Fig.12. Effect of fixing agents concentration on the color strength of dyed cotton fabric using blue direct dye 3% shade.

Fastness properties of dyed cotton fabrics using reactive dyes.

Tables 2 &.3 show, the overall fastness properties of dyeing cotton fabrics using red and blue reactive dye, at different concentration of the dye and using 10g/L fixing agent respectively. It is clear from the data Tables 2 & 3 that, the overall fastness properties to rubbing, their values range between good to very good in case of wet and from very good to excellent in case of dry, the overall fastness properties to washing, and perspiration for the dyed cotton fabrics their values ranging from very good to excellent, the improve in the fastness upon using fixing agent after dyeing this is may be due to more electrostatic interactions and van der Walls forces, over that the covalent bond which had been formed between the reactive dye and the cellulosic fabrics.

*Fastness properties of dyed cotton fabrics using direct dyes***.**

Tables 4 & 5 show, the overall fastness properties of dyeing cotton fabrics using red and blue direct dye, at different concentration of the dye and using 10g/L fixing agent respectively. It is clear from the data Tables4 & 5 that, the overall fastness properties to rubbing, their values is good in case of wet and is very good in case of dry, the overall fastness properties to washing, and perspiration for the dyed cotton fabrics their values ranging from good to very good, the improve in the fastness upon using fixing agent after dyeing this is may be due to more electrostatic interactions and van der Walls forces are created.

Conclusions

The uses of 10 g/L of prepared fixing agent for treatment of the dyed cotton fabric using either reactive or direct dyes, could be considered as optimum conditions**.**

The overall fastness properties for the dyed cotton fabrics upon using either red or blue reactive dyes to rubbing, are improved upon using fixing agent, and their values range between good to very good in case of wet and from very good to excellent in case of dry rubbing, also the overall fastness properties to washing, and perspiration their values ranging from very good to excellent.

The overall fastness properties to rubbing washing, and perspiration for the dyed cotton fabrics upon using either red or blue direct dye, are improved upon using fixing agent and their values ranging from good to very good**.**

TABLE 2. Fastness properties of dyed cotton fabrics using reactive red, and fixed after dyeing using 10g/L fixing agent.

Fixing Used	Conc. of dye	Fastness to rubbing		Wash fastness			Fastness to Perspiration					
							Alkaline			Acidic		
		Dry	Wet	Alt	SC	SW	Alt	SC	SW	Alt	SC	SW
Commercial Fix	1%	4	3-4	4	4-5	4-5	4	4	4	4-5	4	4
	2%	4-5	4	4	4-5	4-5	4	4	4	4-5	4	4
	3%	4	3	3-4	4-5	3	4	4	4	4-5	4	4
Fix.1	1%	4-5	3-4	4	5	5	4-5	4	4	4-5	4	4
	2%	4-5	4	4	5	5	4-5	4	4	4-5	4	4
	3%	4-5	3-4	3-4	4-5	3	4-5	4	4	4-5	4	4
Fix.2	1%	4-5	3-4	4	5	5	4-5	4	4	4-5	4	4
	2%	4-5	4	4	5	5	4-5	4	4	4-5	4	4
	3%	4-5	3-4	3-4	4-5	3	4-5	4	4	4-5	4	4
Fix.3	1%	4-5	3-4	4	5	5	4-5	4	4	4-5	4	4
	2%	4-5	4	4	5	5	4-5	4	4	4-5	4	4
	3%	4-5	3-4	3-4	4-5	3	4-5	4	4	4-5	4	4

Alt: color change of dyed sample; SC: staining on cotton; SW: staining on wool, Wash-scale (1-5).

TABLE 3. Fastness properties of dyed cotton fabrics using reactive blue, and fixed after dyeing using 10g/L fixing agent.

Fixing Used	Concentration of dye	Fastness to rubbing		Wash fastness			Fastness to Perspiration					
							Alkaline			Acidic		
		Dry	Wet	Alt	SC	SW	Alt	SC	SW	Alt	SC	SW
Commercial Fix	1%	4	3-4	3-4	4-5	4	4	4-5	4-5	4	4-5	4-5
	2%	4	3-4	3-4	4	3-4	4	4-5	4-5	4	4-5	4-5
	3%	4	3-4	3-4	3-4	3	4	4-5	4-5	4	4-5	4-5
Fix.1	1%	4-5	4	4	5	4-5	4-5	5	5	4-5	4-5	4-5
	2%	4-5	4	4	4-5	4	4-5	5	5	4-5	4-5	4-5
	3%	4-5	4	4	4	3-4	4-5	5	5	4-5	4-5	4-5
Fix.2	1%	4-5	4	4	4-5	4	4-5	5	5	4-5	4-5	4-5
	2%	4-5	4	4	4-5	4	4-5	5	5	4-5	4-5	4-5
	3%	4-5	4	4	4	3-4	4-5	5	5	4-5	4-5	4-5
Fix.3	1%	4-5	4	4	4-5	4	4-5	5	5	4-5	4-5	4-5
	2%	4-5	4	4	4-5	4	4-5	5	5	4-5	4-5	4-5
	3%	4-5	4	4	4	3-4	4-5	5	5	4-5	4-5	4-5

Alt: color change of dyed sample; SC: staining on cotton; SW: staining on wool, Wash-scale (1-5).

TABLE 4. Fastness properties of dyed cotton fabrics using direct red, and fixed after dyeing using 10g/L fixing agent.

Fixing used	Concentration of dye	Fastness to rubbing		Wash fastness			Fastness to Perspiration					
							Alkaline			Acidic		
		Dry	Wet	Alt	SC	SW	Alt	SC	SW	Alt	SC	SW
Commercial Fix	1%	4	3	3	4	3	3	3	3	4	3-4	3-4
	2%	4	3	3	4	3-4	3	3	3	4	3-4	3-4
	3%	3	2-3	2-3	4-5	3-4	3	2-3	2-3	4	3-4	3-4
Fix.1	1%	4	3-4	3-4	4-5	3	3-4	3	3	4-5	4	4
	2%	4	3	3-4	4-5	4	3-4	3	3	4-5	4	4
	3%	3-4	2-3	3	5	4	3-4	3	3	4-5	4	4
Fix.2	1%	4	3-4	3-4	4-5	3	3-4	3	3	4-5	4	4
	2%	4	3	3-4	4-5	4	3-4	3	3	4-5	4	4
	3%	3-4	2-3	3	5	4	3-4	3	3	4-5	4	4
Fix.3	1%	4	3	3-4	4-5	4	3-4	3	3	4-5	4	4
	2%	4	3	3-4	4-5	4	3-4	3	3	4-5	4	4
	3%	4	3	3-4	4-5	4	3-4	3	3	4-5	4	4

Alt: color change of dyed sample; SC: staining on cotton; SW: staining on wool, Wash-scale (1-5).

TABLE 5. Fastness properties of dyed cotton fabrics using direct blue, and fixed after dyeing using 10g/L fixing agent.

Fixing used	Concentration of dye	Fastness to rubbing		Wash fastness			Fastness to Perspiration					
							Alkaline			Acidic		
		Dry	Wet	Alt	SC	SW	Alt	SC	SW	Alt	SC	SW
Commercial Fix	1%	4	3-4	3-4	4-5	4	4-5	3-4	3-4	3-4	4	4
	2%	3-4	3	3	4-5	4	4-5	3-4	3-4	3-4	4	4
	3%	4	3	3	4-5	4	4	3-4	3-4	3-4	4	4
Fix.1	1%	4	3-4	3-4	5	4-5	4-5	4	4	4	4-5	4-5
	2%	4	3	3	4-5	4	4-5	4	4	4	4-5	4-5
	3%	4	3-4	3-4	5	4-5	4-5	4	4	4	4-5	4-5
Fix.2	1%	4-5	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	2%	4-5	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	3%	4-5	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
Fix.3	1%	4-5	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	2%	4-5	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5
	3%	4-5	4	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5	4-5

Alt: color change of dyed sample; SC: staining on cotton; SW: staining on wool, Wash-scale (1-5).

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