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# Cationic dye fixing agents on cotton fabric

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Negative
(-1)

Positive
(+1)

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## Abstract

Reactive dye is a dye that can react with fibre to form a covalent bond which forms a permanent attachment in the fibre and cannot be removed by repeated washings under neutral conditions.

Consequently, the dye becomes part of the fibre leading to outstanding colour fastness to washing.

Although reactive dyes are widely used for the exhaustion dyeing of cotton and other cellulosic fibres, these dyes suffer from the disadvantage that the dye-fibre reaction is not 100% efficient. The dyes react not only with the fibre nucleophile but also with nucleophiles present in the dyebath leading to dye hydrolysis. It therefore becomes necessary to use dye fixing agents to improve the fastness properties of the dyed fabrics.

This article reports on synthesis, characterisation and the application of dye fixing agents on cotton fabric. It discusses the two chemistries of dye fixing agents: one based on polyamine and the other based on diallyl dimethyl ammonium chloride (DADMAC).

Polyamine-based dye fixing agent was synthesised by a condensation reaction of diethylene triamine and Dicyaddiamide in the presence of diethylene glycol. PolyDADMAC was synthesised by the free radical initiated addition polymerisation of diallyl dimethyl ammonium chloride (DADMAC). The structural elucidation of synthesised polyamine and PolyDADMAC fixing agents was performed by Fourier transform infrared (FTIR) spectrophotometry. The structural change of dye-fixed fabric was investigated by FTIR in comparison with the untreated cotton-dyed fabric. Spectrophotometric analysis was used to determine the amount of dye in the dye bath after dyeing. The rate of exhaustion, rate of fixation and efficiency of dye-fixing agents were calculated from the absorbance values obtained after dyeing, washing and dye fixation respectively. The absorbance values were noted at the wavelength of maximum absorption ($\lambda max$, 540 nm) after adequate dilution (1:50). The dye-fixing experiments were performed on reactive dye, direct dye and indigo-dyed denim fabric. The dye fixing efficiency was evaluated by subjecting the dye-fixed fabrics for washing fastness study as per the standard ISO test methods in comparison with the control sample. The impact of dye fixing agents on the colour coordinates of the dyed fabrics were also studied by computer colour matching system (CCM) and reported in the article.

Keywords: dye-fixing agent; polyamine; PolyDADMAC; FTIR; spectrophotometric analysis

## Introduction

Cotton and other cellulosic substrates comprise more than 40% of world textile consumption [1], which is mainly made up of cellulose (Figure 1) [21]. Several classes of dyes can be used to dye cellulosic fibres, viz., vat, direct, reactive, sulphur colourants, however characteristically, reactive dyes are most in use with more than 50% of world consumption [22]. Reactive dyes are very popular in the textile industry for their excellent fastness properties and diversity of shades [13]. Reactive dyes are desirable because of their excellent wash fastness, which arises from covalent bond formation between dye and fibre, through either nucleophilic substitution or by addition reaction – and sometimes by both mechanisms for a dye with two or more reactive groups [6].

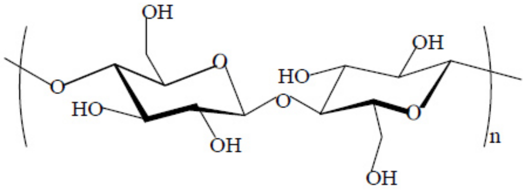


Figure 1: Chemical structure of cotton fabric (cellulose)

Reactive dyeing essentially takes place in two stages. In the first stage, reactive dyeing is carried out in presence of electrolyte where the reactive dye is adsorbed onto the cellulosic substrate through hydrogen bonding and Vander Waals forces. Electrolyte is added to overcome the long-range repulsion forces operating between the anionic dye molecule and the negative ζ-potential acquired by cotton surface in aqueous medium.

In the second stage, sodium carbonate is used as an alkali to maintain the pH level between 10.5 and 11.5 for the formation of cellulosate ions (Cell-O$^-$) within the substrate for covalent bond formation between dye and fibre, allowing fixation to take place [1 However, hydrolysis of the dye occurs simultaneously at this pH [2]. Alkaline application conditions in reactive dyeing result not only in covalent dye-fibre fixation but also an alkali-induced hydrolysis of the reactive dye [7]. A reactive dye that can react with cellulosate ion can certainly react with hydroxyl ion of water. In a reactive dyeing process, therefore, two competing reactions (Equation-1 and 2) proceed simultaneously [2].

$$\text{Dye-X} + \text{H}^{+}\ {}^{-}\text{O-Cell} \rightarrow \text{Dye-O-Cell} + \text{HX} \quad (1)$$

$$\text{Dye-X} + \text{H}^{+}\ {}^{-}\text{OH} \rightarrow \text{Dye-OH} + \text{HX} \quad (2)$$

(X = any reactive group)

The first reaction is the desired one leading to dye fixation on cotton material, while the second is a side reaction leading to the formation of an inactive hydroxyl derivative of the dye. The hydrolysed dye does not react with cotton but is held only substantively like direct dyes, leading to poor wash fastness [2]. Since the hydrolysed dye is not covalently bonded, it keeps getting removed during washing treatments causing poor wash fastness. Accordingly, these reactive dyeings require a multistep wash-off process involving various aqueous rinses and washings in order for the dyeing to achieve the characteristics of very high wash fastness. To achieve maximum wash fastness of a reactive dyeing, the hydrolysed dye should be completely removed, as otherwise it would lower the wash fastness of the dyeing because of its lower substantivity to cellulose. Therefore, it is necessary to remove the hydrolysed dye by soaping treatment at the boil and then fixing the hydrolysed dye molecules with a dye fixing agent to prevent further colour bleeding during subsequent washing and to improve various colour fastness properties [16].

There have been several attempts to improve the fastness properties of dyeing by treating the dyed fabric with metal salts or with formaldehyde, impregnation with resins and after-treatment with cationic auxiliaries. The use of cationic after-treatment auxiliaries has proved particularly effective [17]. The cationic after-treatment auxiliaries or the cationic dye fixing agents for cotton fabrics are based on various chemistries viz., polyamine, polycationic, dicyano resin and reactive resin types [21]. Also, in recent years, formaldehyde-free fixing agents have been developed to replace those containing formaldehyde. In this article, synthesis, characterisation and application of Polyamine dye fixing agent and Polycationic dye fixing agents are discussed in detail. These are formaldehyde free, cationic dye fixing agents.

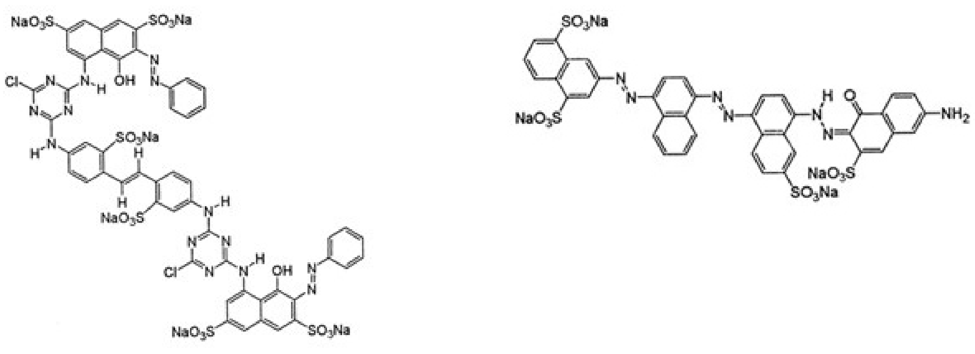
Polyamine dye fixing agent is the earliest-known dye-fixing agent and was synthesised by polycondensation of diethylenetriamine and dicyandiamide. It has been reported that the good colour fastness properties of polyamine dye fixing agent could be attributed to the presence of cationic and reactive groups. The cationic group could interact with anionic dyes and reactive group can be crosslinked with cellulose. Many cationic polymers have been applied to cotton fabrics with a view to enhance the uptake of anionic dyes and the mechanism of interactions involved can be interpreted by the participation of electrostatic forces between the dyes and the basic cationic groups in the polymer [21]. The poly dially dimethyl ammonium chloride (PolyDADMAC) dye-fixing agent would be one of the most optimum polycationic fixing agents, which is the polymer of monomer dially dimethyl ammonium chloride (DADMAC) [19], composed of five-membered pyrrolidone rings, six-membered piperdine rings and little branched structures. Moreover, Cellulose and dimethyldiallylammonium chloride have similar conformational structures, and this would be expected to contribute to the strong interactions [5], thus, PolyDADMAC can be widely applied in the fixing of different dyes on cotton fabric.

## Experimental

***Materials and methods***

Industrially bleached 100% woven cotton fabric was purchased from Eland Fashion India Pvt. Ltd. Indigo-dyed denim fabric was purchased from Jagadamba Textile, Bangalore, India. Multi-fibre strip-Type DW complying with ISO 105-F10 was purchased from James H Heal, UK. Glauber salt was supplied by Malti Enterprises, Tarapur, India. Soda ash was supplied by Remichem Industries, Bhiwandi, India. Acetic acid was supplied by Sarex Overseas, Tarapur, India. UK. Rota dyer machine from *R. B. Electronic* & Engineering Pvt. Ltd., Gyrowash from James H Heal, computer colour matching system and UV visible spectrophotometer (Spectra Scan 5100H) of Premier Colour scan Instruments Pvt. Ltd., Perspirometer from MAG Solvics Pvt. Ltd. and α-FTIR from Bruker were used for testing.

Regarding the commercial reactive dyes, C.I. Reactive Red 152 was supplied by Colortex Pvt. Ltd. (Mumbai, India) and C.I. Direct Blue 71 was supplied by Atul Ltd. The structure of these dyestuffs is given in Figure 2 below.



C.I. Reactive Red 152 C.I. Direct Blue 71

Figure 2: Molecular structure of the dyestuffs used in the experiment

***Synthesis of formaldehyde-free polyamine dye-fixing agent - Fixanol (Conc)***

Polyamine dye fixing agent was synthesised by a condensation reaction of diethylene triamine and dicyandiamide in the presence of diethylene glycol. This condensation reaction produces a complex polymer with 80% solid content, as shown in Figure 3. The synthesised product is named Fixanol (Conc).

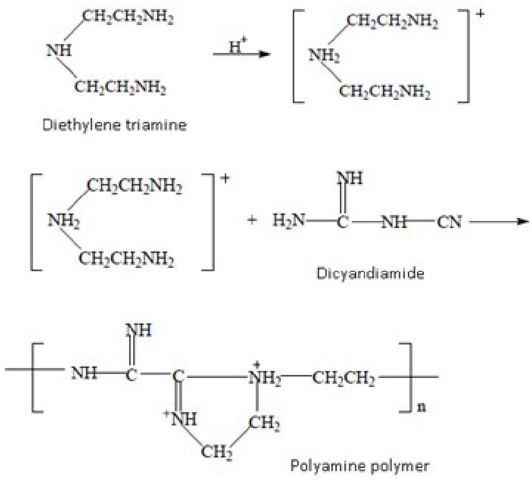



Figure 3. Synthesis of polyamine fixing agent (Yu et al., 2009)

***Synthesis of formaldehyde-free poly diallyl dimethyl ammonium chloride (PolyDADMAC) dye-fixing agent - Katafix-BC and Ferafix-PDM***

PolyDADMAC was synthesised by the free radical initiated addition polymerisation of diallyl dimethyl ammonium chloride (DADMAC), as shown in Figure 4. The synthesised polyDADMAC, liquid concentrate, was named as Katafix-BC. Ferafix-PDM, the powder form, was obtained by spray-drying liquid concentrate.

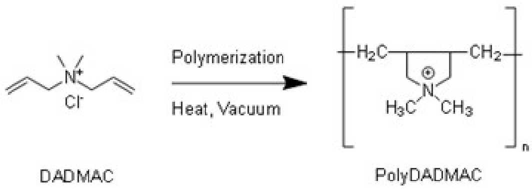



Figure 4. Synthesis of polyDADMAC [15]

***Characterization studies – Fourier Transform Infrared spectroscopy (FTIR)***

The structural elucidation of synthesised dye fixing agents was performed using a α-FTIR (Fourier transform infrared) spectrophotometer (Bruker-Platinum ATR). FTIR spectroscopy was also carried out for the untreated cotton fabric and the cotton fabric treated with dye fixing agents in order to determine the interactions of functional groups of dye fixing agents with the dyed fabric. The α-FTIR was set with 24 scans with 4 $cm^{-1}$ resolution between 600 and 40,00 $cm^{-1}$ and the ATR Platinum Diamond 1 Refl #45E2D3A2D as the accessory.

***Exhaust dyeing:***

100% cotton fabric was dyed with (a) 6% C.I. Reactive Red 152 and (b) 4% C.I. Direct Blue 71 by exhaust application. For reactive dyeing, the dye bath was prepared with 6% C.I. Reactive Red 152 dye on the basis of weight of cotton fabric, keeping the MLR of 1:10. The machine was initially run at room temperature for five minutes and then, after adding 60gpl Glauber salt, the temperature of the dye bath was slowly raised to 80$^{o}$C. With a gradient of 1.5$^{o}$C, the temperature of the dye bath was slowly raised to 80$^{o}$C and maintained at that level for 30 minutes. Dyeing was further continued for 30 minutes, with the addition of 20 gpl soda ash. After the completion of dyeing, the dyebath was drained and the fabric was neutralised with 0.5gpl Acetic acid at 50$^{o}$C for 10 minutes, followed by a soaping treatment at 90$^{o}$C, for 15 minutes. The dyed fabric was then rinsed, dried and taken for dye fixing application. Similarly, for direct dyeing, the dye bath was prepared at room temperature with 4% C.I Direct Blue 71 dye on the basis of the weight of cotton fabric, 20gl Glauber salt and 5gpl soda ash keeping the

MLR of 1:10. Dyeing was started at room temperature and, slowly, the dyeing temperature was raised to 95$^o$C, keeping 1.5$^o$C minimum as the temperature gradient. Dyeing was continued for 60 minutes. After the completion of dyeing, the dyebath was drained and the fabric was rinsed thoroughly with cold water, dried and taken for dye fixing application.

***Dye fixing treatment on dyed fabrics***

As shown in Figure 5, the cotton fabric was dyed with anionic dyes (reactive dye and direct dye) and then treated with the synthesised dye fixing agents at 40$^o$C for 20 minutes, followed by drying. The dosages of dye fixing agents taken are given in Table 1 below.

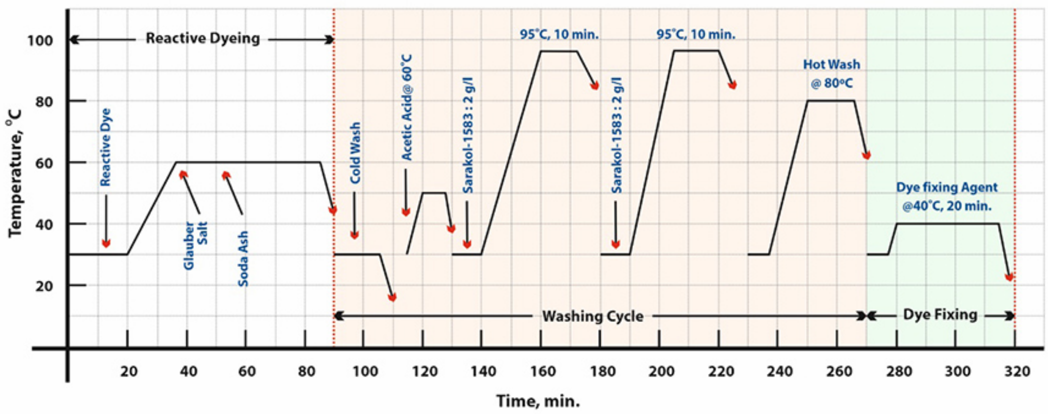



Figure 5. Process flow-dyeing and dye-fixing process

| | Dosages taken for the study | | |
|---|---|---|---|
| Synthesised dye fixing agents | For C.I. Reactive Red 152 | For C.I. Direct Blue 71 | For Indigo denim |
| Fixanol Conc | 0.25% | 1.5% | 1% |
| Katafix-BC | 0.5% | 3% | 2% |
| Ferafix-PDM | 0.2% | 1-2% | 1-1.5% |

Table 1. Dye fixing agents and their dosages

***Determination of dye exhaustion and fixation***

The proportion of the amount of dye adsorbed by the fabric after dyeing to the primary amount of dyes, is called as the exhaustion rate (%E) of the dyes. Spectrophotometric analysis was used to determine the amount of dye in the dye bath. The absorbance of the dye solution before dyeing, after dyeing and washing liquor was determined by Ultraviolet-Visible (UV-Vis) spectrophotometry at the wavelength of maximum absorption ($\lambda max$, 540 nm) after adequate dilution (1:50). The concentration of the dye was determined according to the previously set dye calibration curve. The dye exhaustion rate (% E) was determined according to Equation 3, where Ao is the absorbance of the original dye solution and At is the absorbance of the residual dye solution after dyeing [10], [18], [9].

$$\%E = \frac{Ao - At}{Ao} \times 100 \quad (3)$$

Dye fixation rate (% F) refers to the number of dyes chemically bonded to the fabric, which can be obtained by calculating the number of fixed and unfixed dyes bonded with the fabric and washed out by the soaping process. The rate of fixation was determined after soaping. The dye fixation rate (%F) was determined after soaping and multiple washings until no dye was removed from the dyed fabric. The washing solutions were collected and the absorbance of the soaping liquor was measured at the maximum wavelength of the dye ($\lambda max$, 540 nm) and from the calibration curve. The dye fixation rate (% F) was determined according to Equation 4, where As is the absorbance of the bath after soaping [10], [18], [9].

$$\%F = \frac{Ao - At - As}{Ao} \times 100 \quad (4)$$

The efficiency of dye fixing agent (%D) was determined in a similar way. The absorbance of the dye fixing drain bath was measured at the maximum wavelength of the dye ($\lambda max$, 540 nm). The efficiency of dye fixing agent (%D) was determined according to Equation 5, where Ax is the absorbance of the dye bath after dye fixing treatment.

$$\%D = \frac{Ao - At - As - Ax}{Ao} \times 100 \quad (5)$$

***Measurement of colourant strength (K/S)***

The estimation of the colourant strength of the dye fixed fabrics was carried out by determining the K/S values using a computer colour matching system (Spectra Scan 5100H, Premier Colour Scan), reflectance spectrophotometer, according to the CIE Lab colour difference concept at standard illuminant D65. This gives reflectance values at wavelengths of 400-700nm. According to the reflectance readings, the colour strength (K/S) value of the dyed samples was determined by the application of the Kubelka-Munk equation, Equation 6, [10] where K is the absorption coefficient and S is the scattering coefficient for a colourant at a specific wavelength, R is the fractional reflectance value of the dye on the substrate at complete opacity [14].

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \quad (6)$$

K/S values expresses the colour yield in linear proportionality to the amount of dye present on the dyed fabric. The K/S value was calculated and directly generated by a spectrophotometer. Each sample was measured four times, and the measurement points were changed randomly [10]. The colour value and tone obtained after dye fixing were evaluated in terms of CIE Lab colour space by using a computer colour matching system. The 'L' axis runs from top to bottom. L=100 represents white, whereas L=0 represents black. The 'a' and 'b' values have no specific numerical limits. Positive a indicates red, whereas negative a indicates green. Positive b indicates yellow, whereas negative b indicates blue. The total colour difference 'dE' is a single value, which takes into account the differences between the L a b of the sample and standard. The value of 'C' indicates chroma coordinate, ie the perpendicular distance from the lightness axis, whereas 'H' indicates the hue angle expressed in degrees, with 0° being a location on the +a axis, then continuing to 90° for the +b axis, 180° for –a, 270° for –b and back to 360° = 0° [3]. As a whole, a combination of these figures enables one to understand the tonal variations.

***Measurement of colour fastness properties***

To study the efficiency of dye fixing agents, the dye fixed fabrics were subjected to a colour fastness study. Colour fastness is one of the important indicators for measuring product quality in the perspective of consumer demand. The washing fastness identifies the loss and change of colour during washing, as well as the staining of colour on other clothes. In this study, the standard test method ISO-105-C10-C3 was used for measuring the washing fastness properties of the dye fixed samples. The recipe consisted of 5gpl standard soap solution and 2gpl sodium carbonate solution. The washing fastness was carried out in Gyrowash (James H Heal) and the machine was run at 60 ℃ for 30 minutes, keeping a 50:1 liquor ratio. Colour fastness to water was carried out as per ISO 105 E01. This test is designed to measure the resistance of coloured textiles to water.

## Results and discussions

***Characterisation of the chemical structure and evaluation of the synthesised dye fixing agents***

An FTIR study was conducted on the synthesised dye fixing agents. Figure 6 shows the FTIR spectrum of polyamine fixing agent. The peak at 3179cm$^{-1}$ indicates the presence of amine group (-NH). The peak at 2190cm$^{-1}$ indicates the presence of C≡N bond. The peak at 1561cm$^{-1}$ could be assigned to NH bend. The peak at 1294cm$^{-1}$ is for C-N stretch. The peak at 777cm$^{-1}$ indicates the N-H wag corresponding to primary/secondary amines. Based on the above analysis, the functional groups are in good agreement with the chemical compound based on polyamine.

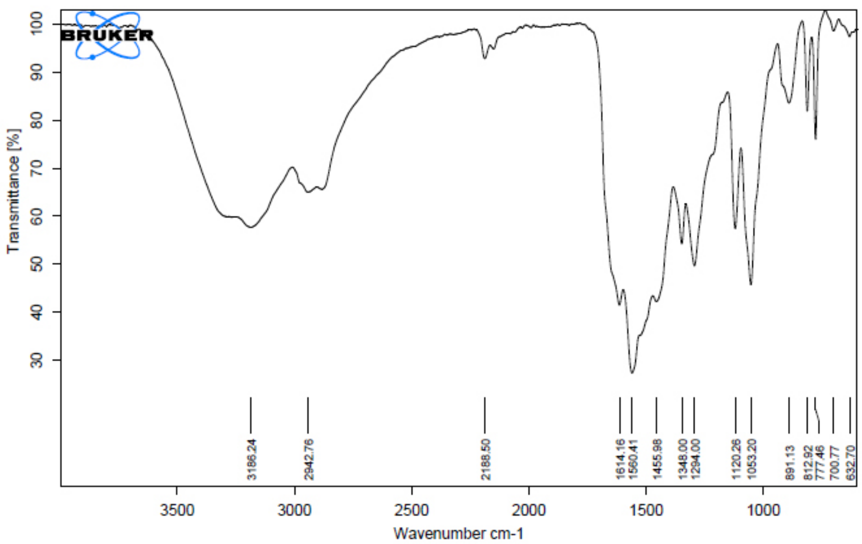



Figure 6. FTIR spectrum of polyamine fixing agent, fixanol (Conc)

Figure 7 shows the FTIR spectrum of PolyDADMAC dye-fixing agent. The broad peak at 3403cm$^{-1}$ indicates the presence of amide group (-NH). The peak at 1634cm$^{-1}$ indicates amide-I (C=O) and sharp peak at 1476cm$^{-1}$ indicates amide-II (C-N stretch). The peaks at 2867cm$^{-1}$, 2944cm$^{-1}$ and sharp peak at 1476cm$^{-1}$ could be assigned to methyl and methylene (-$CH_3$ and -$CH_2$) stretching and quaternary ammonium group -$N^+(CH_3)_2(CH_2)_2$ bending vibration of pyrrolidinium rings from DADMAC unit respectively. The peaks in-between 900-990cm$^{-1}$ indicate vinyl terminals ($-CH=CH_2$) in DADMAC [20], [11]. Based on the above analysis, the functional groups are in good agreement with the chemical compound of PolyDADMAC.

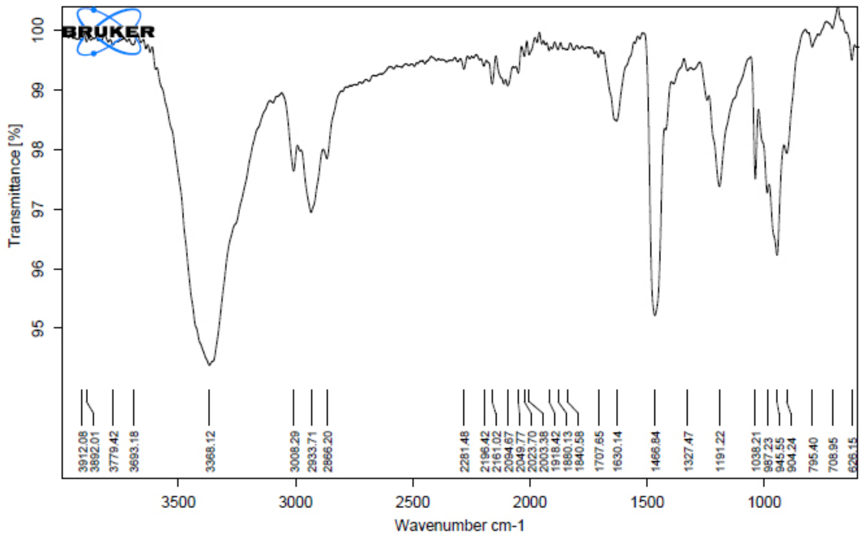



Figure 7. FTIR spectrum of PolyDADMAC fixing agent, Katafix-BC

***Mechanism of dye fixing agent***

Under the recommended conditions, the dye fixing agent has a property to form a covalent bond with the hydrolysed dye molecule, forming a complex. Because of complex formation, the dye molecule loses its substantivity, which in turn improves the washing fastness of the dye-fixed fabric. Due to the loss of substantivity, the dye molecule loses its ability to stain the adjacent fabric. For a polyamine fixing agent, it is reported that the good colour fastness properties could be attributed to presence of cationic and reactive groups. The cationic group could interact with anionic dyes and the reactive group can be cross-linked with cellulose. For PolyDADMAC, the mechanism of interactions involved can be interpreted by the participation of electrostatic forces between the dyes and the basic cationic groups in the polymer [21]. Moreover, cellulose and diallyl dimethyl ammonium chloride have similar conformational structures, and this would be expected to contribute to the strong interactions [5]. The mechanism of polyamine and the PolyDADMAC dye-fixing agent is shown in Figures 8 and 9 respectively [21].

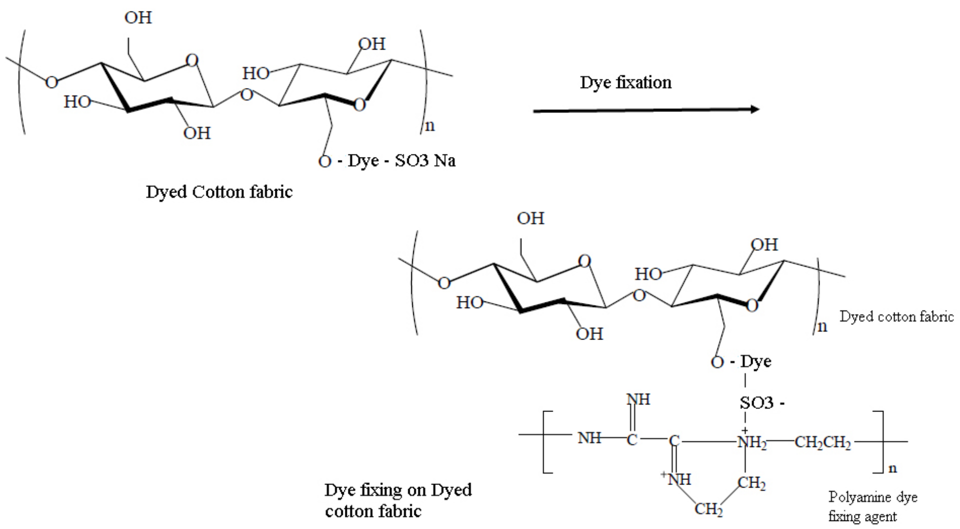
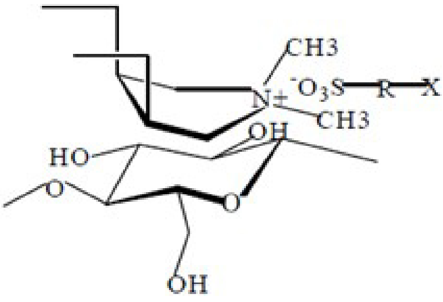



Figure 8. Dye-fixing mechanism of polyamine dye-fixing agent on dyed fabric




R = dye

Figure 9. Dye fixing mechanism of PolyDADMAC on dyed fabric

***Interaction of dye-fixing agents on dyed cotton fabric***

The FTIR spectra of untreated and treated dye-fixed cotton fabric are given in Figures 10 and 11. The spectrum of the dye-fixed cotton fabric is markedly different from the untreated fabric in terms of peak intensity. In both the spectrums, the peaks of dye-fixed fabrics are bigger and sharper, which indicates the effective reaction of dye-fixing agent with dyed cotton fabric.

The FTIR spectra of untreated (A) and treated (B) cotton fabric (dye fixed with Fixanol (Conc) were illustrated in Figure 10. The broad absorption band within 3,273.93cm$^{-1}$ and 1,028.35cm$^{-1}$ for the untreated cotton fabric revealed the presence of numerous hydroxyl groups. However, the (primary-OH) band of the dye-fixed cotton fabric becomes narrow with a higher intensity within 3,276cm$^{-1}$ and 1,028.46cm$^{-1}$, indicating the increased nitrogen content of the dye-fixed fabric. These distinct, narrow, high-intensity bands confirm the presence of quaternary ammonium group (Kamel et al., 2009). Compared with untreated cotton fabric, dye-fixed fabric showed narrow hydrogen bonded OH stretching peak at 3,276cm$^{-1}$ and aliphatic C-H stretching at 2,894.73cm$^{-1}$ [4]. A small peak at 2,102cm$^{-1}$ on the dye-fixed fabric could be attributed to the presence of C≡N bond, which confirms the reaction of Fixanol (Conc) with the dyed cotton fabric. Also, a smaller peak with high intensity, around 1,628.60cm$^{-1}$, is usually related to the OH bending vibrations of adsorbed water, however, it can be also assigned to N-H asymmetric deformation [8]. Sharp peak at 1,202.89cm$^{-1}$ could be assigned to the C-N stretching vibration [4]. Sharp peak at 1,426.82cm$^{-1}$ could be assigned to the bending vibration of C-N. Characteristic peaks which are observed at 2,102cm$^{-1}$, 1,628.60cm$^{-1}$,1,426.82cm$^{-1}$ and 1,202.89cm$^{-1}$ clearly indicates the presence of $–CH_2-N+R_3$ quaternary ammonium group. It fully demonstrated that the quaternary ammonium group was chemically adsorbed on the dye-fixed cotton fabric and that the reaction between cotton fabric and Fixanol (Conc) had occurred [9].

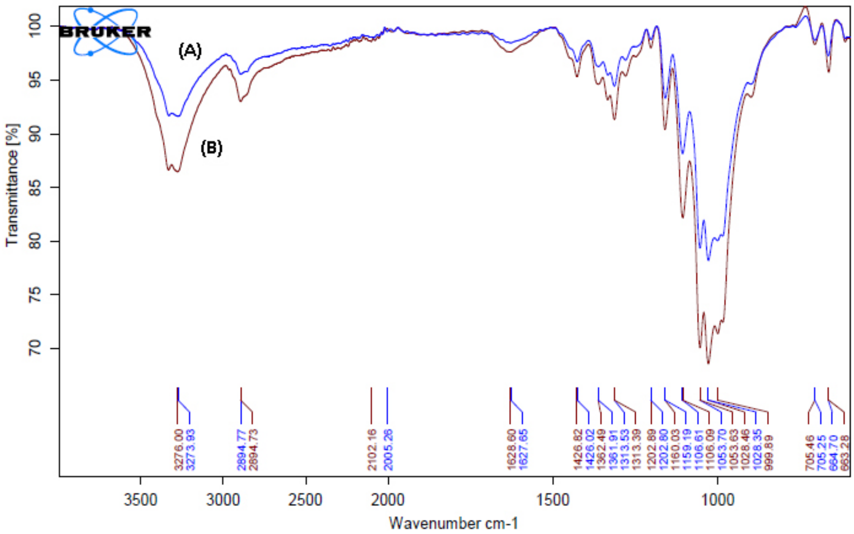



Figure 10. FTIR spectra of (A) untreated cotton fabric and (B) treated with 0.25% Fixanol (Conc)

The FTIR spectra of untreated cotton (A) and treated (B) cotton fabric (dye fixed with 0.5% Katafix-BC) are illustrated in Figure 11**.** In the FTIR spectrum of the dye-fixed cotton fabric, the peak at 1,639.77 $cm^{-1}$ could be assigned to N-H asymmetric deformation [8]. A sharp peak at 1,426.08$cm^{-1}$ could be assigned to the bending vibration of C-N. This observation clearly indicated the presence of –$CH_2$-N+$R_3$ type nitrogen (quaternary ammonium group) bending vibration of pyrrolidinium rings from the DADMAC unit. It fully demonstrated that the quaternary ammonium group was chemically adsorbed on the dye-fixed cotton fabric and that the reaction between the cotton fabric and Katafix-BC had occurred [9]. Moreover, the absorption intensity of dye-fixed fabric at 3,275.42$cm^{-1}$, 1,639.77$cm^{-1}$, 1,426.08$cm^{-1}$ and 1,053.46$cm^{-1}$ was higher than the untreated cotton fabric. This is because the PolyDADMAC also have similar absorptions at those shifts, which would increase the intensity of similar absorptions. These observations have further suggested that the PolyDADMAC have penetrated into the cotton fabric, forming strong reaction linkages with the dyed cotton fabric.

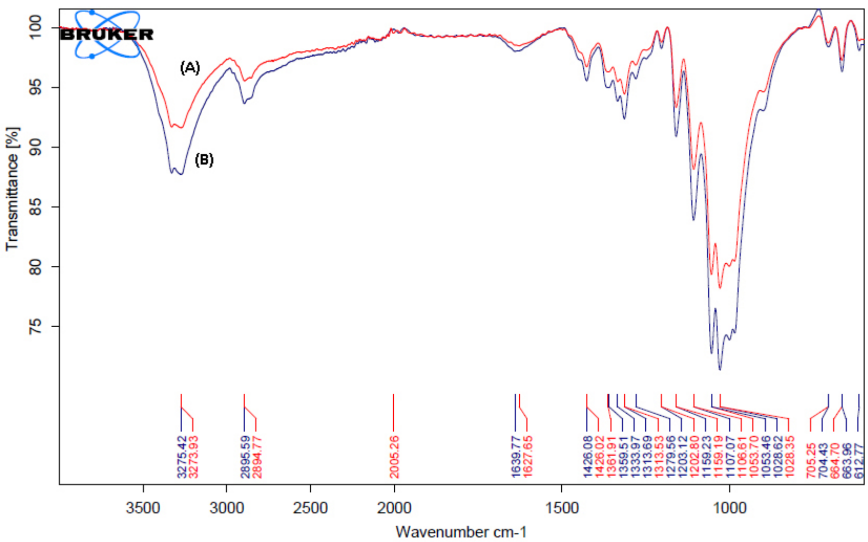



Figure 11. FTIR spectra of (A) untreated cotton fabric and (B) treated with 0.5% Katafix-BC

### *Dye exhaustion and fixation*

The dye uptake of the fabric was determined spectrophotometrically by detecting the amount of dye remaining in the dyebath after the dyeing had been completed. Concentration of the dye was determined according to its calibration curve (Beer-Lambert law) in water since a linear relationship is usually found between absorbance and concentration. The dye exhaustion (%E), dye fixation (%F) and efficiency of the dye-fixing agent (%D) were calculated from Equations 3, 4 and 5 respectively, and summarised in Table 2. The rate of exhaustion of C.I. Reactive Red 152 on cotton fabric was found to 98.51%, whereas the rate of fixation was found to be 94.16%. The efficiency of all the dye-fixing agents (%D) was found to be 94.14-94.16%. It is observed that %D and %F values are nearly the same, which indicates that there is no further bleeding of hydrolysed dye molecule after dye fixing treatment, proving the efficiency of the synthesised dye fixing agents.

| **Dyeing of 100% cotton fabric with 6% C.I. Reactive Red 152** | | | | |
|---|---|---|---|---|
| Rate of exhaustion and fixation | | Efficiency of dye fixing agent (%D) | | |
| | | 0.25% Fixanol(Conc) | 0.5% Katafix-BC | 0.2% Ferafix-PDM |
| Rate of exhaustion (%E) | 98.51% | 94.16% | 94.14% | 94.16% |
| Rate of fixation (%F) | 94.16% | | | |

Table 2. Rate of exhaustion and fixation and efficiency of dye-fixing agent

***Fastness properties of the dye-fixed fabric***

Table 3 summarises the colour fastness results of the dye-fixed fabrics. Reactive dyed fabric dye fixed with Fixanol (Conc), Katafix-BC and Ferafix-PDM shows 1-1.5 unit improvement on the grey scale, implying very good improvement in washing fastness property. Performance of Fixanol (Conc) on -irect dyed and indigo-dyed denim fabric shows 0.5 unit improvement in the washing fastness, whereas Katafix-BC and Ferafix-PDM shows 1.5-2 unit improvement in the fastness rating. A similar trend was observed in the water fastness study. From the observations made, it could be summarised that the polyamine fixing agent, Fixanol (Conc), is highly effective on reactive dyes, whereas PolyDADMAC fixing agents, Katafix-BC and Ferafix-PDM, are equally effective on reactive and direct dyestuffs and indigo-dyed denims.

| | **Colour fastness to washing** ISO 105 C10 | | | **Colour fastness to water** ISO 105 E01 | | |
|---|---|---|---|---|---|---|
| | Staining of cotton component in multifibre strip | | | | | |
| **Samples** | C.I. Reactive Red 152 | C.I. Direct Blue 71 | Indigo dyed Denim | C.I. Reactive Red 152 | C.I. Direct Blue 71 | Indigo dyed Denim |
| Blank | 3 | 1-2 | 4 | 3 | 1-2 | 4 |
| Fixanol (Conc) | 4 | 2-3 | 4 | 4 | 2 | 4 |
| Katafix-BC | 4-5+ | 3-4+ | 4-5 | 4-5+ | 4 | 4-5 |
| Ferafix-PDM | 4-5 | 4 | 4 | 4-5 | 4 | 4 |

Table 3. Colour fastness properties of dye-fixed cotton fabric

***Colourant strength and total colour difference***

Table 4 shows the L a b values of the fabrics dyed with C.I. Reactive Red 152 and dye fixed with various dye fixing agent, while Figure 9 shows the respective colour difference and colour coordinates. From Table 4 Fixanol (Conc) slightly supresses the redder and yellower component of fabric dyed with C.I. Reactive Red 152 and hence the dye-fixed fabric appears redder with a slightly more bluish tone than the unfixed fabric. Katafix-BC and Ferafix-PDM do not have much impact on the L a b values and hence the final fabric does not show any notable shade changes. Fixanol (Conc) in Figure 12a, shows a slight tonal shift in the bluer region, whereas in Figure 12b and 12c, no tonal shift is observed with Katafix-BC and Ferafix-PDM respectively.

| Dye fixing agents | Colour coordinates | | | | | | K/S | % Colourant strength |
|---|---|---|---|---|---|---|---|---|
| | L | a | b | C | H | dE | | |
| Blank | 40.914 | 55.665 | - 3.520 | 55.776 | 356.383 | - | 92.899 | 100 |
| 0.25% Fixanol (Conc) | 40.657 | 55.131 | - 4.364 | 55.303 | 355.476 | 1.031 | 92.573 | 100 |
| 0.5% Katafix-BC | 10.591 | 55.493 | - 3.771 | 55.621 | 356.114 | 0.444 | 94.768 | 102 |
| 0.2% Ferafix-PDM | 40.874 | 55.864 | - 3.809 | 55.994 | 356.101 | 0.353 | 93.651 | 100 |

Table 4. Colour strength values of dye-ixed fabrics, 6% C.I. Reactive Red 152

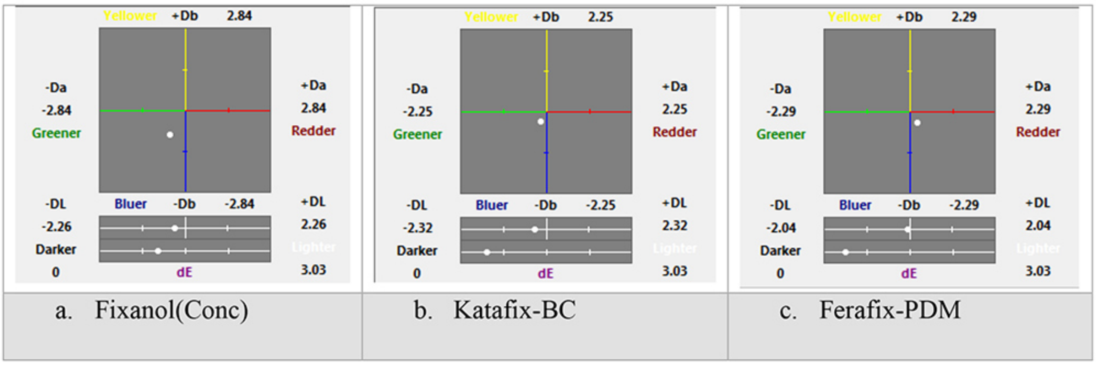



Figure 12. Pictorial representation: colour coordinates of dye fixed fabrics - C.I. Reactive Red 152

## Conclusion

In recent years, more and more formaldehyde-free fixing agents have been developed to replace those fixatives containing formaldehyde. Our synthesised dye fixing agents, Fixanol (Conc), Katafix-BC and Ferafix-PDM have developed to shorten manufacturing process times. FTIR analysis of Fixanol Conc and Katafix-BC confirmed the expected structure of polyamine and PolyDADMAC, respectively. The structural modification of dye-fixed fabric, as interpreted by FTIR spectrums, also confirmed the effective interaction of Polyamine and PolyDADMAC dye-fixing agent with cotton dyed fabric. The rate of dye exhaustion, the rate of fixation and the efficiency of dye-fixing agents have also proven the interaction of dye-fixing agents with the dyed fabric. Besides, the colour fastness properties of the dye-fixed fabric were also satisfactory. The polyamine dye-fixing agent has shown minimal shade changes on the dye-fixed fabric. This property can be used in the operations where shade matching is required at the final stage. The PolyDADMAC fixing agents did not show any notable shade changes on the fabric. Thus, it can be concluded that the synthesised dye fixing agents exhibited very good dye fixing properties, hence meeting the customers' demand.