



MODIFICATION OF COTTON FABRIC FOR INCREASED DYEABILITY WITH REACTIVE DYES: A REVIEW

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ABSTRACT

Reactive dyes hold a position of great importance in the textile colouration industry. Their use for dyeing of cotton has been very well established. As opposed to their advantages such as bright colours and very good wash fastness, their drawbacks of low dye utilisation and high electrolyte requirement have posed serious concerns to environmentalists. Over years various efforts have been directed at developing more eco-friendly processes in dyeing cotton with reactive dyes. The various approaches include, modifying dye structure to increase dye fibre interactions, dyeing process alterations to decrease electrolyte and alkali requirements and also modifying the cotton fibre to make it more receptive to the dye. This review presents an overview of researches in the field of cotton substrate modification for enhancing dyeability with reactive dyes.

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INTRODUCTION

The importance of reactive dyes for cotton fabric has been well established. Some estimates point out that cotton fulfils an enormous 50% of the world's fibre consumption and of this a very substantial volume is being coloured with reactive dyes. The reason for their commercial popularity is obvious given their brilliant shades, good tinctorial value, good fastness properties, ease of application and an acceptable price. In fact, these properties have led to reactive dyes engulfing approximately 30% of the textile dye market share.

With growing popularity of reactive dyes for dyeing of cotton, environmental problems associated with their use have received attention. However, the application of these dyes, causes high and undesirable levels of dissolved solids and oxygen demand in the effluent. This is due to the use of considerable quantities of inorganic salt and alkali to ensure efficient utilisation and fixation of the reactive dyes (Khatri *et al.*, 2015). Their low substantivity towards cellulose leading to poor exhaustion, makes it mandatory to use large quantities of electrolyte for its exhaust application. This high a dosage of an electrolyte such as sodium sulphate or sodium chloride (up to 100 g/l) and an alkali (up to 20 g/l) leads to serious environmental problems (Agarwal, B.J., 2014). High electrolyte concentrations in discharged dye baths result in

increased salinity in natural water bodies and adversely affect its flora and fauna. Sodium sulphate even has detrimental effects on the concrete pipe lines and also leads to increased acidity of water ways (Lewis *et al.*, 1997). Together with this, exhaustion of the dye bath liquor is only 65-70% which thus leads to wastage of dye: and a large amount being passed in the effluent. Unfixed reactive dye and the hydrolysed dye along with alkali used for fixation are environmentally hazardous and increase the pollution load. Certain reactive dyes, like mono- and di-chlorotriazine, or fluorotriazine type of reactive dyes may cause the passage of organo-halogen in the discharge effluent. The removal of this unfixed dye from the substrate requires extensive use of energy and resources. Different approaches to alleviate these problems have been attempted by dyers and researchers. These various approaches can be grouped under the following three heads (Chattopadhyay, D.P., 2001):

1. Modification of the dye structure to make it more substantive to cellulose. (Possibly adding a positive charge on the dye)
2. Cationization of cellulose through chemical reaction with compounds containing cationic groups to improve dye-fibre affinity.
3. Dyeing process modification by controlling salt and alkali dosing.

The present review will discuss briefly the modification of the cotton fibre substrate in order to increase reactive dyeing efficiency and hence reduce the environmental burden caused by excess use of electrolyte, alkali and the presence of hydrolysed / unfixed colourant in the effluent.

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Primarily all modification of cotton is by the means of cationization, which refers to the introduction of cationic character to the fibre or in other words, introduction of a +ve charge that would nullify the negative zeta potential developed on the fibre surface. The surface of textile fibres in neutral water solution is negatively charged due to dissociation of functional groups and adsorption of ions from the solution, inducing formation of electrical double layer in aqueous solutions of electrolytes. Dissociated carboxyl groups formed by oxidation of aldehyde groups on terminal glucoside units also contribute to the negative charge of the cotton surface apart from dissociation of functional hydroxyl group. This negative surface charge of textile fibres, known as zeta potential, hampers anionic dye adsorption on the fibre surface. (Ristic *et al.*, 2012)

The cationic modifying agents hence work towards strengthening the fibre's attraction to anionic dyes. In the absence of this intervention, dyers are compelled to overcome the -ve zeta potential on the fibre by using large amounts of electrolytes.

Introducing a cationic monomer/ Epoxy compound / Amino group

Primarily the focus was on introduction of amino groups into the cellulose structure to increase its substantivity towards anionic dyes. Glycidyl trimethylammonium chloride, popularly known as Glytac A was one of the earliest commercially popular cationic modifying agent. Its variant 2,3-epoxypropyl-trimethylammonium chloride has also been popularly used.

Glytac A is a popular epoxy compound for modification of cellulose with pad batch method. Modified fabric shows various advantages, such as electrolyte free dyeing, excellent colour fastness and colour strength as reported by Hauser and Tappa (2001). Rupin, M. (1976), Dvorsky and Cerovsky and Rippon have also studied the dyeability of cellulose substrates modified with Glytac A and its precursor 1-chloro-2-hydroxy-3-trimethylammonium propane. The modified substrate showed ease of neutral dyeing, with no salt and double colour yields as compared with those achieved by the conventional salt-alkali method on untreated cotton (Lewis *et al.*, 1997). An Epichlorohydrin precursor of glytac A, structurally named as 3-chloro-2-hydroxypropyl trimethyl ammonium chloride (CHPTAC) has also been reportedly used for assisting cotton to form ionic bonds with negatively charged reactive dye, by rendering cotton a cationic charge. Many more researchers have worked with CHPTAC for process improvement in the modification of cotton. Single bath pad - batch process containing CHPTAC and alkali, two bath pad-bake process and simultaneous mercerization and cationization with CHPTAC have been used for its application and proved successful in enhancing depth of shade on cotton reactive dyeing without salt addition leading to an environment friendly process. Recently, Nallathambi and Rengaswami (2016) focused on exhaust method to apply CHPTAC on knit cotton hosiery fabric for industrial application.

Acharya, S, *et al.* (2014) carried out studies on the effect of the level of cationization of cotton fabric i.e.. the concentration of CHPTAC on the % exhaustion kinetics of the dye. In this study, cotton fabric was cationized using five different concentrations of CHPTAC, and the real-time exhaustion of two reactive dyes on cationized fabrics dyed without salt was

monitored with a focus to assess the effect of the cationization level on the exhaustion of reactive dyes on cationised cotton fabrics. The authors concluded that the optimum concentration of CHPTAC for cationization depends upon the dye. Cationization is a viable option for reducing the quantity of effluent discharged by dyeing operations and also minimising its environmental impact. Cationization is effective in lowering the quantity of water, energy and chemicals required for dyeing cotton fabrics.

Lewis *et al.* (1992) proposed a water-soluble modifying agent, N, N-dimethylazetidinium chloride (DMAC), which could be applied through a pad-dry-bake procedure. This resulted in a substrate that is readily dyeable under pH 7 in the absence of electrolytes. The colour obtained had normal wash and light fastness. Lewis and Lei showed that the both, Glytac A and DMAAC pre-treatments were equally effective in enhancing dyeing with all types of reactive dyes. Wang *et al.* (2002) reported a fibre reactive compound, AAHTAPC (Amino alkyl hydroxy trimethyl propane chloride). The cotton fabric was recommended to be padded in a solution of 50g/l AAHTAPC and 30g/l NaOH and baked at 125°C for 6 min. The modified cotton on dyeing showed exhaustion, fixation and color yield higher than that of untreated ones in absence of salt. But a great disadvantage of dyeing of AAHTAPC modified cotton was the tendency of ring dyeing as dye molecules rushed into specific quaternary sites.

The problem of unevenness of dyeing was resolved with the use of Betaine (N, N, N-trimethyl glycine) (Ma, *et al.*, 2005). This is a quaternary ammonium compound. Cotton fabrics were immersed into a solution containing betaine, hydrochloric acid and dicyandiamide with 90% pick up and dried at 80°C for 3 min, baked at 150°C for 40 seconds. Such a fabric subsequently dyed showed colour strength, fastness properties of a satisfactory level. Nevertheless, there was a reduction in tensile strength of the cationised cotton as the treatment was carried out under acidic pH at elevated temperatures.

Pad-dry-cure processes with amino compounds such as EDTA, DMEA lead to the formation of ester linkage between carboxyl group of EDTA and hydroxyl groups of cellulose. The treated fabric is dyed at a pH of 4 for promoting exhaustion due to the protonated amino groups. This is followed by dyeing at a pH of 11 for fixation. Satisfactory dyeing properties led this process to for commercialisation (Das *et al.*, 2016).

Modification with cationic amino polymers

The advantages of using a polymer for modification over a monomer is that though synthetic monomers can penetrate well into a substrate, they are less substantive, hence a larger concentration is required, whereas such a problem can be avoided with the use of synthetic polymers.

Hercosett 125, a product of Hercules Powder Corporation, is a reactive polymer which can introduce a cationic charge to cotton. Thus, it is a Polyamide Epichlorohydrin (PAE) type of polymer. Burkinshaw *et al.* (2000) used Polyepichlorohydrin resin to modify cotton so that salt and alkali free reactive dyeing could be achieved. The treatment was done by pad - dry process followed by dyeing in exhaust method. The researchers reported that the dye uptake was higher than untreated one. The dyeing was optimum at pH 7 thus opening up the possibility of alkali free dyeing. Colourfastness measurements revealed excellent wash fastness and fair light

fastness. One of major merits mentioned here was absence of ring dyeing due to migration of dye to the yarn surface. Lei *et al.* (1990) studied the effect of pretreating cotton with a PAE resin in combination with Ethylene diamine (EDA). It has been reported that the use of EDA improved the efficiency of treatment and improved the reactivity of the substrate towards anionic dyes. The treatment resulted in substrates that are dyeable with reactive dyes under weakly acidic conditions without the need to add an electrolyte. Dyeings produced were bright, level and of appreciable wash fastness.

Another study conducted by Wu *et al.* (1993) reported that pretreatment of cotton with poly epichlorohydrin with dimethylamine (DMA) produces a modified cotton which could be dyed under neutral conditions with highly reactive dyes in the absence of salt or even with low reactivity reactive dyes using small amounts of the recommended electrolyte.

Blackburn *et al.* (2003) introduced Poly (4-vinyl pyridine) quaternary ammonium compound for cationisation of cotton. They suggested that the obvious advantage was that both the pretreatment and dyeing could be carried out through exhaust techniques. The polymer was of a higher molecular weight and thus had better substantivity for the cotton substrate. The dyeing could be done in the absence of salt or alkali and hence resulted in lesser dye hydrolysis and wastage. Hence it provided a much needed eco-friendly process.

Han *et al.* (2008) introduced cationic di-block copolymer, a quaternary ammonium compound synthesised from [2-methacryloyloxy ethyl] trimethylammonium chloride to produce a cationized fibre. A patent was filed in the United States by Collins *et al.* in 1999. A method for dyeing cellulosic materials with reactive dye by pretreating the fabric with a polycationic, polynucleophilic polymeric agent was disclosed. The agent was a polyquaternary amine material especially a poly(DADMAC) or polyvinylpyridine, particularly one including nucleophilic sites such as primary amino groups, e.g. polyallylamine/DADMAC copolymers and polyvinylpyridines quaternised with w-aminoalkyl groups. The dyes react with the nucleophilic centres so dye bath exhaustion is facilitated by dye fixation rather than substantivity to the substrate. There is no need to use salts to drive substantivity and post dyeing washing to remove unreacted/hydrolysed dye is much simpler. Material dyed by the methods was reported to have good wash fastness properties.

PVAmHCl (Poly Vinylamine chloride) was explored by Ma *et al.* (2005). The chemical was applied via a 2-dip, 2-nip padding method and subsequently baked at 100^o C for 10 minutes. The optimum concentration of PVAmHCl was reported to be 10 g/l. Exhaustion, fixation, colour fastness to wash and rubbing of exhaust dyed pretreated cotton was excellent. Periyasamy *et al.* (2011) confirmed the increased dye fixation on pretreatment with PVAmHCl. The fastness properties of dyed Lyocell/cotton fabrics pretreated with PVAmHCl when compared with those of conventional dyeing were found to be better. The above mentioned cationic polymers, which were primarily quaternary ammonium salts were successful as substrate modifiers, but the presence of primary and secondary amines altered the hue of dyes and also adversely affected the wet fastness properties due to strong electrostatic attraction between fibre and hydrolysed dye. To counteract these disadvantages, Xiaoxu *et al.* (2010) used

tertiary amine cationic polyacrylamide (TACPAM). Results obtained showed that higher molecular mass resulted in higher exhaustion, but lower fixation as higher molecular mass of modifying agent affected the action between treated cotton and dye and ability of dye to penetrate.

Lewis *et al.* (1992) undertook various studies to improve the substantivity of cotton for no-salt dyeing. They attempted modification of cotton with N- methylolacrylamide (NMA) and also 2,4-dichloro-6-(2-pyridino ethylamino)-s-triazine (DCPEAT). They have obtained success with both reagents. The major drawback with NMA is that it releases hazardous formaldehyde during curing as well as on storage. With DCPEAT it was noted that the amount of fixed dye decreased with an increase in the concentration of the modifying reagent. This reagent also granted a notable antimicrobial property. One of the more recent cationic polymer to be used for modification of cotton is the polyamino carboxylic acid (PACA's). The PACA's contain two different functional groups, i.e. carboxyl groups and amine groups. Ameri Dehabadi *et al.* (2013) in a study on pretreatment of cotton fabrics with polyamino carboxylic acids for salt free dyeing of cotton with reactive dyes have summarised that the presence of NH₂ groups in PACA's improve the exhaustion of the reactive dyes. The K/S values have been measured and indicate that PACA's reduce the electrolyte requirement for dyeing with reactive dyes. They also found that the fastness properties were also not affected by this pretreatment when compared to reactive dyed untreated cotton fabric. In a study by Agrawal, B.J. (2014), attempt has been made to modify cotton, in order to perform reactive dyeing at neutral pH and without the addition of salt or alkali. They pretreated cotton with a highly reactive polymer viz. poly acrylic acid and cross-linked it with either epichlorohydrin (C1) or hexamethylene tetramine-hydroquinone (C11) cross-linking agents, by a pad-dry-cure technique. It has been reported that such cross-linked cotton fabric can successfully be dyed with various types of reactive dyes. The colour strength of all the dyed samples was adequate and quite comparable with conventionally dyed samples. It is also said that cross linking with hexamethylene tetramine-hydroquinone (C11) gave better dyeing results as compared to those samples where the poly acrylic acid was cross linked with epichlorohydrin (C1). Through infrared studies the researchers conclude that this is because hexamethylene tetramine-hydroquinone (C11) is a nitrogenous cross linking agent and here the N—H stretching is responsible for a better dyeing performance. The fastness properties of such dyeings is also reported to be very good.

Modification using Chitosan

Most of the amines discussed above are highly toxic in nature and cause irritation to eyes, skin and respiratory organs and other medical conditions like headache, dizziness, shortness of breathing, nausea and vomiting. Thus, the above means of cationization of cotton need to be modified to make them free from eco-hazards. Chitosan, a polymer derived from chitin, is seen as a possible substitute for various hazardous chemicals used for the cationization of cotton. Chitosan is a linear polysaccharide which consists of β (1, 4) linked D-glucosamine and N-acetyl-D-glucosamine which are allotted randomly in its structure. Chitosan is a biocompatible and the safest modifying agent that has been used in pretreatment of cotton to make it suitable for salt free dyeing with reactive dyes. Chitosan forms crosslinks with the cotton fibre and

provides the amino groups for its cationisation, rendering it a positive charge. Chitosan treatment has proved to increase both the substantivity and reactivity of fibres towards reactive dyes under neutral conditions. Chitosan offers not only positively charged amine Group but also positive charged hydroxyl groups (Aktek and Millat, 2017). Houshyar *et al.* (2002), undertook the treatment of cotton with chitosan to render it dyeable with reactive dyes under more environmentally friendly conditions. They experimented with various application processes and concluded that pad dry method ensured the highest dye uptake with slight reduction of light and wash fastness.

Various chitosan derivatives have also been explored. A water-soluble, fibre reactive chitosan derivative, NMA-HTCC, i.e., O-acrylamidomethyl-N- [(2-hydroxy-3 trimethylammonium) propyl] chitosan chloride was studied by Lim *et al.* (2004). Much later in the year 2014, Bhuiyan *et al.* focused on exhaust dyeing of cotton knit fabric and compared colour strength of chitosan treated fabric with an increase in concentration of chitosan and compared with untreated sample. It was found that as the chitosan concentration increased the colour depth also increased. The colour fastness properties except wet rub fastness were also on the upside in the treated cotton fabrics. The wet rub fastness showed a slight decline. Studies have also attempted grafting cotton with amino terminated chitosan dendrimer hybrid named chitosan-poly (propylene imine) (CS-PPI). The results of colour uptake have been satisfactory (Sadeghi-Kiakhan *et al.*, 2015). A novel technique by Samanta *et al.* (2015), undertook a process where a mixture of chitosan and PEG was used to treat cotton muslin fabric. It is established that at 2% chitosan and 2% poly ethylene glycol concentration, the obtained colour strength was higher than the untreated conventionally dyed fabric. Bandyopadhyay *et al.* (1998) reported treatment of cotton fabric with a solution of chitosan in 2% aqueous acetic acid by pad-dry technique. It was seen that this pretreatment could cut down the amount of salt required by about 50% in the dyeing with vinyl sulphone type reactive dyes. An increase of 15-50% in dye fixation on chitosan-treated fabric was found in case of cold brand reactive dyes, as compared with untreated dyed fabric. Singha *et al.* (2013) worked on application of chitosan on cellulosic. They have consulted that when the fabric is treated with chitosan, the primary hydroxyl groups of cellulose is partly modified into amide groups, which then causes the cellulose to act like wool fibre and thus reactive dyes can be dyed on cotton at neutral PH in the absence of electrolyte and alkali.

Other methods of modification

Dendrimers have also been used to carry out cationic modification of cotton to enable environment friendly, salt free dyeing of cotton with reactive dyes. Dendric polymers are highly branched, spherical three dimensional structure, containing a large number of functional end groups which have the ability to impart special characteristics such as solubility, substantivity to a dye and surface activity. Various studies have focused on the application of such dendrimers on cotton. Zhang *et al.* (2007), synthesised a water-soluble amino-terminated hyperbranched polymer from methyl acrylate and diethylene triamine by melt polycondensation. The cotton fabric was treated with an aqueous solution of this polymer and treated cotton was subsequently dyed with reactive dyes. The treated and dyed cotton fabric displayed a markedly enhanced colour strength, even when dyeing had been carried

out in the absence of the electrolyte. The washing fastness, rubbing fastness and levelling properties of the dyed, treated cotton fabrics were also better compared to that obtained by conventional dyeing on the unmodified fabric. Burkinshaw *et al.* (2000) have suggested that cotton pretreated with a dendrimer displays markedly enhanced colour strength with reactive dyes, even when dyeing is carried out in the absence of both electrolyte and alkali. The researchers also remarked that on competitive dyeing between untreated and dendrimer treated cotton, the treated cotton shows a substantially higher colour uptake and hence this offers a possibility of patterning through differential dyeing creating effects such as cross dyed fabrics etc.

Grafting of cellulose with cationic agent has opened up a new dimension to establish zero salt reactive dyeing. Graft polymerization was employed to convert low molecular weight cationic monomer to high molecular weight cationic polymer that could be fixed covalently with cellulose. Srikulkit and Larpsuriyakul (2002) performed graft polymerization of cellulose with MAPTAC (methacryloylamino propyl trimethylammonium chloride). The grafting was done simultaneously with hydrogen peroxide bleaching. After dyeing the modified cotton, results obtained showed that colour strength and fixation showed increased with increasing concentration of MAPTAC. This was reported to be because of more cationic groups on cotton hence more of the available anionic dyes were attracted. Later, Zhang *et al.* (2007) generated an amino-terminated hyper branched polymer grafted cotton fibre (HGCF) to avoid salt addition during reactive dyeing. Here also the grafted cotton showed appreciable colour strength and colour fastness when dyed in the absence of salt.

Liu and Yao (2011) studied salt-free dyeing cotton fabric by grafting thiourea to cotton fibres. Thiourea grafted cotton fabric (TUGCF) was prepared by epoxidizing cotton fibres with epichlorohydrin in water and subsequently grafting in aqueous solution of thiourea. Grafted fabric was subsequently dyed with reactive dyes in the absence of salt. The results displayed significantly enhanced colour strength, showing better dyeing properties when compared to the untreated cotton fabric dyed with conventional methods, in terms of washing fastness, rubbing fastness, and levelness.

Summary

Cotton has an un paralleled position amongst the textile fibres. It offers a unique range of properties like superior comfort, softness, strength, hygroscopicity and many others. Of all synthetic dyes, reactive dyes are most preferred for dyeing of cotton as they offer bright shades, are chemically bonded to the fibre which is being dyed offering superior colour fastness and are also inexpensive to apply. Reactive dyes, in spite of being a dyers dream, pose problems in application as anionic in nature, their exhaustion onto cotton is challenging. The exhaustion is brought about by the use of large quantities of an electrolyte to suppress the negative charge on the fibre and overcome the electrostatic repulsion between the anionic dye and the fibre. These dyes also give rise to heavily coloured effluents due to their low exhaustion. Such drawbacks of reactive dyes are major concerns for environmentalists. A large number of efforts have been made at improving exhaustion of reactive dyes and reducing the quantity of electrolyte needed. Majority of researches have focused on

modification of cotton by cationising its surface so as to suppress the negative charge and improve exhaustion even in the absence of salt. All efforts in this direction have been successful in providing a substrate which is suitable for a more environmental friendly, low or no salt dyeing. Chitosan has been one of the most popular agents in this regard due to it being a natural, biodegradable agent.

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