

Review

Past, present and future prospects of cotton cross-linking: New insight into nano particles

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ABSTRACT

The desire for cotton fabrics having a reduced tendency to crease has been recognized for considerable time during the recorded history. Cross-linking of cellulose has been introduced as a necessary process for various purposes especially creating a cotton fabric with anti-wrinkling properties. Generally, in a cross-linked cotton fabric, cellulose chains are bridged across through chemical reaction with certain compounds. Cellulose cross-linking traced back more than 80 years and is still being actively researched. Over the past few years, there has been an enormous attention on coapplication of nano and biotechnology with crosslinking agents. Various approaches have been developed to cross-link cellulose along with imparting other properties. In this paper, literatures on cotton cross-linking were reviewed from early time to now. The formaldehyde and non-formaldehyde based cross-linking agents, applications of enzyme and nanotechnology, new approaches and future aspects of cotton cross-linking have been thoroughly discussed.

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1. Introduction

Cross-linking of cellulose is an important textile chemical process which can be defined as the stabilization of cellulosic fibers through chemical reaction with certain compounds in such a way that the cellulose chains are bridged across.

Cellulose crosslinkers can be divided into two types: those that self-polymerize as well as crosslink cellulose and those that crosslink cellulose, known as cellulose reactants. In the first theory three-dimensional polymers are formed while in the second theory, the molecules of cross-linking agents can form covalent bonds by reacting with the cellulose hydroxyl groups.

Since it has been believed that chain slippage under moist conditions is responsible for wrinkling, cross-linking adjacent cellulose chains could be logically a way of improving crease recovery (Bajaj, 2002; Ibrahim, Bayazeed, Refai, & Hebeish, 1986; Mark, Wooding, & Atlas, 1971; Tomasino, 1992; Yang, 1991a, 1991b, 2001; Wei & Yang, 1999).

Besides durable press finishes, cross-linking agents have been used in antimicrobial, flame retardant and water repellent finishes. In recent years they are also being applied along with bio and nano treatments.

In the present review, we have tried to put together an overview of all the cross-linking agents have been discovered for cotton, their effects, weaknesses and strengths.

2. Past prospect

The first major steps in overcoming the inherent drawbacks of natural fibers made them easy to launder and substantially crease resistant in wear, were made in 1928 with the advent of the crease-resisting process for cellulosic fabrics. This advance came about with the application of resin finishing agents to cellulosic fabrics. The way in which chemical finishing agents impart crease resistance was somewhat debatable, although it was generally accepted that it involved the formation of crosslinks between adjacent cellulose molecules or microfibrils (Mark et al., 1971).

The idea of introducing resins inside the cotton fibers to achieve crease resistant cotton showed that phenol/formaldehyde or urea/formaldehyde could be reacted together and produce the small molecules that could penetrate into the fibers. If these were applied to fabric, a subsequent backing of the impregnated fabric would cause the small molecules to condense into macromolecules inside the fibers. Urea-formaldehyde suffered from the disadvantage that treated fabrics absorbed chlorine during hypochlorite bleaching in laundering, resulting in severe tendering of the fabric (Bajaj, 2002).

In order to overcome the chlorine-retention problem, methylolmelamines were introduced to the textile industry. Since the chloramines of melamine were yellow in color, white fabrics finished with this resin, though resistant to rendering by chlorine, became yellow during bleaching. Secondly, use of trimethylolmelamine depravities imparted the relatively stiff hand to the fabric (Bajaj, 2002; Mark et al., 1971; Tomasino, 1992).

The earliest attempt to produce a cross-linking reactant that did not contain free NH-groups and would not absorb chlorine was the production of tetramethylolacetylenediurea. Attention was then turned to dimethylol ethyleneurea. As this compound had only two methylol groups and no free NH-groups, it could not form a thermosetting resin and did not absorb chlorine and therefore it was very effective in producing crease-resistance properties. The most serious disadvantage of this product was that it caused a marked deterioration in the light fastness of many direct or reactive dyes (Bajaj, 2002; Mark et al., 1971; Tomasino, 1992).

The next approach was the introduction of the dimethylol ether of dimethyloluron. As with DMEU, multiple laundering and

hypochlorite bleaching results in a decrease in resistance chlorine damage. However the decay is slower than obtained with DMEU.

Triazone resins were the next products but because of the trouble of fish odors encountered with the use of them, attempts were made to produce other systems that would crosslink cellulose, and resulted in the production of Dimethyloldihydroxyethyleneurea (DMDHEU) (Mark et al., 1971; Tomasino, 1992).

Although dimethyloldihydroxyethyleneurea was the most widely reactant for the production of durable press garments, its use resulted in fabrics that were chlorine retentive and also prone to some degree of yellowing.

Next investigations led to the use of dimethylol carbamates. The methyl and ethyl carbamates were carcinogenic and no longer used but the propyl and hydroxyethyl were safe (Bajaj, 2002; Mark et al., 1971; Tomasino, 1992).

To conclude, important features of the first cotton cross-linking agents are summarized in Table 1

3. Present prospect

N-methylol agents have long been used by the textile industry as durable press finishes producing wrinkle-resistant cotton fabrics (Huang, 2000; Shin & Huang, 2002).

However, because they release formaldehyde either from treated fabrics or during finishing processes, their use in textile industries is limited. Formaldehyde identified to have impact on human health and the environment. For this purpose, many efforts have been done to reduce the formaldehyde released from finished fabrics. These efforts range from after-washing the finished fabric to developing non-formaldehyde reactants. Some of the methods of reducing formaldehyde release are summarized in Fig. 1.

Many classes of chemical compounds with lower formaldehyde content have been extensively investigated such as α -hydroxyalkylamides, polymers from urea and glutaraldehyde, and diamidodihydroxyethane products. Several formaldehyde-free cross-linking systems have been developed including the commercially available addition product of 1,3-dimethylurea and glyoxal (4,5-dihydroxy-1,3-dimethylimidazolidinone, DHDMI), systems based on glyoxal with co-reactive additives and several polycarboxylic acids (Andrews, Welch, & Trask-Morrell, 1999; Welch, 1988, 1994, 2001a, 2001b, 1990; Yang & Wang, 1996a, 1996b, 1997, 1998).

Most researchers have focused on dialdehydes as cross-linking agents for cotton cellulose (Frick & Harper, 1982a, 1982b, 1983). Glyoxal as a durable press finishing agent, with Lewis acids such as magnesium chloride and aluminum chloride as the catalysts was included in the studies (Welch & Danna, 1982; Yang, Wei, & Mcilwaine, 2000d). In 2000, Yang and his coworkers studied the performance of glutaraldehyde in cross-linking cellulose through its two aldehyde groups (Yang et al., 2000d).

Gonzales and a coworker used magnesium chloride as a catalyst for glyoxal (Gonzales & Guthrie, 1969), and Matsui and his coworker applied a process in which treated fabrics were bleached with hydrogen peroxide after cross-linking was carried out with zinc borofluoride as a catalyst (Hosokawa, 1973). Worth et al., treated cotton fabrics and cotton-polyester fabrics with glyoxal and reactive silicone with catalyst mixtures of aluminum sulfate and magnesium sulfate (Worth, 1981; Worth & Freeman, 1981). Welch reported that the aluminum salt was best for glyoxal among the catalysts used for N-methylol compounds for crease recovery finishes (Welch, 1983). In 2005, Lee and a coworker treated cotton fabrics with a glyoxal/glycol mixture with aluminum as a catalyst (Lee & Kim, 2005). They reported that use of sodium hydrogen sulfate as an additive to the glyoxal/glycol, improved the whiteness and strength

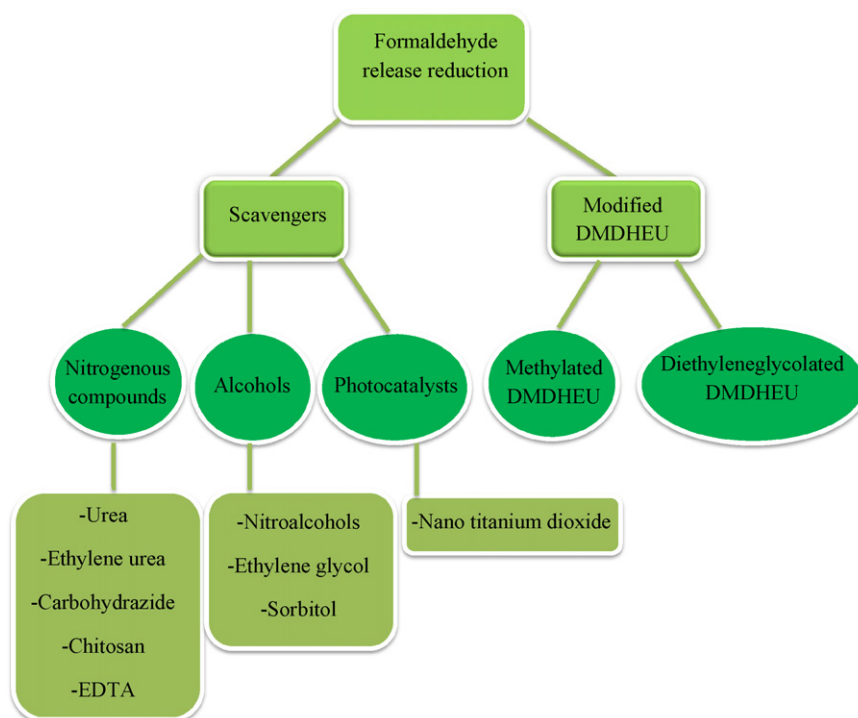
Table 1

Important features of the first cotton cross-linking agents (Mark et al., 1971; Tomasino, 1992; Bajaj, 2002).

Anti-wrinkling agent	Advantages	Disadvantages
Phenol/formaldehyde	-Good anti-crease	-Stiff fabric hand -Bad odor -Poor chlorine resistance -Acid degradation of treated fabrics -High formaldehyde release -Stiff fabric hand
Urea/formaldehyde	-Easy curing -Excellent anti-crease	-Bad odor -Poor laundry durability -Poor hydrolysis resistance -Negative effect on light fastness of direct and reactive dyes -Poor chlorine resistance -Acid degradation of treated fabrics -High formaldehyde release
Melamine/formaldehyde	-Higher laundry durability -Synergistic effect with phosphorus flame retardants	-Fabric yellowing caused by chlorine bleaches -Stiff fabric hand (especially in tri-products) -High formaldehyde release
Tetramethylolacetylenediurea	-Good chlorine resistance	-Fabric yellowing
DMEU Dimethylolethyleneurea	-Easy curing -Good anti-crease -Lower formaldehyde release -Lower stiffness	-Poor hydrolysis resistance -Poor chlorine resistance -Poor laundry durability -Negative effect on light fastness of direct and reactive dyes
Triazones	-Excellent chlorine resistance with metal salts -Excellent anti-crease	-Fish odor
DMDHEU Dimethyldihydroxyethyleneurea	-Lower free formaldehyde -Possibility of post curing -Acceptable laundering durability -Acceptable hydrolysis resistance -Acceptable chlorine resistance	-Negative effect on light fastness of direct and reactive dyes (less than DMEU)
Carbamate	-Extreme stability to alkaline hydrolysis -Good laundry durability -Good chlorine resistance	-Harsh curing condition (stronger catalyst/higher curing temperature) -Great strength and abrasion loss -High formaldehyde release

retention of the treated fabrics but reduced the WRA. The presence of polyurethane in a glyoxal/glycol padding bath increased both the WRA and tearing strength retention but reduced the whiteness of treated fabrics (Lee & Kim, 2005).

Among the new agents being developed, poly carboxylic acids are the most promising non-formaldehyde durable press finishes (Schramm & Rinderer, 2000; Trask-Morrell & Andrews, 1991; Yang, 1993a, 1993b; Yang & Gu, 1999; Yang & Wang, 1996a, 1996b,

**Fig. 1.** Methods of reducing formaldehyde release.

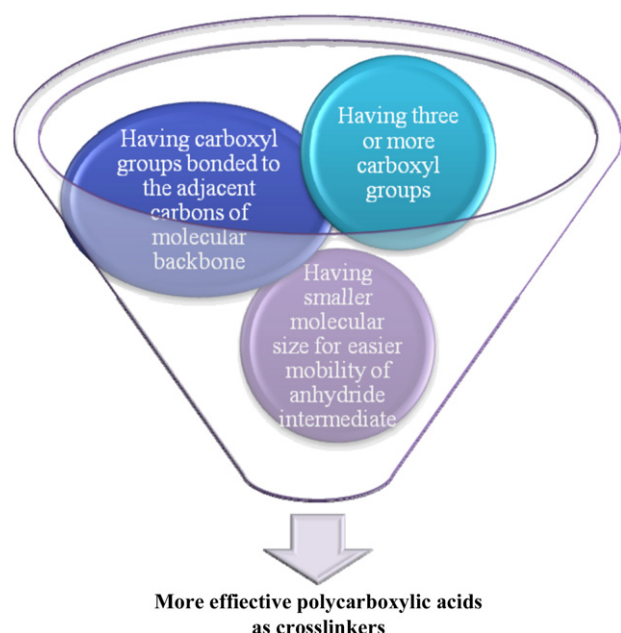
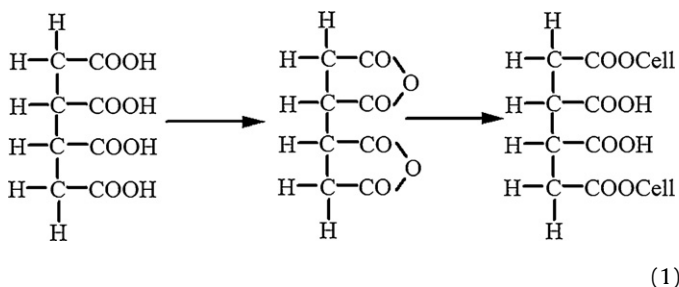


Fig. 2. Properties of effective polycarboxylic acids as cellulose cross-linking agent.

1998; Yang, Wang, & Lu, 2000c). Research on the esterification cross-linking process and its use to impart durable smooth-drying properties to cellulosic fabrics has been reviewed by Welch (1992, 1994) and Lämmermann (1992) (Shet & Yabani, 1981). In 1988, Welch reported that tetracarboxylic acids are able to impart high levels of wrinkle resistance to cotton fabrics (Welch, 1988).

Cellulose esterification by polycarboxylic acids proceeded in two steps: formation of a cyclic anhydride intermediate by the dehydration of two carboxyl groups, and the reaction between cellulose and the anhydride intermediate to form an ester. It has been found that those carboxylic acids that form cyclic anhydride intermediates can esterify cotton cellulose (Yang, 1993a, 1993b; Yang & Gu, 1999; Yang et al., 2000c). Polycarboxylic acids with three or more carboxyl groups bonded to the adjacent carbons of their molecular backbone are effective cross-linking agents for cellulose (Yang & Wang, 1998). Reaction (1) shows the formation of an initial cyclic anhydride, its reaction to form a covalent bond to cotton cellulose, followed by formation of a second anhydride, which subsequently reacts with another cellulosic hydroxyl to complete a crosslink. BTCA has been chosen as an example.



In 1997 (Yang & Wang, 1997), Yang and his coworkers reported that there is a clear link between the molecular weight of a polycarboxylic acid and its effectiveness for cross-linking cotton cellulose. They found that esterification of cotton cellulose by a polycarboxylic acid requires not only the formation of a five-membered cyclic anhydride intermediate, but also the mobility of the anhydride intermediate to access the cellulosic hydroxyl so that the esterification can take place, because both the acid and cotton cellulose are in solid states. According to their work, the reactivity

of a polycarboxylic acid for esterifying cellulose is affected by the size of its molecule. Properties of effective polycarboxylic acids as cross-linking agents are concluded in Fig. 2 (Yang & Wang, 1998; Yang, Wang, & Kang, 1997).

Some of the most commonly used polycarboxylic acids are reviewed briefly.

3.1. Citric acid (CA)

Citric acid (CA), a tri-functional carboxylic acid known as a cost-effective and environmentally friendly cotton cross-linking agent but it was not satisfactory with its performance due to its tendency to discolor the treated fabrics (Andrews, 1990; Fouda & Fahmy, 2011; Schramm & Rinderer, 1999a, 1999b; Welch & Peters, 1999; Yang & Lu, 1999; Yang & Xu, 1998).

Lu et al. (Lu & Yang, 1999) investigated the chemical changes taking place on the CA-treated fabric under elevated temperatures. They evaluated the relation between curing temperature/time, finish concentration and fabric yellowing. They found that yellowing increases with an increase in those mentioned parameters. Their study also indicated that sodium hypophosphite as a catalyst results in less fabric yellowing than monosodium phosphate. They found that the yellowing effect of CA can be attributed to the dehydration of CA and formation of unsaturated carboxylic acids (cis-aconitic acid (CAA) and trans-aconitic acid (TAA)) on the fabric.

Ibrahim et al. studied the use of CA as an ester cross-linking agent for cotton fabrics along with different additives including tri-ethanolamine hydrochloride (TEA-HCl) as a nitrogenous additive, Poly ethyleneglycole-600 (PEG-600), various catalysts and some silicone softeners (Ibrahim, Abo-Shosha, Elnagdy, & Gaffar, 2002). They found that incorporation of PEG-600 results in improving tear strength (TS) and whiteness index (WI) along with reduction in wrinkle recovery angle (WRA) and soil stain release rating (SRR). TEA-HCl in the finishing formulation increases the WRA and WI along with a little reduction in TS and SRR. Among the esterifying catalysts used, they reported the following order of effectiveness: $\text{NaH}_2\text{PO}_4 \cdot \text{H}_2\text{O} > \text{K}_2\text{HPO}_4 > \text{Na}_3\text{-citrate} > \text{Na}_2\text{-tartrate}$.

Inclusion of silicone softener results in an improvement in softening degree, WRA, TS along with a decrease in SRR and WI values.

Yang and Xu (1998) evaluated the effectiveness of poly maleic acid (PMA)/CA and ter-polymer maleic acid (TPMA)/CA as crease-resistance agent for cotton. They revealed that combination of PMA or TPMA with CA significantly improves the wrinkle-recovery angle of finished cottons. The TPMA/CA combination is more effective. The synergistic effect of combining TPMA with CA can be due to the in situ reaction between CA and a polymeric carboxylic acid (PMA or TPMA) on the fabric under curing conditions and the formation of more flexible cross-linking networks. These finishing agents reported to be more cost effective than BTCA.

3.2. 1,2,3,4-Butanetetracarboxylic acid (BTCA)

Among the various effective polycarboxylic acids, 1,2,3,4-butanetetracarboxylic acid (BTCA) has proved to be the most efficient cross-linking agent for cotton fabrics (Morris, Morris, & Trask-Morrell, 1992; Yang & Wang, 2000; Yang, Lu, & Lickfield, 2002).

Esterification of cotton cellulose with BTCA to form crosslinks is believed to proceed through a mechanism involving formation of an intermediate cyclic anhydride, which then reacts with the cellulosic hydroxyl to complete the ester linkage (Bertoniere, King, & Welch, 1994).

It has been suggested that the catalyst may accelerate both the formation of anhydride and its reaction with cellulose (Welch, 1992). In 1996, Morris and his coworkers (Morris, Morris, & Trask-Morrell, 1996) explored the interactions between

phosphorus-containing catalysts and BTCA to explain the catalyst role. Sodium hypophosphite has proved to be the most effective catalyst for cross-linking of cotton by polycarboxylic acids, offering the greatest reduction in curing temperature with the least fabric yellowing. The role NaH_2PO_2 plays as a catalyst for cotton esterification has been studied in the past (Morris et al., 1996).

Morris et al. (1996) suggested that BTCA forms complexes with NaH_2PO_2 or NaH_2PO_4 in solutions based on the increased solubility of BTCA upon the addition of those compounds. They reported that in a BTCA– NaH_2PO_2 mixture, BTCA forms an anhydride at temperatures lower than BTCA alone. In 2000, Gu et al. (Gu & Yang, 2000) investigated the effects of sodium hypophosphite on the formation of cyclic anhydride intermediates of polycarboxylic acids at elevated temperatures. They reported that in a crystalline state, BTCA starts to form five membered cyclic anhydrides when the temperature reaches the vicinity of its melting point. Sodium hypophosphite weakens the hydrogen bonding between carboxylic acid groups of BTCA, thus making it possible for BTCA to form the cyclic anhydride at temperatures significantly lower than its melting point.

Cyclic anhydride can be formed by heating. However, it can be formed more effectively even at low temperature in the presence of a sodium carboxylate catalyst. On the basis of thermal analysis, Trask-Morrell, Andrews, and Graves (1991) reported that the reaction time and reaction energy for anhydride formation both decreased in the presence of a catalyst. In this process BTCA is transformed to a carboxylate anion by sodium carboxylate, and the carboxylate anion is converted to anhydride by heat and formed an ester linkage with cellulose through the nucleophilic substitution reaction (Lee & Kim, 2001).

3.3. Malic acid

It has been considered that malic acid, α -hydroxy succinic acid, is not capable of forming crosslinks in the cellulosic materials unless activated by other polycarboxylic acids such as CA or BTCA, because only two carboxylic acids per molecule are available for the formation of one anhydride intermediate (Kim, Jang, & Ko, 2000). Kim et al. (2000) reported that dicarboxylic malic acid with sodium hypophosphite catalyst without the addition of other cross-linkers is able to improve wrinkle resistance of cotton. They believed that the cross-linking of cotton is due to the trimeric α,β -malic acid, a tetracarboxylic acid, which can form two anhydride rings during curing. According to their findings, the amount of SHP required in the malic acid finish is significantly lower than the usual amounts of SHP in BTCA or CA finishes which claimed to be the advantage of the malic/SHP system. Their findings also indicated that inclusion of reactive polyurethane resin in the formulation causes improvement in the mechanical strength retention of cross-linked fabric and additional increase in wrinkle resistance.

3.4. Maleic acid

Chen, Yang, and Qiu (2005) synthesized oligomeric poly(maleic acid) (PMA) as a new non-formaldehyde durable press finishing agent for cotton. They claimed that PMA is more cost-effective than BTCA and causes no fabric yellowing. According to their findings, the fabric treated with PMA is equally effective in cross-linking cotton fabrics as DMDHEU while imparted higher strength retention than it. Yang (1993a, 1993b) found that sodium hypophosphite accelerates the formation of the cyclic anhydride of poly(maleic acid) on cotton.

Peng, Yang, and Wang (2012) suggested the combination of maleic acid (MA) and sodium hypophosphite as a formaldehyde-free and cost-effective durable press finishing system with superior fabric strength retention.

They claimed that wrinkle resistance of the cotton fabrics was achieved by the esterification of cotton cellulose by MA; and the addition of H–P–(residual of sodium hypophosphite) to $>\text{C}=\text{C}<$ of MA, which takes place at temperatures significantly higher than those of the esterification of cellulose.

3.5. Itaconic acid (ITA)

Research has been done by Yang et al. in 2001 (Mao & Yang, 2001) on the effectiveness of poly itaconic acid (PITA) synthesized in an aqueous solution and ITA polymerizing in situ for cross-linking cellulose and imparting wrinkle resistance to the cotton fabric. They reported that although the tensile strength loss of the cotton fabric cross-linked by ITA polymerizing in situ is very similar to that cross-linked by PITA applied as a polymer, ITA is more effective in esterifying cotton cellulose. In 2003 (Yang, Hu, & Lickfield, 2003a), they reported that the cotton fabric treated with ITA, lost more tensile strength than that treated with PITA due to cellulose degradation and the tensile strength loss caused by cross-linking for PITA treated fabric is significantly higher than that for ITA treated fabric. This was believed to be due to more concentrated cross-linkages formed on the near surface of the fabric treated with PITA.

3.6. Simultaneous application of maleic and itaconic acid

Choi (1992) reported that cotton fabric treated with two unsaturated bi-functional carboxylic acids, i.e., maleic acid (MA) and itaconic acid (ITA), in the presence of potassium persulfate as an initiator (1.5% of the acids) showed substantial increase in wrinkle resistance. Choi concluded that in situ copolymerization of MA and ITA takes place on the cotton fabric, and the copolymer esterifies and crosslinks cotton cellulose. However, no experimental evidence for the existence of the copolymer on the treated fabric reported and no data supported the durability of the treated cotton to home laundering.

Neither maleic acid (MA) nor itaconic acid (ITA) polymerizes under normal conditions used for vinyl monomers. In 1999, Yang et al. (Yang & Lu, 1999) reported that mixture of MA and ITA can homo and copolymerize on the cotton at elevated temperatures when both potassium persulfate and sodium phosphate are present and that the concentration of potassium persulfate in the finishing system has a major effect on in situ polymerization and the performance of the treated fabric. They also found that itaconic acid is more effective than maleic acid in providing wrinkle resistance to cotton fabric. In 2000 (Yang & Lu, 2000), the same researchers used MALDI/TOF mass spectroscopy and MALLA data and confirmed that the in situ copolymerization of MA and ITA takes place on the cotton. They reported that the molecular weight of the copolymer of MA and ITA formed in situ on the cotton fabric is in the approximate range of 2000–3000.

3.7. Other present cross-linking agents

In spite of the effectiveness of BTCA, the commercial use of BTCA has been deterred due to the significantly higher cost of it compared with the formaldehyde-based compounds. More research was carried out by Lewis et al. (Lewis & Voncina, 1997a, 1997b) to find an alternative polycarboxylic acid to replace the most effective but expensive cross-linking agent BTCA. They reported the preparation of 2-hydroxy-4-6-di-thiosuccinyl-s-triazine (HDTST).

In the other work the same group studied the optimum conditions for effective cross-linking of cotton fabric with HDTST in the presence of SHP (Lewis & Voncina, 1997a, 1997b).

Mao et al. (Mao & Yang, 2001) also investigated the formation of cyclic anhydrides by 1,2,4-butanetricarboxylic acid (BTA) and 1,2,3-propanetricarboxylic acid (PCA).

According to their findings, when an acid molecule is bonded to cotton through an ester linkage, only PCA form a second anhydride intermediate. Thus, PCA reported to be more effective cross-linking agent and impart higher levels of wrinkle resistance than BTA with or without use of catalyst.

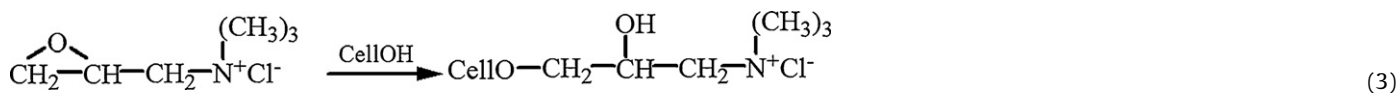
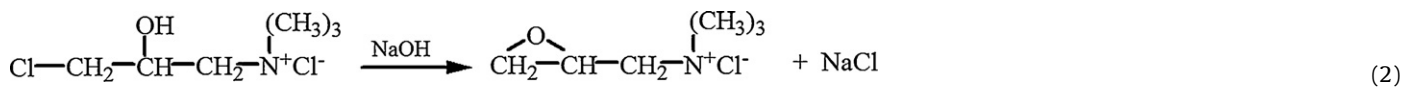
In 2007, Li and his coworkers (Li, Jiang, Wang, Meng, & Qing, 2007) synthesized a novel anionic aqueous polyurethane (NAPU) modified with silane coupling agent and maleic anhydride. They claimed that cotton fabrics treated with NAPU retained their tensile strength and tear strength better than treated with CA. Although whiteness of the fabrics treated with NAPU was lower than the untreated cotton and the fabrics treated with CA, the color change was still within the acceptable range.

3.8. Ionic cross-linking

A new method based on ionic cross-linking for non-formaldehyde, easy care finishing of cotton fabric was suggested by Hashem, Refaie, and Hebeish (2005).

The ionic cross-linking was accomplished by subjecting cotton fabric successively to partial carboxymethylation using monochloro acetic acid and sodium hydroxide then cationization using 3-chloro-2-hydroxypropyl trimethyl ammonium chloride (Quat-188). While the partial carboxymethylation reaction introduced the negatively charged groups in the cellulose chains of cotton, the cationization reaction introduced the positively charged groups thereby causing ionic cross-linking. Ionic cross-linking of cotton fabric significantly improved its wet crease recovery angle, tensile strength and elongation at break with a little improvement in dry recovery angle compared with that of untreated cotton fabric.

The reaction of cotton fabric with 3-chloro-2-hydroxypropyl trimethyl ammonium chloride was suggested as reactions (2) and (3) (Hebeish, Hashem, Abdel-Rahman, & El-Hilw, 2006)

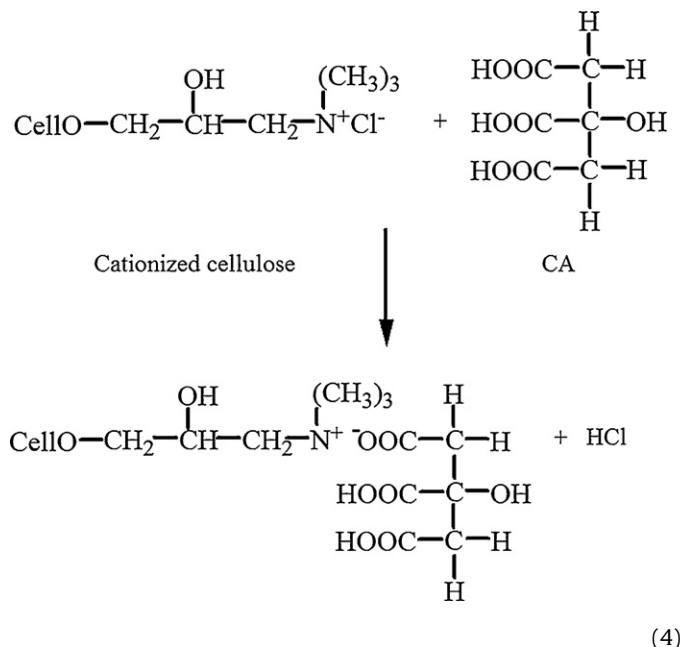


3.9. Ester and ionic cross-linking

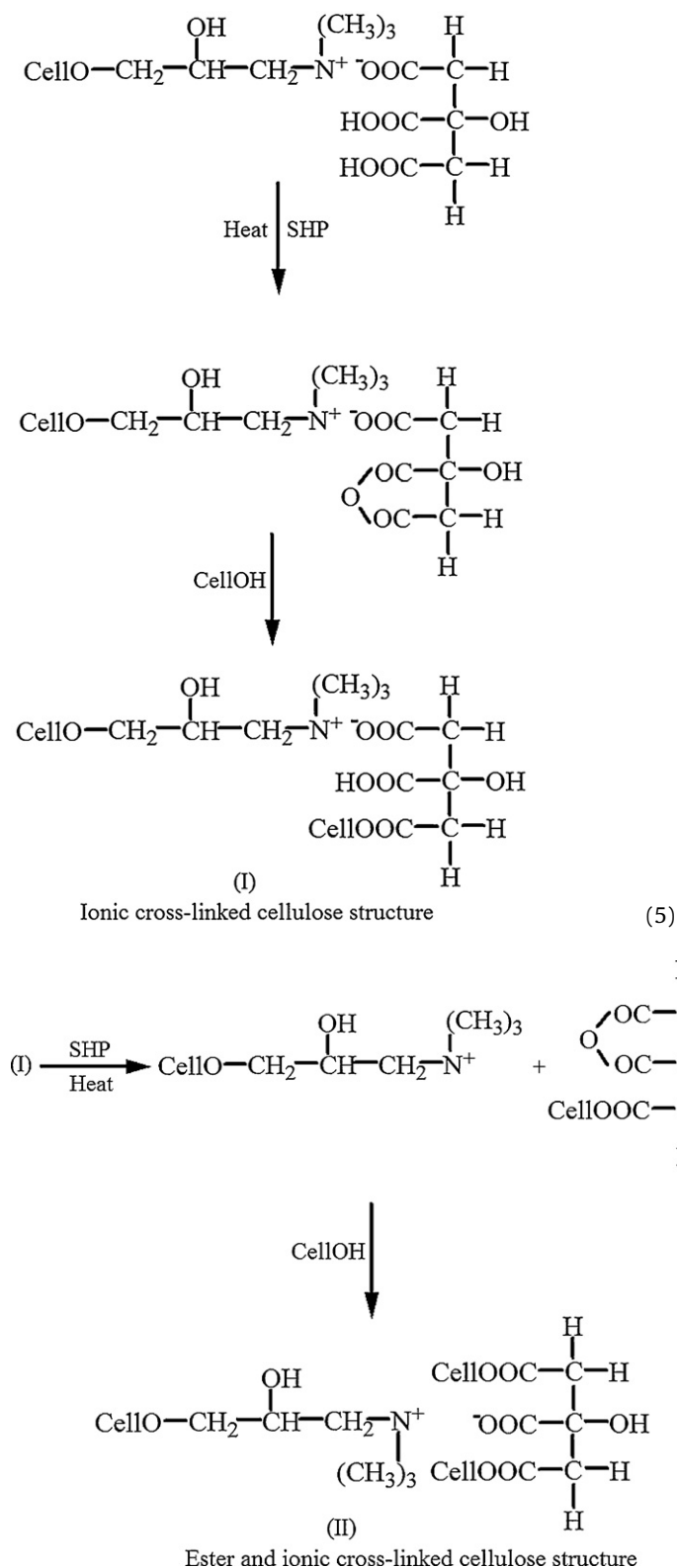
In 2006 (Hebeish et al., 2006), the same group introduced cationic groups in the molecular structure of cotton cellulose prior to polycarboxylic acid finishing treatments. In their study, cationization of cotton fabric by using 3-chloro-2-hydroxypropyl trimethyl ammonium chloride (Quat-188) in alkaline medium was followed by the BTCA or CA cross-linking along with SHP as a

catalyst. They claimed that the following reactions occurred between the cationized cotton fabric and CA:

(1) Formation of ionic bonds during the finishing treatment as shown in reaction (4):



(2) Esterification of cationized cotton with CA and formation of ionic cross-linking as shown in reactions (5) and (6) (Hebeish et al., 2006).



In 2011 (Hashem, Elshakankery, Abd El-Aziz, Fouda, & Fahmy, 2011), pre-cationized cotton fabric was cross-linked using ammonium citrate in presence of sodium hypophosphite at 180 °C for 90 s by Hashem et al. Results showed that the pre-cationization of cotton fabric having 0.09% nitrogen followed by cross-linking with 6% ammonium citrate brings about an enhancement in both dry and wet recovery angles along with a slight improvement in tensile strength.

Nazari et al. investigated the antibacterial, anti-wrinkling and self-cleaning properties of cationized cotton treated with nano TiO₂ and BTCA. According to their findings, cationization pre-treatment of cotton fabric leads to improve the self-cleaning property (Nazari, Montazer, & Rahimi, 2009a; Nazari, Montazer, Rashidi, Yazdanshenas, & Anary-Abbasinejad, 2009b; Nazari, Montazer, Moghadam, & Anary-Abbasinejad, 2011).

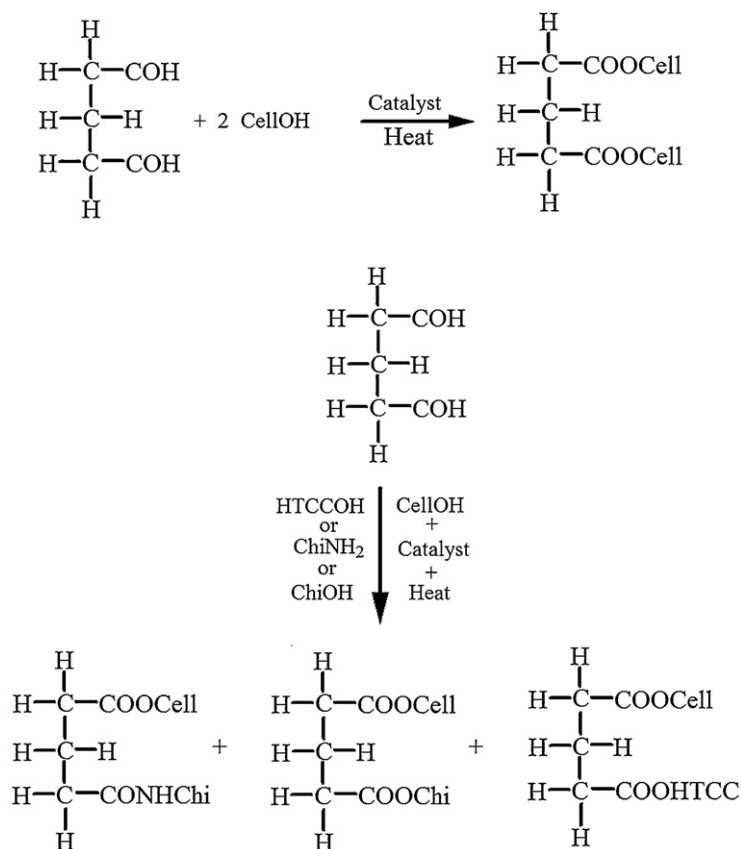
3.10. Co-application of cross-linking agents and antimicrobial, flame retardant and water repellent finishing treatments

In addition to cotton crease resistant finish, antimicrobial and flame retardant finishing of cotton fabrics are necessary treatments as cotton textiles have poor resistance to microorganisms (Dastjerdi & Montazer, 2010; Fu, Shen, Jiang, Huang, & Yan, 2011; Gao & Cranston, 2006; Ibrahim, Abdel Rehim, & El-Batal, 2010; Kim, Kim, & Rhee, 2010) and are highly combustible (Wakelyn & Turner, 1998). Also water repellency is an important property for some functional fabrics. Nowadays, use of the mentioned finishing agents in combination with bonding agents has been extensively investigated.

3.10.1. Antimicrobial property

Chitosan has attracted considerable interests due to its biological activities such as antimicrobial effects (Kim, Kim, & Choi, 1997; Lim & Hudson, 2004a, 2004b; Xie, Liw, & Chen, 2007; Yang, Liu, & Guan, 1999; Zheng, Zhu, & Sun, 2000). In recent years, many researchers have been studied the antimicrobial property of treated fabrics with chitosan (Fahmy & Fouda, 2008; Lim & Hudson, 2004a, 2004b; Sepahi Rad, Montazer, & Rahimi, 2011; Shenassa, Khodarahmi, Hemmatinejad, & Montazer, 2007; Shi, Zhao, Zhang, Su, & Tan, 2008; Yang, Dou, Liang, & Shen, 2005a).

In this sense, the chemical fixation of the chitosan by covalent cross-linking into the cellulose may improve the endurance and efficiency of the resultant functionalized textiles. Kim, Nan, Choi, and Jang (2003) applied *N*-(2-hydroxy) propyl-3-trimethyl ammonium chitosan chloride (HTCC) on the cotton fabric and found that it imparted good but not durable antimicrobial properties to the treated fabric. They suggested using a cross-linking agent in



(8)

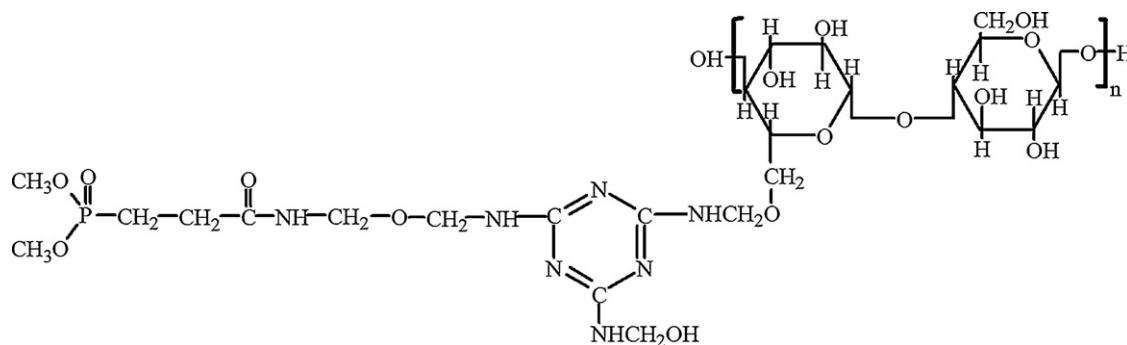
Huang, Wu, Chen, and Lian (2008) mixed low molecular weight chitosan (LWCS) with DMDHEU and found that the addition of LWCS increased the tensile strength retention and crease recovery of the treated fabrics. Fouda, El Shafei, Sharaf, and Hebeish (2009) used microwave curing system to cure cotton fabrics treated with different cross-linking agents along with chitosan. Compared to conventional curing system, the microwave curing was found advantageous in production of cotton fabrics with easy care antibacterial properties without high losses in strength properties.

The simultaneous cross-linking and antimicrobial finishing of bleached cotton and cationized cotton by 3-chloro-2-hydroxypropyl trimethyl ammonium chloride (Quat-188) with butane tetracarboxylic acid and nano titanium dioxide was proposed by Nazari et al. (2009a).

In 2010, Hosseini et al. suggested 10% CA, 3% AgNO₃ and 1.14 g SHP as an effective finishing agent in improving anti-wrinkling and antibacterial properties of denim fabrics (Hosseini & Mirbagheri, 2010).

3.10.2. Flame retardancy

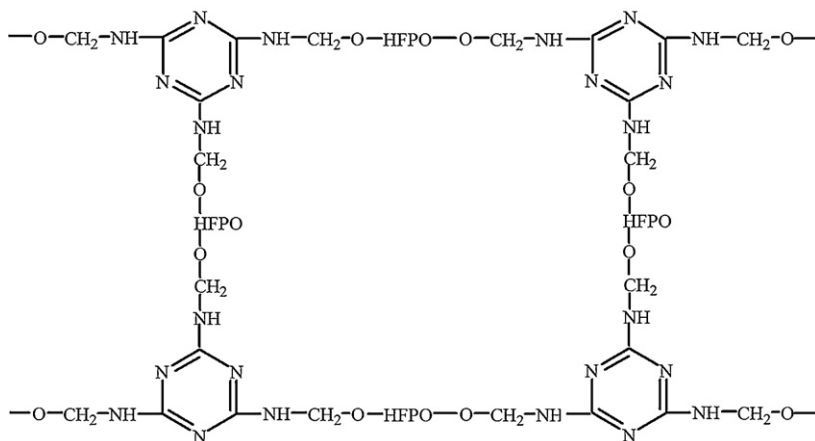
The use of organophosphorus chemicals to reduce the flammability of cotton has been the focus of textile flame retardant finishing since 1960s. Among the various flame retardant agents developed, *N*-methylol dimethylphosphonopropionamide (MDPA) and its commercial versions with the names of “Pyrovatex CP” or “Pyrovatex CP New”, have been one of the most widely used flame retardant finishing agents for cotton since 1960s (Cheng & Yang, 2009; Wu & Yang, 2004). Utilizing flame retardant agents in combination with bonding agents have been extensively investigated. It was reported that MDPA was covalently bound to cellulose by the reaction between its *N*-methylol group and trimethylolmelamine (TMM) used as a co-reactant in the finishing system. Wu et al. (Weil & Levchik, 2008; Yang & Qiu, 2007; Yang, Wu, & Xu, 2005b) developed a flame retardant finishing system for cotton based on a hydroxy-functional organophosphorus oligomer (HFPO). It was reported that MDPA was bound to cotton cellulose by the reactions of TMM with both MDPA and cellulose as shown in reaction (9):



(9)

TMM and DMDHEU were used as the bonding agents in the finishing system to form a covalent linkage between HFPO and cotton. HFPO, like MDPA, is also a condensed-phase flame retardant agent whereas TMM or DMDHEU functions as a nitrogen provider to enhance the performance of HFPO due to nitrogen–phosphorus synergism.

The data also revealed that MDPA can react directly with cellulose through its *N*-methylol group. However, HFPO does not have any reactive functional group to form covalent bonding with cotton, and it requires a bonding agent, such as TMM, to bond it to cotton. The cross-linked polymeric network of HFPO/TMM is shown in reaction (10) (Wu & Yang, 2006a, 2006b):



They also concluded that the cotton fabric treated with MDPA/TMM had a higher initial LOI than that treated with HFPO/TMM when both finishing solutions have the same phosphorus content and same TMM concentration. This believed to be due to the fact that MDPA/TMM system contains significantly higher nitrogen content, which enhances the flame resistant performance by means of phosphorus nitrogen synergism. The cotton fabric treated with both HFPO/TMM and MDPA/TMM had low levels of cross-linking and low strength loss (Wu & Yang, 2007).

In 2008, Wu et al. (Wu & Yang, 2008a, 2008b) claimed that the treatment using the combination of maleic acid and sodium hypophosphite leads the cross-linking of cotton cellulose and is effective in reducing the flammability of cotton fleece. They also reported the applications of phosphorus-containing maleic acid oligomers (PMAO), synthesized by aqueous free radical polymerization of maleic acid, as an effective flame-retardant agent for cotton fleece.

A novel flame-retardant for cotton fabric using sodium hypophosphite (SHP), maleic acid (MA), triethanolamine (TEA) and nano TiO₂ was suggested by Lessan, Montazer, and Moghadam (2011). The proposed treatment claimed to form more non-flammable char residue and increase char formation after heating. They believed that phosphorus deposited on the SHP treated samples acts as the most effective parameter in the char forming and decreasing the flammability of the treated fabrics. Also, nano TiO₂ considered as an effective compound in increasing the char formation.

3.10.3. Water repellency

Fluorocarbon resin is the most effective treating agent for making fabrics water repellent (Li, Fub, Wang, & Liuc, 2008; Shao, Sun, Meng, & Qing, 2004). To improve the washing durability of water repellency, some cross-linking agents are usually used along with

the water repellent agents. Montazer (1996) applied BTCA, with the optimum concentration of 5 g/L, and different concentrations of fluorocarbon agent for water repellent finishing of cotton fabrics.

BTCA is confirmed to be an effective cross-linking agent with sodium hypophosphite (SHP) catalyst for washing durability improvement of cotton fabrics treated with fluorocarbon resin. It has been reported that the water repellency of the sample treated with fluorocarbon resin and 8% BTCA is much higher than the sample treated only with fluorocarbon resin (Xu & Shyr, 2001). This kind of difference can be seen especially after fifty washing cycles and subsequent heat treatments.

(10)

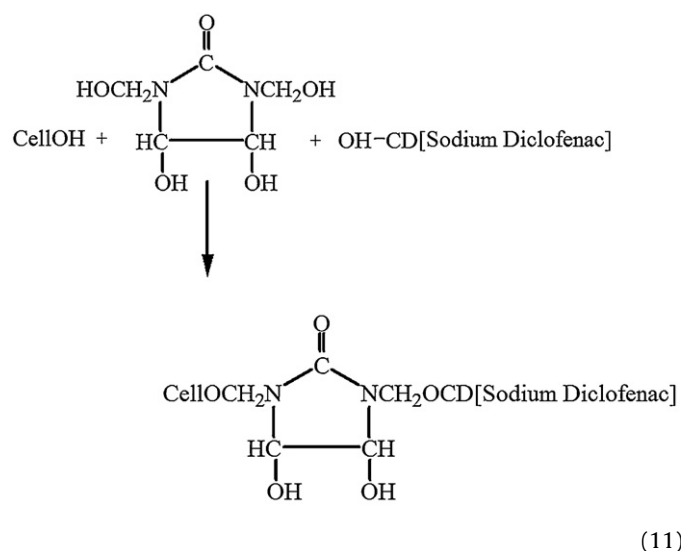
In 2008 (Li et al., 2008), an acrylate monomer containing hydroxyethyl sulfone as cross-linking group was synthesized and a novel per-fluorinated acrylate copolymer (NPAC) was prepared by emulsion polymerization and applied on cotton fabrics. The finished fabric revealed good and durable water repellency property. Moreover, the treatment resulted in an increase in crease recovery.

3.11. Use of cross-linking agents as stabilizers

3.11.1. Stabilization of β -CDs on textiles

The use of cyclodextrins and their derivatives (CDs) in the textile domain is a challenge that arose in the early 1980s. β -cyclodextrin (β -CD) has a special structure and has been applied in finishing and dyeing processes (Ibrahim, Eid, & El-Zairy, 2011). The stabilization of β -CDs on textiles has been widely investigated. Denter et al. (Denter & Schollmeyer, 1996) and Reuscher and coworkers fixed a monochlorotriazinyl β -CD derivative onto different polymer materials, including cotton fibers. In the other study, the possibility of fixing β -CD permanently to cotton and wool fibers with poly(carboxylic acid)s as binding and cross-linking agents was proposed. Montazer et al. (Montazer & Jolaei, 2010a, 2010b) used three different cross-linking agents, including two non-formaldehyde cross-linking agents (CA and BTCA) and one formaldehyde based cross-linking agent (DMDHEU) to stabilize β -CD on the spacer polyester fabric. Among the different cross-linking agents, the best yield was found when BTCA was used for finishing 3D polyester fabric.

They (Montazer & Bigdeli Mehr, 2010) also used cross-linking agent based on DMDHEU for binding complex of β -cyclodextrin and sodium diclofenac to wound dressings. The reaction of methylol groups of DMDHEU with hydroxyl groups of cotton and hydroxyl groups of β -cyclodextrin is illustrated in reaction (11):



3.12. Co-application of cross-linking agents and nano and bio technology

3.12.1. Biotechnology and cross-linking treatments

As the durable press finishing process results in less fiber flexibility, it has negative effect on fabric handle (Dadashian, Montazer, & Ferdowsi, 2010; Kim & Song, 2006). Enzyme technology has been widely used to improve handle and appearance of cotton fabrics. In 2003, Yang et al. reported the effects of pre and post cellulase treatment on the tensile strength, flex abrasion and handle of BTCA–cotton treated fabrics (Yang, Zhou, Lickfield, & Parachura, 2003). They found that cellulase treatment of wrinkle resistant cottons decreases their tensile strength and abrasion resistance but improves handle. While the pre cellulase treatment imparts better handle, it causes higher losses in fabric mechanical strength. Therefore improving fabric handle by pre-cellulase treatment must be limited in heavy cotton fabrics with high original strength.

Hebeish et al. (2009) investigated the effects of bio-treatment using cellulase on easy-care properties of DMDHEU-treated cotton fabrics. Bio-treatment was carried out before and after cross-linking. Their findings indicated that fabric bio-polishing treatment had particularly no effect on wrinkle recovery angle. The surface roughness obtained with pre-cross-linking reported to be a bit higher than those of post-cross-linking. Besides, post-cross-linking resulted in higher losses in strength properties.

3.12.2. Nanotechnology and cross-linking treatments

3.12.2.1. Nanometer materials. The use of nanometer titanium dioxide or other nanometer materials as catalysts have been investigated for many years to enhance the catalytic effect on the improvement of some properties (Dastjerdi, Montazer, & Shahsavan, 2009, 2010; Dastjerdi & Montazer, 2011; Ki, Kim, Kwon, & Jeong, 2007; Mihailovic et al., 2009; Montazer & Pakdel, 2010; Montazer, Pakdel, & Moghadam, 2010; Montazer, Asghari, & Pakdel, 2011a; Montazer, Behzadnia, Pakdel, Karim Rahimi, & Bameni Moghadam, 2011b; Montazer, Pakdel, & Behzadnia, 2011c; Montazer, Pakdel, & Moghadam, 2011d; Yang & Zhu, 2004). Using the nanometer TiO_2 as a catalyst to improve the crease recovery property of fabrics has been one of the current topics (Montazer & Pakdel, 2011a, 2011b; Nazari, Montazer, Moghadam, Rashidi, & Yazdanshenas, 2010). It has been shown that the electron of the nanometer titanium dioxide particle in the valence band will be excited to the conduction band under the radiation of ultra-violet (UV) light whose wavelength is lower than 400 nm for a few microseconds (ms) to raise the electron to the surface of the

nanometer particle. This phenomenon is called photo-reduction. Then it can reduce the water molecule and hydroxyl ion that are absorbed on the surface of the other nanometer material in the reaction system to create a series reaction shown in Scheme 1.

The carboxylic acid group and the vinyl double bond of the acid cross-linking agents could be activated by those radicals therefore could react with cellulose molecule (cross-linking) and react with each other (condensation). In 2005, Wang et al. (Wang & Chen, 2005a, 2005b) used four cross-linking agents of carboxylic acids (1,2,3,4-butane tetracarboxylic acid, maleic acid, succinic acid, and citric acid) to treat cotton fabrics in the presence of nanometer TiO_2 as catalyst under the irradiation of UV light which has the wavelength at 254 nm and under the treatment of electronic field (EF). According to their results, the DCRA, WCRA, and softness values of the treated fabrics for all the cross-linking agents increased with the increasing of irradiation time period, but tensile strength retention (TSR) values decreased in all cases. They claimed that the catalytic effect of EF treatment were higher than those obtained by UV irradiation at a given time period.

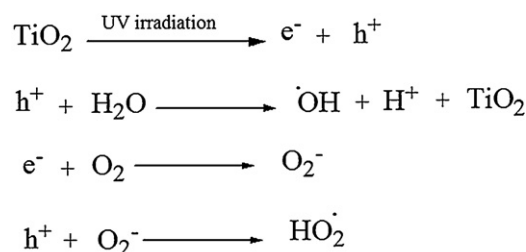
In 2006 (Chen & Wang, 2006), the same group investigated the mechanism of cross-linking with succinic acid and the photo-reduction of the succinic acid in the presence of nanometer TiO_2 catalyst in water medium. The suggested possible cross-linking reactions are as shown in Scheme 2 however, no experimental evidence for confirming their suggested mechanism has been reported.

In order to study the wrinkle resistance finishing of cotton fabrics using BTCA cross-linking agent combined with sodium hypophosphite catalyst and nano TiO_2 co-catalyst, Yuen et al. (2009) evaluated the effect of changes in the concentration of nano TiO_2 on some of the properties of treated fabrics. They found that the addition of nano TiO_2 could further enhance the wrinkle resistance and decrease the stiffness of the cotton fabrics with little effect on the tearing and tensile strength. Nazari et al. (2009b) utilized CA and BTCA to treat bleached cotton fabrics in the presence of nano TiO_2 and SHP as catalysts under three different curing conditions including: UV irradiation (UV), high temperature (High temp) and a combination of UV and high temperature (UV-High temp). According to their findings, the cross-linking process occurred more effectively under UV-High temp condition than those obtained by UV or High temp, alone.

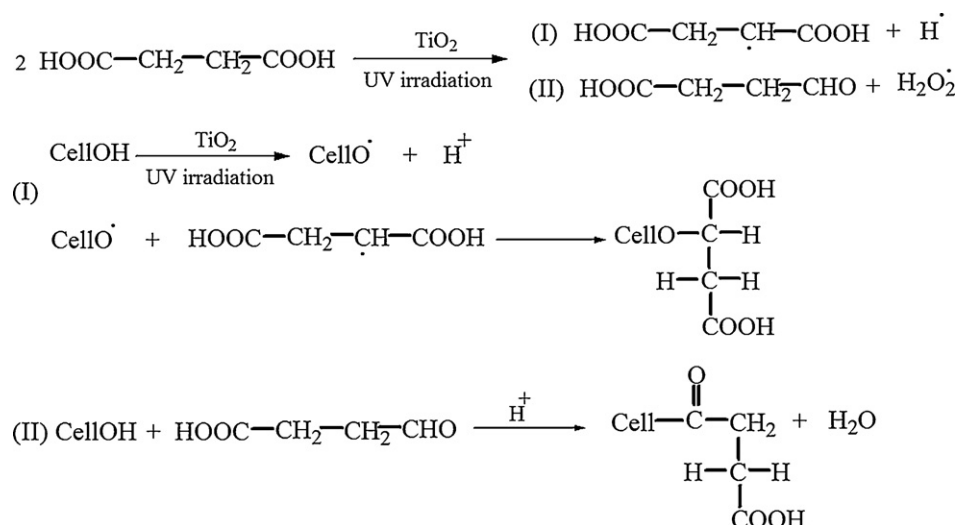
They claimed that in the UV-Temp curing method, both cross-linking mechanisms of cotton fabric based on UV and Temp occurred. They believed that, as irradiation of nano TiO_2 with UV light in the wet carboxylic acid leads to the formation of positive holes on TiO_2 surface and proton, these holes as well as proton may act as a Lewis acid catalyst and activate the carbonyl group of acid toward the addition of hydroxyl group of cotton fibers, which finally leads to an ester bond formation.

Their proposed mechanism is shown in Scheme 3.

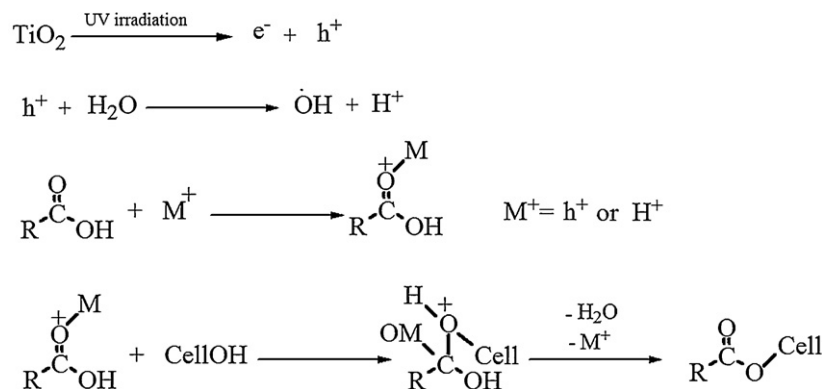
Based on these findings, nano TiO_2 acts as a co-catalyst to enhance the anti-creasing properties of polycarboxylic/SHP treated cotton.



Scheme 1. TiO_2 photocatalytic reactions.



Scheme 2. Mechanism of cellulose cross-linking with succinic acid using nano TiO₂ suggested by Wang et al. (Chen & Wang, 2006).



Scheme 3. Mechanism of cellulose cross-linking with poly carboxylic acids using nano TiO₂ suggested by Nazari et al. (2009b).

3.12.2.2. Sol-gel treatment. Sol-gel technology provides a new way to functionalize fabrics by improving their physical properties (Alongi, Ciobanu, Tata, Carosio, & Malucelli, 2011). In textile materials, this technique is able to modify the surface thus imparting superior mechanical, hydrophobic or oleophobic surface properties to the substrate.

In 2004 Schramm et al. (Chen & Wang, 2006) investigated the effect of two step finishing procedure (BTCA treatment, sol-gel tetraethoxysilane and 3-glycidioxypropyltrimethoxysilane (TEOS/GPTMS) treatment) on the properties of the cotton fabric. Their findings made evident that the coating with a sol-gel solution results in a remarkable improvement in the abrasion resistance, while the dry wrinkle recovery angle values could be moderately improved.

Huang, Yang, Lin, and Lian (2007) applied mixed sol of tetraethoxysilane (TEOS)-titanium (IV) n-butoxide (TTB)/DMDHEU on cotton fabric and explored its effect on the physical properties of treated fabrics. They reported that hydrogen bonds formed between SiO₂ and DMDHEU and with an increase in the mole ratio of TEOS the treated fabrics revealed improved anti-wrinkle properties and strength retention.

3.13. Major deficiency of cross-linking agents

The tensile strength, tearing strength and abrasion resistance of the cotton fabrics treated with cross-linking agents are significantly

reduced as a result of this finishing process (Yang, Wei, & Lickfield, 2000a; Yang, Wei, & Lickfield, 2000b).

The use of both *N*-methylol compounds, such as DMDHEU and poly-functional carboxylic acids has been shown to cause reduction in the physical properties of finished fabric (Lickfield, Yang, Quian, Wei, & Chen, 1999).

Fig. 3 illustrates the mechanical strength loss associated with durable press finish.

Some of the more important aspects of strength loss occurred in wrinkle resistance finish have been summarized by Lickfield et al. (1999):

1. The catalysts used for DMDHEU systems cause degradation of cellulose, thus reducing the tensile strength of cotton fabric. The magnitude of fabric strength loss is affected by the temperature, time, and concentration of the catalyst. The selection of the catalyst system and its concentration is crucial for optimizing the tensile strength retention of the finished fabrics.
2. Both DMDHEU and polycarboxylic acid crosslinks can be removed from finished fabric using an alkali treatment. The fabric tensile strength gradually increases as the hydrolysis of the cross-linked fabric progresses, indicating that the fabric strength loss due to cross-linking the cellulose molecules is reversible and that it can be restored by removing the cross-links. The remaining of the strength loss, which has been described as being due to acid-induced de-polymerization, is permanent and is not reversible upon hydrolysis of the cross-links.

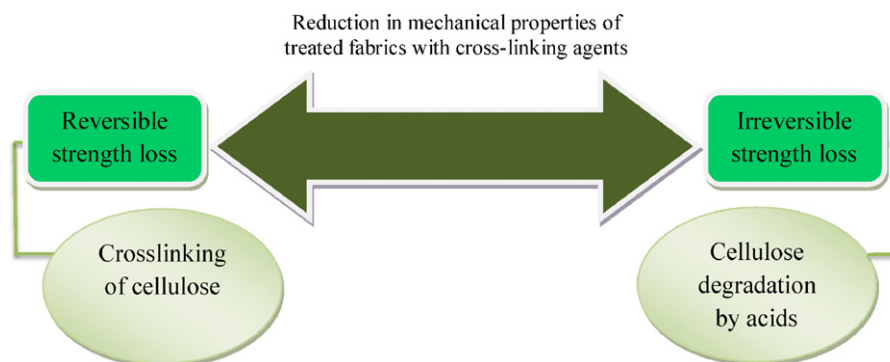


Fig. 3. Strength loss due to acid degradation and cross-linking.

- Exposure of a cotton fabric treated with a polycarboxylic acid at elevated temperatures reduces the fabric tensile strength. The magnitude of fabric strength loss is affected by the temperature and time of the exposure, the concentration of acid applied to the fabric, the dissociation constant of the acid, and the pH of the acid solution applied to the fabric.
- There is a direct correlation between tensile strength loss of the treated fabric and the molecular weight of cellulose.

According to the data reported by Lickfield et al. (1999) the total tensile strength loss increases as the WRA of the treated fabric increases, and a linear relationship exists between these two properties. This relation between the tensile strength loss and wrinkle resistance for cross-linked cotton fabrics appears to be independent of all of the differences in the molecular structure and reactivity of the polycarboxylic acids.

Yang et al. (Kang, Yang, Wei, & Lickfield, 1998) revealed that, the magnitude of tensile strength loss for BTCA–cotton treated fabrics in the presence of a catalyst is far less than in the absence of it. This may be due to the accelerated effect of the catalyst on esterification of cellulose which reduces the acidity of the fabric and results in less acid-catalyzed de-polymerization.

Therefore, studies have been carried out to improve treated cotton fabric strength retention. In 1962, Rebenfeld suggested that exerting tension on the fibers during curing would result in more resilient, stiffer and less extensible fabrics (Rebenfeld, 1962, 1964). According to Xu et al. findings (Xu & Li, 2000a, 2000b), the tension during curing can improve fabric strength from 45% to 75% without creating negative effects on wrinkle resistance.

Zhou, Yang, and Lickfield (2004) applied poly(vinyl alcohol) (PVA) as an additive to improve the abrasion resistance of the cotton fabric treated by BTCA. Their method was an effective way in improving the abrasion resistance of the cross-linked cotton fabric with no negative effect on the wrinkle recovery angle. The reaction of PVA and BTCA may form a protective layer on the fiber surface, reinforcing the mechanically weak points on the fiber surface.

Also various researchers have investigated the abrasion resistance of DP-finished cotton fabrics. Fabric softeners, including amino-functional silicone polymers and high density polyethylene emulsions, hydroxyl-functional compounds, such as diethylene glycol and triethanolamine, have been investigated as additives to improve the abrasion resistance of the DP-finished cotton fabric (Zhou et al., 2004). But those reactive additives were all small molecules and were able to penetrate into the fiber interior and to react with the cross-linking agents inside the fiber. Consequently they have a negative effect on the wrinkle resistance of the cross-linked cotton fabrics.

Fahmy, Abo-Shosha, and Ibrahim (2009) found that cross-linking cotton fabric with solution of poly (*N*-vinyl-2-pyrrolidone) (PVP) results in wrinkle resistance and improvement in some

properties of the treated fabrics such as resiliency and tensile strength. They also reported that the incorporation of PVP in the easy-care finishing of cotton fabrics with DMDHEU enhances tensile strength along with decreasing wrinkle recovery rating as well as whiteness index of treated fabrics whereas the ester cross-linking with CA, in presence of PVP, enhances wrinkle recovery angle, tensile strength and whiteness index.

4. Future prospect

During the last 80 years, significant progress has been made in introducing cross-linking agents to cotton fabrics.

To date, a variety of cross-linking agents have been employed to overcome the limitations of the conventional finishing agents. However, more efficient cross-linking agents that can impart multifunctional properties to finished fabrics are still desired. In addition, the currently reported cellulose cross-linking agents have some negative effects on mechanical properties and proposing a cross-linking agent with no diverse influence on treated fabrics is still a challenge.

Most of the today multifunctional textiles are the result of cooperative treatment of fabrics with cross-linking agents and other finishing chemicals. Therefore introduction of cross-linking agents that can simultaneously crosslink cellulose and impart some other properties to it is in demand.

Considering the growing role of bio and nano technology in textile industry, bio and nano-cross-linking finishes will offer a very broad field of research, to address the corresponding challenges and then, to open new perspectives.

Future efforts may be focused on use of nano particles as fillers or cross-linking agents which because of their small size may be able to enter in between the polymer chains. By achieving this goal we can benefit from both nano properties and cross-linking, obtaining multifunctional textiles. Besides, unlike chemical cross-linking that has the disadvantage of imparting rigidity to the treated fabrics, future nano cross-linkers seem not to interfere much to the polymer flexibility. For instance, there has been some evidence that carbon nanotubes (CNTs) and carboxylated CNTs may cause improvements in crease resistance and strength of cotton fabrics by penetrating into cellulosic chains, which requires further investigations.

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