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# Application of reactive cyclodextrin poly butyl acrylate preformed polymers containing nano-ZnO to cotton fabrics and their impact on fabric performance

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ABSTRACT

Synthesis of reactive preformed polymers (PFP) and their application to cotton fabrics along with epichlorohydrin and/or ZnO nanoparticles are carried out as per the conventional pad-dry-cure method. PFP refers to monochlorotrazinyl β-cyclodextrin grafted with Butyl acrylate. The impact of grafting of this reactive preformed polymer on performance of cotton fabrics was studied. These grafting of PFP to cotton fabrics in presence of ZnO nanoparticles and /or epichlorohydrin imparts antibacterial activity which withstands 20 times washing while keeping 70% of this activity. These grafted fabrics acquire improved air permeability indicating better breathability and comfortability of garments make thereof. Also reported was the characterization of the grafted fabrics using IR spectral analysis and scanning electron microscopy. A ZnO nanoparticle was also characterized using UV–vis spectrophotometer.



## 1. Introduction

CDs are composed of glucose units, linked through 1,4-glycosidic bonds. α, β, and γ, cyclodextrins contain six, seven, and eight monosaccharide moieties, respectively. They produce a large variety of supramolecular inclusion complexes with numerous organic or inorganic species as shown in Rusa et al. (2004), Dodziuk (2002) and Belosludov et al. (2002). The commercial availability of cyclodextrins, lack of toxicity, and water solubility determine their success in many different applications spanning from pharmaceuticals to cosmetics, from food manufacturing to chromatography and textile processes as shown in Del Valle (2004), Huang, Gerber, Lu, and Tonelli (2001), Loftsson and Masson (2001), Wang and Chen (2005a), Szente and Szejtli (2004) and Walowitz, Fike, and Jayme (2003). The key factor of the cyclodextrin of binding different materials is the internal cavity. While the external surface and the rims of the truncated cone are quite hydrophilic, the inner cavity of a CD is composed of hydrophobic moieties. As a result, when aqueous solutions of CDs are mixed with polymers or smaller organic hydrophobic molecules, a rapid inclusion process takes place and often a white crystalline inclusion compound precipitates Becheri, Lo Nostro, Ninham, and Baglioni (2003). Such powerful capability of CDs to include hydrophobic molecules can be used in textile finishing. Indeed, several papers and patents report relevant applications of cyclodextrin for anti-microbial, aroma finishing, and in textile dyeing through the formation of physical bonds to different fibers Lee, Yoon, and Ko (2000), Savarino, Viscardi, Quagliotto, Montoneri, and Barni (1999) Wang and Chen (2005b)). Permanent grafting of different chemicals results in specific performances and new properties to these fabrics. Monochlorotriazinyl-β-cyclodextrin (CDMCT) is advocated as a great tool for surface modification of natural and synthetic fibers done by Reuscher and Hinsenkorn (1996). The reactive chlorine atom of the triazinyl groups can react with nucleophilic residues of these fibers as explained by Scheme 1 in case of cellulosic fabric. Once chemically grafted onto cellulosic substrates, these materials can be used for stabilization of active ingredients and nanoparticles as well as for production of smart textiles and textile products with highly specific properties as shown in Sawhney et al. (2008). the nanostructure are capable of enhancing the physical properties of textiles, in areas such as antibacterial, water repellency, soil resistance, antistatic, and so forth.

Nanocoating the surface of textiles for footwear is one approach to the production of highly active surfaces to have UV-blocking, antimicrobial properties and self-cleaning properties. The self-cleaning property can be imparted by nano-$TiO_2$/nano-ZnO coating as shown by Cavalli, Francesco Trotta, Carlotti, Possetti, and Trotta (2007).

While nano-Ag will impart anti-microbial property. Nanoparticle coating may affect the other fabric properties like dyeing property, strength, bending rigidity and air permeability and friction as show by Gorensek and Recelj (2007).

In previous work Hebeish, El Shafei, and Shaarawy (2009) we have extensively studied the chemistry of grafting of cotton with reactive preformed polymer (PFP), namely monochlorotriazinyl β-cyclodextrin grafted with poly Butyl Acrylate. The importance

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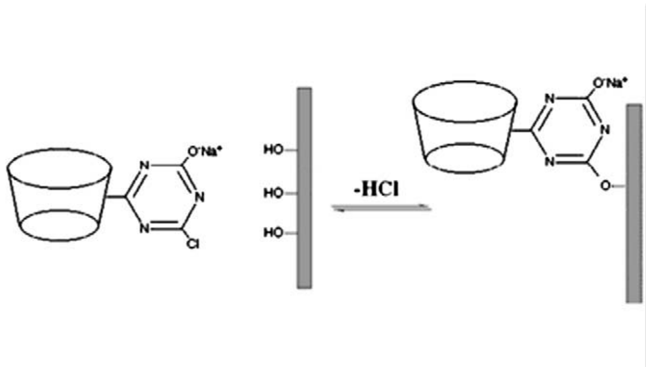


**Scheme 1.** Grafting reaction of CDMCT with a cellulosic fabric.

of such work and its scope in the realm of textile finishing were investigated.

As a continuity of our aforementioned work, we – in current work – present the impact of grafting of PFP to cotton fabrics when applied to the latter alone as well as together with ZnO nanoparticles and/or epichlorohydrin. Application to cotton fabric of the preformed polymer in question was preformed as per the conventional pad-thermo-fixation method and onset of this on nitrogen content, wrinkle-recovery angle, tensile strength, elongation at break as well as anti-microbial and air permeability properties were examined. Also examined were the characterization of PFP-cotton graft copolymers using IR spectral analysis and scanning electron microscopy (SEM). Furthermore, characterization of ZnO nanoparticles using UV–vis spectrophotometry was performed.

## 2. Experimental

### *2.1. Materials*

Scoured and rinsed 100% woven bleached cotton fabric ($75 \times 30$ g/m$^2$) was kindly supplied by Misr Company for Spinning and Weaving, Mehala El-Kubra, Egypt. Reactive cyclodextrin (RCD) which is monochlorotriazinyl β cyclodextrin MCT-β-CD was purchased by Wacker-Chemie GmbH (Munich, Germany). Butyl Acrylate BuA monomer, Fluka, Potassium persulphate (KPS), Ammonium persulphate (APS), Zinc nitrate, soluble starch, sodium hydroxide and ethyl alcohol were all of laboratory grade chemicals.

### *2.2. Synthesis of reactive preformed polymer*

The reactive preformed polymer, which is RCD grafted with poly (Bua), was prepared using an aqueous solution containing RCD 0.635 mole/l; BuA (0.6 mole/l), and KPS/APS 0.01 mole/l (at molar ratio 4:6) at this end the polymerization reaction was allowed to proceed for 3 h at 65 °C. For complete polymerization, the reaction was allowed to continue for other 18 h but at 30 °C. The graft add-on on the fabric due to reaction of the reactive preformed polymer with cotton fabric was determined through monitoring the *N*-content of cyclodextrin – poly (BuA) graft containing the triazinyl moieties.

### *2.3. Application through grafting of reactive preformed polymer to cotton fabric*

The cotton fabric was padded with reactive performed polymer at different concentrations of MCT-β-CD grafted with poly (BuA) (5–20%) and of non ionic wetting agent in presence or absence of 1% epichlorohydrin and 2% sod. carbonate using laboratory padder, then cured at 120 °C in an oven for 10 min.

### *2.4. Synthesis of ZnO nanoparticles*

The zinc oxide (ZnO) nanoparticles were prepared by wet chemical method using zinc nitrate and sodium hydroxide as precursors and soluble starch as stabilizing agent. Soluble starch of 0.1% concentration was prepared by dissolving the required weight of soluble starch in 500 mL of distilled water. Zinc nitrate, 14.874 g (0.1 mol), was added to this starch solution. Then the solution was kept under constant stirring using magnetic stirrer to completely dissolve the zinc nitrate. After complete dissolution of zinc nitrate, 0.2 mol of sodium hydroxide solution was added under constant stirring, drop by drop touching the walls of the vessel. The reaction was allowed to proceed for 2 h after complete addition of sodium hydroxide. After the completion of reaction, the solution was allowed to settle for overnight and the supernatant solution was then discarded carefully. Thus obtained nanoparticles were washed three times using distilled water. Washing was carried out to remove the byproducts and the excess starch that were bound with the nanoparticles. After washing, the nanoparticles were dried at 80 °C for overnight. During drying, complete conversion of $Zn(OH)_2$ into ZnO takes place.

### *2.5. Coating of cotton fabrics with ZnO nanoparticles*

ZnO nanoparticles, prepared as described above, were applied onto cotton using pad-dry-cure method. The cotton fabric was immersed in the solution containing ZnO (2%) and the reactive performed polymer at concentration range of (5–20%) in presence of non ionic wetting agent. The fabric was then passed through a padding mangle, A 100% wet pick-up was maintained for all treatments. After padding, the fabric was air-dried followed by curing at 140 °C for 3 min. at the end, The fabric was then immersed for 5 min in 2 g/l of sodium lauryl sulfate to remove unbound nanoparticles. Then the fabric was rinsed at least 10 times to completely take out all the soap solution. The fabric thus washed was air-dried. Simultaneously, bulk-ZnO coating was carried out for comparison.

### *2.6. Characterizations*

- FTIR spectra of fabric were recorded with Perkin Elmer Fourier-Transform Infrared (FTIR) spectrometer using KBR pellets.
- Nitrogen percent N% was measured using Kjeldhal method Vogel (1966).
- UV–visible spectra of ZnO suspended in deionized water were recorded in Specord 50 ANALYTIKJENA® spectrophotometer, from 200 to 800 nm.
- Air permeability was tested in air permeability tester KES-F8-AP1 designed by Kato Tech. Co., Ltd., Japan. Initially, the air resistance (Pa.s/m) is measured and its inverse gave the air permeability (m/Pa.s).
- Scanning Electron Microscopy (SEM) of RCD, reactive preformed polymer and fabric treated with reactive preformed polymer were obtained using SEM microanalyzer (ZXA-84oA-JEOL Tokyo-Japan). The specimens in the form of films were mounted on the specimen stabs and sputtered with thin gold films. The micrographs were taken at magnification of 1000× using TOKV accelerating voltage.

## 3. Results and discussion

### *3.1. Characterization of cotton fabric grafting with reactive preformed polymer (PFP) i.e. RCD grafted with poly (BuA)*

#### *3.1.1. IR spectral analysis*

Fig. 1 shows the IR spectra of the untreated cotton and cotton samples grafted with PFP in presence and absence of epichlorohy-

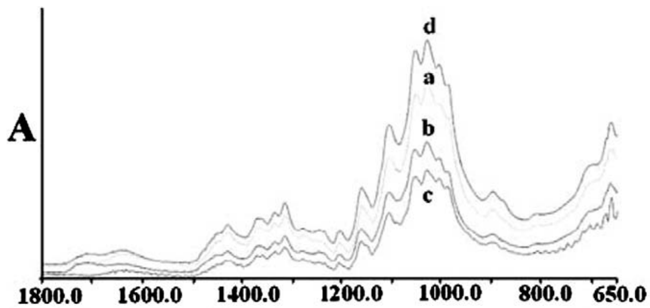


**Fig. 1.** Partial infrared spectrum of fabric samples (a) untreated cotton (b) treated with epichlorohydrin (c) treated with 20% PFP (d) treated with epichlorohydrin +20%PFP.

drin. Because of the similarity of the structure of cellulose (untreated sample) and CD moieties of the PFP they can barely be distinguished on the spectrum, even though the PFP is applied along with epichlorohydrin. The fabric treated with epichlorohydrin shows an additional peak at around 2900–3000 $cm^{-1}$, confirming the introduction of the epoxy groups, but it disappears when PFP containing CD moieties was applied. The differences at absorbance values were observed when spectrums are detailed between 3800–2800 $cm^{-1}$ (Fig. 1a) and 1800–650 $cm^{-1}$ (Fig. 1b). Fig. 1a shows that PFP blocks more –OH groups in the fabric than the epichlorohydrin reagent does, due to the lowered typical O–H absorption band at around 3300 $cm^{-1}$. When PFP and epichlorohydrin are applied together, the spectrum is similar to that of the PFP-containing cotton applied sample. The appearance of the increased absorption band at the 1000–1100 $cm^{-1}$ region in Fig. 1b (spectrum1 d) confirms that grafting PFP onto the fabric has been better achieved when applied with epichlorohydrin, which also highlights the crosslinks between PFP and epichlorohydrin.

### *3.1.2. Scanning electron microscope (SEM)*

Figs. 2 and 3 show SEM of blank cotton, cotton treated with RCD-g-poly (BuA), i.e. PFP, respectively. The differences in pictures show that crosslinking polymer is formed in situ within the fibrous network. The resulting polymer forms a coating of the fibers as shown in figures.

### *3.1.3. Characterization of ZnO Nano particles by using UV visible absorption spectrum*

ZnO nanomaterials have some excellent properties like exceptional mechanical strength, antistatic, antibacterial and UV absorption properties (Thünemann and Ruland 2000). UV visible spectrum taken for ZnO nanoparticle synthesized with 0.5% soluble starch (Fig. 4) shows peak absorption at 361 nm. By using effective mass approximation, the size (diameter) was calculated to be 40 nm.

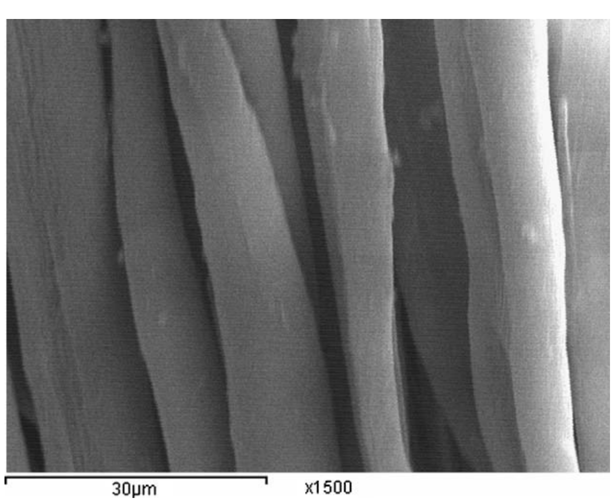


**Fig. 2.** SEM of blank cotton, cotton treated with RCD-g-poly (BuA).

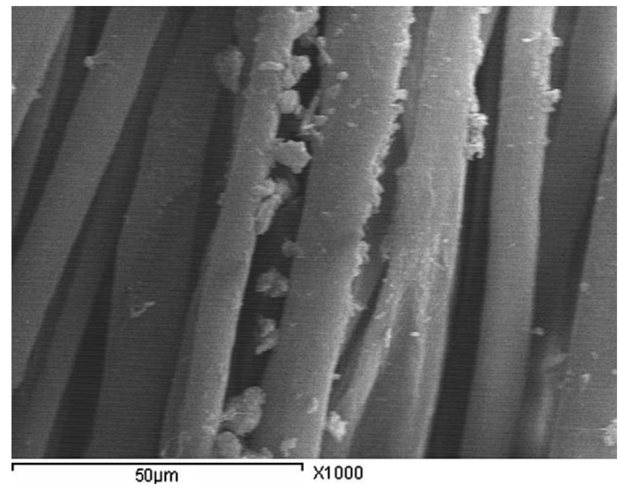


**Fig. 3.** SEM of blank cotton, cotton treated with RCD-g-poly (BuA).

### *3.2. Application of RCD-g-poly (BuA) to cotton fabric*

The RCD molecule, with its many functional hydroxyl groups can be further functionalized with a vinyl monomer such as butyl acrylate as reported elsewhere Hebeish et al. (2009). This graft polymer may be formed reactive preformed polymer (PFP) because it bears monochlorotriazinyl β cyclodextrin moieties. The CD content of these reactive preformed polymers can be adjusted by changing the length of the grafting arms. This synthetic scheme is applicable to a wide variety of vinyl monomers, and it could become a general method for preparing CD-containing preformed polymer of different chemical compositions. Because of the high functionality (–OH groups) of CDs, CD-containing polymers synthesized with a modified interfacial polymerization process can be applied to a cotton fabric to impart special properties. The inclusion of CD's cavity with ZnO nanomaterials has some excellent properties like exceptional mechanical strength, antistatic, antibacterial and UV absorption properties.

Presence of functional (reactive) chlorine atom in RCD-g-poly (BuA) makes it a reactive preformed polymer which can be grafted

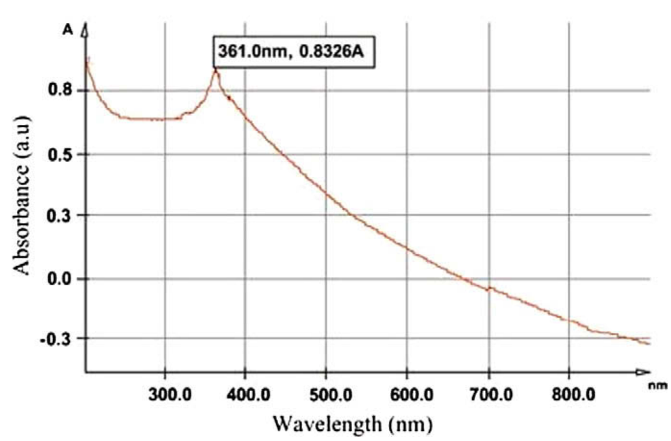


**Fig. 4.** UV visible absorption spectrum of nano-ZnO solution.

to cotton fabric in a way similar to reactive dyes as detailed previously Hebeish et al. (2009). Once grafted, presence of cyclodextrin moiety with its torus – shaped cavity – allows incorporation of guest molecules to impart anti-microbial properties and improve physical properties and air permeability of treated cotton. Presence of poly (BuA) in this reactive preformed polymer is expected, to render the fabric soft. Application to cotton fabric in presence and absence of epichlorohydrin of the preformed polymer under investigation was preformed as per the conventional pad-thermofixation method and the onset of this on nitrogen content, wrinkle-recovery angle, tensile strength and elongation at break were examined. Also examined were the antibacterial and air permeability properties as well as characterization of both ZnO nanoparticles and the PFP-cotton graft copolymers when the latter were prepared containing nano-ZnO and/or epichlorohydrin.

### 3.2.1. Nitrogen content

3Fig. 5 shows the effect of concentration of preformed polymer PFP on the extent of its grafting to cotton (expressed as N%) when grafting was effected in presence and absence of epichlorohydrin. Obviously, the N% increases by increasing the PFP concentration up to 20% where the N% exhibits its highest value. This is observed in presence and absence of epichlorohydrin, but with the certainty that these highest N% values amount to 0.165%N and 0.142%N respectively. That is, the extent of reaction of PFP with cotton is significantly, higher in presence than in absence of epichlorohydrin.

Epichlorohydrin as a conventional crosslinking agent for cotton seems to create bridges through reaction with the hydroxyl groups of the CD moieties of the PFP on one side and with the hydroxyl groups of cotton on the other side. In this way extra amount of PFP are chemically bonded to the cotton fabric. Stated in other words, the PFP is grafted to cotton in absence of epichlorohydrin only through a substitution reaction involving the monochlorotriazinyl group of PFP and the cotton hydroxyls. In presence of epichlorohydrin, on the other hand, extra chemical bonding reveling from reaction of it with the hydroxyl groups of both the PFP and cotton will come to play. Needless to say that such extra chemical bonding will associated with additional amount of PFP chemically attached to cotton. That is why the extent of grafting (expressed as N%) is significantly higher in presence than in absence of epichlorohydrin.

### 3.2.2. Wrinkle-recovery angle

Fig. 6 depicts the dependence of the wrinkle-recovery angle (WRA) on the concentration of reactive preformed polymer PFP when the latter was grafted to cotton fabric in presence and absence of epichlorohydrin. The WRA of the grafted fabrics were measured in warp and weft directions, and the measurements were repeated three times. The results given in Fig. 6 are the sum of the mean values of the wrinkle-recovery angle values in warp and weft directions. It is well-known that the larger the WRA value, the greater crease resistance the fabric exhibits.

It is seen Fig. 6 that grafting of PFP onto cotton fabric improves the WRA being dependent upon the concentration of PFP and whether epichlorohydrin is involved in the grafting mechanism. Without epichlorohydrin the WRA increases from 94° to 150° by increasing PFP concentration from zero to 15%. Involvement of epichlorohydrin increases the WEA from 94° to 210° at the same range of PFP concentration. At higher PFP concentration (i.e. 20%), the WRA tends to decrease but the decrements are not so significant. This is observed in presence and absence of epichlorohydrin.

Enhancement in WRA by only grafting PFP could be associated with fiber stabilization and the soft nature of the graft. Grafting PFP in presence of epichlorohydrin render the cotton fabric more stabilized and assume larger WRA values as result of fiber resilience due to crosslinking.

### 3.2.3. Tensile strength

Fig. 7 illustrate the effect of concentration of reactive preformed polymer PFP (i.e. RCD-*g*-poly(BuA) on the tensile strength of cotton fabric treated there with in presence and absence of epichlorohydrin. The results signify that the tensile strength decreases as the PFP concentration increases within the range studied. They also signify that the decrements in tensile strength are greater in presence than in absence of epichlorohydrin. It is logical that formation

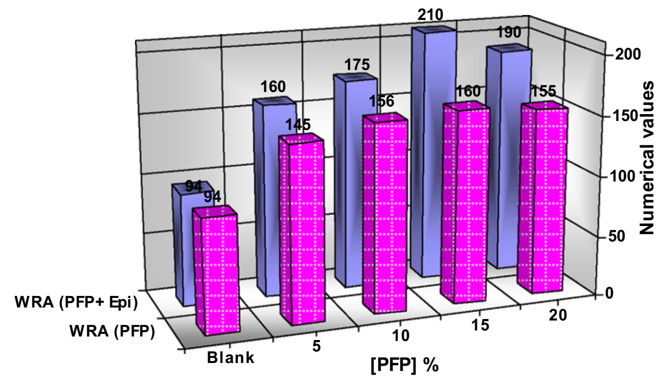


**Fig. 6.** Effect of grafting using different concentration of reactive preformed polymer PFP (RCD-*g*-poly (BuA) in presence and absence of epichlorohydrin on the wrinkle-recovery angle (WRA) of grafted cotton fabric.

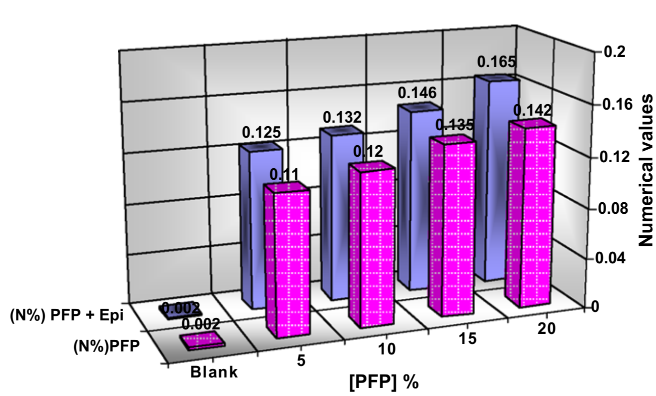


**Fig. 5.** Effect of different concentration of preformed polymer PFP (RCD-*g*-poly (BuA) on the nitrogen% in presence and absence of epichlorohydrin.

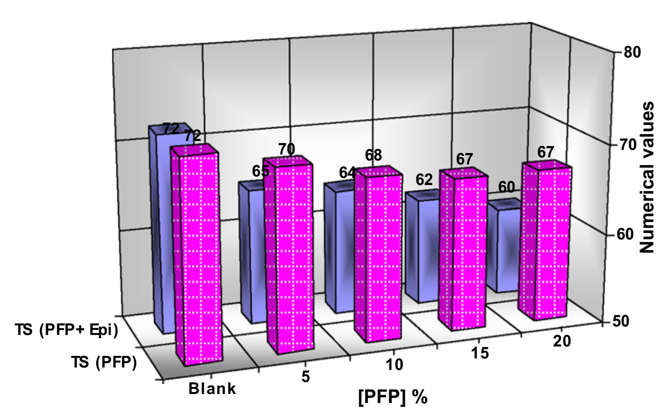


**Fig. 7.** Effect of grafting using different concentration of reactive preformed polymer PFP (RCD-*g*-poly (BuA) in presence and absence of epichlorohydrin on tensile strength of grafted cotton fabric.

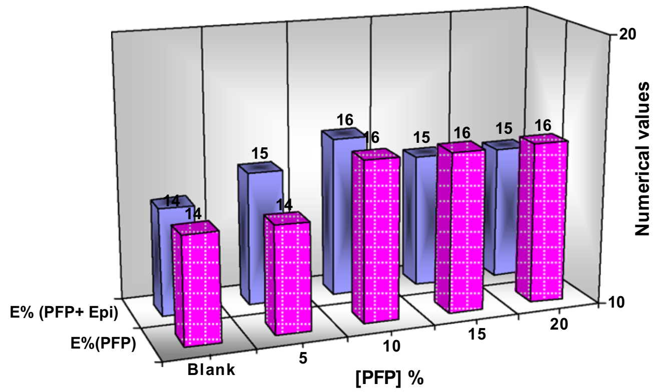


**Fig. 8.** Effect of grafting using different concentrations of reactive preformed polymer PFP (RCD-g-poly (BuA) in presence and absence of epichlorohydrin on elongation at break of grafted cotton fabric.

of higher amounts of graft is accompanied by greater disruption in the molecular structure of cotton thereby decreasing the tensile strength. Besides crosslinks are possible formed when grafting of PFP was carried out in presence of epichlorohydrin and as a consequence, higher decrement in tensile strength is observed. For instance, cotton fabric before grafting exhibited tensile strength of 72 kg this is against tensile strengths of 67 kg and 60 kg grafting using 20% PFP in absence and presence of epichlorohydrin, respectively.

### 3.2.4. Elongation at break

Fig. 8 shows the elongation at break of cotton fabric before and after grafting using different concentrations of PFP in presence and absence of epichlorohydrin. By and large elongation at break displays values which are higher after than before grafting. Grafting using concentrations of PFP ranging from 5% to 20% has practically no effect on elongation at break. Presence of epichlorohydrin during grafting has also practically no effect on elongation at break. For example before grafting the cotton fabric exhibited elongation at break of 14%. This is against a near value of 16% after grafting in presence and absence of epichlorohydrin regardless of PFP concentration. The increase in elongation at break after grafting could be interpreted in terms of structure distribution of the cotton structure by the grafts. However, the soft nature of the poly BuA segment in the graft cannot be ruled.

### 3.3. Antibacterial property

Fig. 9 shows the antibacterial abilities of cotton fabric grafted with the reactive preformed polymer PFP in presence and absence of ZnO nanoparticles. PFP refers to RCD-*g*-poly BuA. It is seen that the antibacterial activity of thus graft fabrics is much higher than that of the unmodified fabric. This finding is brought into focus as a consequence of the existence of different forms of antibacterial agents on the fabrics like *Staphylococcus aureus* (G+ve), *Escherichia coli* (G−ve), *Aspergillus flavus* (fungus) and *Candida* albicans (fungus) according to the diffusion disc method Grayer and Harbone (1994), Irob, Moo-Young, and Anderson (1996).

For the PFP graft copolymers, the antibacterial agents in particular ZnO nanoparticles are included in the torus shaped cavities of CD of the graft, while absorbed physically on the unmodified cotton fabrics. This renders the antibacterial agents easy to wash from the fabrics, and concludes that of the antibacterial property of the PFP-cotton graft copolymers is much better than the unmodified fabrics. Accordingly, the antibacterial abilities of cotton fabrics grafted with PFP (RCD-*g*-poly BuA) in presence of ZnO nanoparticles retained more than 70% of their values even after 20 times washing. In contrast, the antibacterial activity was almost lost in case of the unmodified cotton fabric. This phenomenon is created from the different existing forms of antibacterial agents on the fabrics. With PFP (RCD-*g*-poly BuA) in presence of ZnO, the nano particles are included in the cavities of RCD, while ZnO nano particles absorbed physically in case of the untreated fabrics. That is why the antibacterial agents on the latter fabrics are easy to wash. Based on this, the durability of antibacterial property of the cotton fabrics-containing PFP (RCD-*g*-poly BuA) in presence of ZnO is much better than the untreated fabric and still kept even after 20 times washing.

### 3.4. Air permeability

Table 1 shows the air permeability of cotton fabrics grafted with the PFP in presence of either bulk-ZnO or nano-ZnO. Whereas Table 2 shows the air permeability of the cotton fabric when it was grafted using different concentrations of PFP in presence of ZnO nanoparticles, with or without epichlorohydrin.

Results of Table 1 make it evident that inclusion of ZnO nanoparticles in chemical coating of cotton fabric during its grafting with the PFP causes substantial increment in the air permeability of the grafted fabric. The same situation is encountered when the

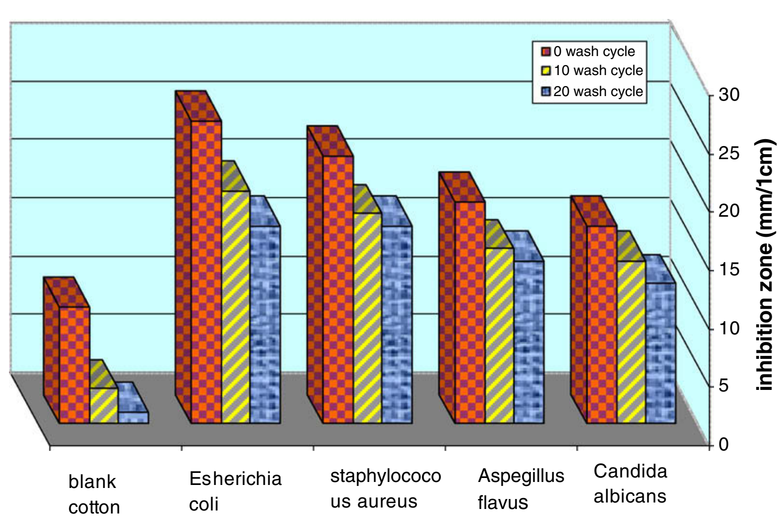


**Fig. 9.** Durability of the antibacterial abilities of cotton graft with PFP and ZnO nano particles and unmodified fabric with ZnO nano particles).

**Table 1**
Effect of ZnO form on air permeability of cotton fabric grafted with PFP along with ZnO.

| Air permeability [m/(kPa.s)] | |
|---|---|
| Control | 14.60 |
| Bulk-ZnO | 15.27 |
| Nano-ZnO | 19.02 |

**Table 2**
Effect of concentration of PFP on air permeability of cotton fabric grafted with PFP along with nano-ZnO in presence and absence of epichlorohydrin.

| | Air permeability [m/(kPa.s)] | |
|---|---|---|
| [PFP] | Absence of epichlorohydrin | Presence of epichlorohydrin |
| Control | 14.60 | 14.60 |
| 5 | 16.88 | 15.27 |
| 10 | 17.20 | 16.02 |
| 15 | 18 | 17.3 |
| 20 | 18.5 | 17.9 |

nano-ZnO was replaced by bulk-ZnO but with the certainty that the air permeability is much higher with ZnO nanoparticles; a point which could be ascribed to the uniform and very thin distribution of the nano-ZnO as compared with bulk-ZnO Yadav et al. (2006).

Results of Table 2 disclose that the air permeability increases by increasing the concentration of the PFP when the latter containing ZnO nanoparticles were grafted to cotton fabric in presence and absence of epichlorohydrin. The results disclose that air permeability is higher in absence than in presence of epichlorohydrin.

Enhancement in air permeability by increasing PFP concentrations could be associated with formation of strong and uniform graft coating at higher concentrations of PFP in which nano-ZnO is very finely dispersed. On the other hand, the adverse effect of epichlorohydrin on the air permeability is most probably owing to certain of crosslinks in the graft coating as well as in the molecular structure of cotton fabric; epichlorohydrin is a well-known conventional crosslinking agent.

Hence besides bridging the PFP to cotton, epichlorohydrin may produce crosslinked-grafted cotton thereby detracting from the function of the graft in enhancing the air permeability. At any event, however, grafting of PFP along with ZnO nanoparticles onto cotton fabric improves the air permeability of the latter. Air permeability is an important property as it connected with garments breathability and eventually comfortability.

## 4. Conclusion

The results reported in this study demonstrate that the impact of the grafting of reactive preformed polymer PFP on performance of cotton fabrics was studied. The results concluded that the extent of grafting of PFP in presence of epichlorohydrin onto cotton, expressed as %N, amounts to 0.125%N and 0.165%N upon using 5% and 20% PFP respectively. Wrinkle-recovery angle exhibits a value of 210° after grafting compared to 94° before grafting. Tensile strength decreases from 72 kg for untreated fabric to 60 kg after grafting 20% PFP in presence of epichlorohydrin. Grafting to cotton fabrics of PFP in presence of ZnO nano particles and/or epichlorohydrin impart antibacterial activity for 20 time cyclic wash. Also, air permeability of the nano-ZnO coated fabrics was significantly higher than the control, thus increased breathability. Nano-ZnO coating on cotton fabrics resulted in uniform and very thin coating due to nano-size and hence reduced friction in comparison with its bulk counterpart. Thus, the nano-ZnO coated cotton fabric is proved to have better strength properties, air permeability and antibacterial properties of treated cotton fabric. Also the treated cotton fabric was characterized using IR spectral analysis and scanning electron microscopy, the ZnO nanoparticles were analyzed through electron microscopy. From that we can concluded that the chemical grafting of cyclodextrins onto cotton fibers represents a useful strategy for the production of antibacterial and comfortable clothing.

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