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RESEARCH ARTICLE

One-Step Process for Dual Antimicrobial and Easy Care Finishing of Cotton Fabric

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Abstract

The technical feasibility of incorporating of dimethyltetradecyl-[trimethyl silane-propyl] ammonium chloride (DTPAC), (commercially available as Sanitize® T99-19) and poly (vinylpyrrolidone) (PVP) as antimicrobial agents in easy care finishing formulation along with glyoxal was investigated. Optimum conditions for a one-step process, like reagent concentrations, fixation temperature and time were investigated. The treated fabrics were monitored for physico-chemical properties, antimicrobial and easy care properties. The antimicrobial properties of the treated cotton fabric marginally increase as the concentration of PVP increase. This is indicated by the increment in the inhibition zone from 22 mm to 26 mm against S. aurous up to 30 g/L PVP to the treatment path. Although, the antimicrobial properties imparted to the treated cotton fabric is obtained from Sanitize® incorporated with addition of PVP. The results also show that, addition of PVP increase the crease recovery angle (CRA) of the treated fabric increased. Pre-carboxymethylation of cotton fabric increase both CRA and tensile strength. FT-IR and SEM were carried out to investigate the chemical and morphological changes of cotton cellulose after crosslinking.

INTRODUCTION

Textiles have long been recognized as media to support the growth of microorganisms such as bacteria and fungi. These microorganisms are found almost everywhere in the environment and can multiply quickly when basic requirements such as moisture, nutrients, and temperature are convenient **[1]**. Proteins in keratinous fibers and carbohydrate in cotton act as nutrients and energy sources under certain conditions. Soil, dust, solutes from sweat and some textiles finishes can also be nutrient sources for microorganism **[1]**.

The growth of microorganisms on textiles causes unwanted affects not only to the textiles itself but also to the wearer. These effects include the generation of unpleasant odor, stain and discoloration of colored fabric, a reduction in fabric strength. For these reasons, it is highly desirable that the growth of microbes on the textiles should prohibit or minimize during their use or storage **[2 - 4]**. Recently, consumer's demand for hygienic clothes and active wear has created a substantial market for antimicrobial textiles products. Consequently, it is highly important to treat cotton textiles with antibacterial finishes especially where the probability of microbial growth is high, the aesthetic and hygiene is important, and the safety is necessary.

Based on their nature and mode of action antibacterial agents can be divided into two types: leaching and non leaching types **[4]**. Leaching type agents are those released from the textiles substance then gets close to microbes and attacks them. The activity decreases over time and fades away as a result of using up microbes. A non leaching type agents form strong bonds to the textile substance and attacks microbes only by direct contact. The activity remains unchanged unless the bonds are broken.

Natural fibers exhibit good properties such as moisture absorption, comfort and soft handling, but they also display some undesirable properties such as poor dimensional stability and crease resistance in laundry and wear.

Textile producers can alter the molecular structure of the fibers to produce a fabric for permanent press or "wash and wear" garments **[5]**. The most effective crosslinking agent for durable press (DP) of cellulose fibers are formaldehyde adducts of urea, which unfortunately releases formaldehyde during production and wear in clothes so treated. The release of formaldehyde from DP treated fabrics is a problem for human carcinogen, allergic and other issues **[6, 7]**. Efforts have been made to develop non-formaldehyde crosslinking treatment as safe alternative. The latter includes mainly ionic crosslinking, glyoxal and polycarboxylic acid **[8]**.

Glyoxal has been advocated as a non-formaldehyde finish because of its low cost ready availability, high functionality, and high solubility in water. The dialdehyde groups in glyoxal molecule make it of considerable interest in the formation of formaldehyde free crosslinked cellulose. The formation of crosslinkers in cotton by glyoxal occurs through hemiacetal and /or bis-acetal **[8]**.

Poly(vinylpyrrolidone) PVP is synthetic nontoxic water-soluble polymer commonly used in a wide range of application including pharmaceutical applications. PVP polymer film formers protective colloid , dye-receptive agent, binder stabilizer and complexing agents.

One of the most recent commercial reactant antibacterial agent added to easy care finishing formulation of cotton fabric is sanitized T99-19. The active ingredient of Sanitize® is dimethyltetradecyl-[trimethyl silane-propyl] ammonium chloride (DTPAC) and having the following structure **[9].**

$$[(CH_3O)_3\text{-Si-}CH_2\text{-}CH_2\text{-}CH_2\text{-}N^+ (CH_2)_2\text{-}C_{18}]\text{-}H_{37}Cl^-$$

In commercial use, easy-care and durable press finishes are frequently combined with other finishes to provide additional properties such as water and oil repellency, flame retardancy, antimicrobial properties and the like. Often the combination of another finish with the cellulose crosslinking finish will result in a more durable effect from the first finish.

So far, there is no work in the literature describing the antimicrobial activity of DTPAC when it incorporated in the easy care finishing formulation based on glyoxal as non-formaldehyde finishing agent for cotton.

With the above in mind, the present work is undertaken to (a) investigate the technical feasibility of incorporating poly(vinylpyrrolidone) (PVP) and DTPAC (commercially available as sanitized T99-19) as antimicrobial agents in the easy care finishing formulation that based on glyoxal for cotton fabrics. (b) Establishment the optimum conditions for application of these compounds in a one-step process, like reagent concentrations, fixation temperature and time. The treated fabrics were monitored for physico-chemical properties, antimicrobial activities as well as easy care properties.

2 Experimental:

2.1 Materials

Mill desized, scoured, and bleached print cloth, plain weave (102 g/m^2) was supplied by Miser Company for Spinning and Weaving, Mehalla El-Kubra, Egypt.

Poly(vinylpyrrolidone) (PVP) with molecular weight 10000 Dalton was supplied by sigma-Aldrich. Glyoxal of 45% solution (Merk), and aluminum sulphate were of laboratory grade chemical. Sanitize® T99-19 (antibacterial agent based on silicon-functional teralkyl ammonium compound in high boiling glycoether) supplied from Sanitized AG, Switzerland.

2.2 Functional Finishing Formulation:

Bleached cotton fabrics were padded twice in aqueous formulation containing Sanitize® (0 - 40 g/L), glyoxal (0 – 50 g/L), aluminum sulphate (0 -10 g/L) and PVP (0 – 60 g/L). The fabrics were then squeezed to a wet pick up of 100% using a laboratory padding machine. The treated samples were dried at 80^o C for 5 min then cured at 140°C for 3 min. Finally, the samples were thoroughly rinsed twice at 50^{o}C for 15 min, and then dried at ambient conditions.

2.3 Testing and Analysis:

- **Dry crease recovery angle (CRA)** was measured according to AATCC standard method 66-1996.
- **Tensile strength and elongation at break** were determined by the strip method according to ASTM, Standard Test Method ''Breaking Load and Elongation of Textile Fabric", D-1682-94 **(10)**.
- **Antibacterial activities** of the fabric sample were evaluated against *S. aureus* (SA) (as gram-positive bacteria) and *E. coli* (EC) (as gram-negative bacteria) according to Agar Diffusion Method (AATCC Test Method 100-2004) **[11]**. The diameter of inhibition zone (mm/1 cm sample) was measured for each treated sample.
- **Air permeability (AP)** describes the property of fabric to let through air in outdoor clothing it is important that the air permeability is as low as possible because it should function as wind protection it was measured according to ASTM (D737 -96) by Toyoseiki Tester.
- **Wettability**: Water absorbency was monitored according to an AATCC Test Method 39-1980 (Evaluation of Wettability) (AATCC, 1980). The time (in seconds) between the contact of water drop with the fabric and the disappearance of the water drop into the fabric called wetting time. The shorter the wetting time, the better the fabric absorbency.
- **Whiteness** of the fabric sample expressed as whiteness index was measured using Color Eye-3100 spectrophotometer supplied with SDL international according to reported method **[12]**.

3. Results and Discussion:

3.1. Crosslinking of cotton fabric with glyoxal - Tentative Mechanism:

Glyoxal reacts with cotton fabric at 140^{o}C and confer the fabric durable press properties. Because glyoxal is bifunctional aldehyde, the formation of bis-acetals or hemiacetals can be taken into consideration **[13]** as shown in scheme (1).

$$O{=}CH\text{-}CH{=}O \;+\; 2\ Cell\text{-}OH \longrightarrow O{=}HC\text{-}CH(O\text{-}Cell)_2 \;+ H_2O \quad .....(1)$$

$$O{=}HC\text{-}CH(O\text{-}cell)_2 \;+\; 2\ Cell\text{-}OH \longrightarrow (Cell\text{-}O)_2\text{-}HC\text{-}CH(O\text{-}Cell)_2 \;+\; H_2O........(2)$$

Scheme 1: Crosslinking of cotton cellulose with glyoxal

DTPAC (Sanitize® T99-19) react with cotton cellulose in acidic medium at high temperature forming a covalent bond (equation 3). Ionic interaction between DTPAC and negatively charged cotton cellulose is also likelihood (equation 4). Moreover it can be also fixed by an ion-pair bond (electrostatic attraction between the negatively charged fiber in aqueous solution and the positively charged quaternary ammonium salt **[14]**.

$$\mathbf{Cell\text{-}OH + (CH_3O)_3Si\text{-}CH_2\text{-}CH_2\text{-}CH_2\text{-}N^+(CH_2)_2\text{-}C_{18}\text{-}H_{37}\ .\ Cl^- \longrightarrow}$$

$$\mathbf{Cell\text{-}O\text{-}Si\text{-}(CH_3O)_2\text{-}CH_2\text{-}CH_2\text{-}CH_2\text{-}N^+(CH_2)_2\text{-}C_{18}\text{-}H_{37}\ Cl^- + CH_3OH}\ ....\ \mathbf{(3)}$$

$$\mathbf{Cell\text{-}O\text{-}Si\text{-}(CH_3O)_2\text{-}CH_2\text{-}CH_2\text{-}CH_2\text{-}N^+[(CH_2)_2\text{-}C_{18}\text{-}H_{37}]\ \ {}^-O\ Cell}\ ....\ \mathbf{(4)}$$

This study aimed essentially at imparting cotton fabric a dual function namely, antimicrobial and easy care properties in one-step process. The treatment sequence and optimum conditions that achieved better performance properties to the fabric were investigated.

3.2. Factors affecting easy care finishing of cotton fabric with glyoxal in presence of PVP and Sanitize®

3.2.1 Effect of PVP concentration:

Bleached print clothes were treated with an aqueous solution containing glyoxal and Sanitize® along with different concentration of PVP. The fabrics were monitored for antimicrobial properties, tensile strength, and elongation at break, wettability, crease recovery angle air permeability, and nitrogen content. Results obtained are set out in table (I).
Results of table (I) depict:

1) The antimicrobial properties of the treated cotton fabric marginally increase as the concentration of PVP increase **(15)**. This is indicated by the increment in the inhibition zone from 22 mm to 26 mm against ***S. aurous*** up to 30 g/L PVP to the treatment path. Although, the antimicrobial properties imparted to the treated cotton fabric is obtained from Sanitize® incorporated with addition of PVP. Moreover, crosslinking of cotton cellulose with glyoxal using pad dry cure techniques in presence of PVP and Sanitize® causes an improvement in the antibacterial properties of the treated cotton fabric. It is also evident that the fabrics finished in the formulation containing 3% PVP, have the highest antibacterial activity, which may be attributed to the creation of active sites bound to fabric surface, resulting due to the ring opening during the fabric treatment conditions.

Table I: Effect of PVP concentration on some physico-chemical and antibacterial properties of cotton fabric treated with glyoxal:

PVP conc. (g/L)	I.Z (mm)		T.S (kg.f)	E (%)	CRA (Deg)	Wett (sec)	N-content (%)	AP Cm^3/ Cm^2.sec.
	S. aurous	*E. coli*						
Blank	**0.0**	**0.0**	**70**	**18**	**95**	**2**	**0.00**	**28.0**
0	**22**	**21**	**59**	**17**	**211**	**2**	**0.26**	**28.0**
20	**22**	**21**	**57**	**17**	**222**	**2**	**0.27**	**27.3**
30	**26**	**24**	**53**	**17**	**245**	**2**	**0.31**	**27.0**
40	**24**	**21**	**53**	**16**	**242**	**2**	**0.39**	**26.3**
60	**24**	**21**	**51**	**16**	**240**	**3**	**0.44**	**26.2**

Conditions used: Glyoxal conc., 30g/L; $Al_2(SO_4)_3$, 4 g/L; Sanitize®, 20 g/L; drying at 80 °C for time 5 min; curing at 140 °C for 3 min.
Antimicrobial properties is evaluated by measuring the inhibition zone against G -ve (*E-coli*); G+ ve (*S. aurous*); (T.S) tensile strength; (E) elongation at break; (CRA) crease recovery angle; (AP) air permeability; (WA) wettability; and (N%) nitrogen percentage.

2) The tensile strength of the treated fabrics slightly decreased as the concentration of PVP increased whereas the elongation at break is kept constant.
3) Fabric wettability is kept approximately unaltered upon increase the PVP concentration. Whereas the fabric nitrogen content increases.
4) Air permeability of the treated cotton fabrics slightly decreased upon increase the concentration of PVP. This may be due that PVP polymer macromolecule covering the interspacing between the yarns in the weaving construction of the cotton fabrics. However, the decrement in air permeability considers marginally and could be ignored.
5) Salient properties observed in Table (I) is the enhancement in the crease recovery angle of the treated fabric upon increase in PVP concentration. Crease recovery angle (CRA) of the treated cotton fabrics increased from 95° with blank to 211° after crosslinking with glyoxal and in absence of PVP. CRA is further increased upon addition of PVP to the finishing formulation and reached its maximum value at 245° upon addition of 30 g/L PVP. Further increase in PVP concentration slightly decreased CRA of the treated cotton fabrics. Enhancement of CRA of cotton fabrics by increasing PVP concentration may be attributed to the ionic interaction between positive charge Sanitized® and PVP under the conditions used **[16]**. Results of table (I) make it clear that addition of PVP to the finishing formulation containing Sanitize® and glyoxal has a favor effect on easy care properties of the treated cotton fabrics without adversely affect the antimicrobial and other properties. Obviously, addition of 30 g/L, PVP represents the optimal concentration.

3.2.2. Effect of Sanitize® concentration

Table II summarize the properties of cotton fabric easy care finished with glyoxal in presence of PVP and different concentration from Sanitize®. Results of table (II) depict the following:

1) Increasing Sanitize® concentration up to 20 g/L enhances CRA. Further increase in Sanitize® concentration has marginal effect on the CRA of the treated cotton fabrics, whereas fabric wettability, air permeability, tensile strength and elongation at break remain approximately unaltered.
2) The antimicrobial activity of the treated cotton fabrics increased as the concentration of Sanitize® in the finishing formulation increased. Higher antimicrobial activities imparted to the cotton fabrics against *S. aurous* and *E. coli* is observed when more than 20 g/L Sanitize® is used. Further increase in Sanitize® concentration exerts no effect on the antimicrobial activity of the treated cotton fabrics.

3.2.3. Effect of glyoxal and aluminum sulfate concentrations on some physico-chemical properties of cotton fabric treated with PVP and Sanitize®

Optimum conditions obtained from tables I and II was used in design reaction scheme between cotton fabric and glyoxal in presence of PVP and Sanitize® under different concentration from glyoxal and aluminum sulfate. The fabrics were monitored for antimicrobial and easy care properties. Results obtained are set out in Table III.

It is evident from table III that, at the same glyoxal concentration, increasing the concentration of aluminum sulfate enhances the CRA up to 4 g/L. Further increase in aluminum sulfate concentration has little effect on CRA of the treated cotton fabric. It is further noticed that; the tensile strength and elongation decreased as the concentration of aluminum sulfate increased. Increasing CRA upon increased aluminum sulfate concentration is attributed to the catalytic effect of aluminum sulfate on catalyzing crosslinking of glyoxal with cellulose hydroxyl groups through the formation of temporary acidity at elevated temperature.

Table II: Effect of Sanitize® concentration on some physico-chemical and antibacterial properties of cotton fabric treated with glyoxal and PVP

Sanitize® conc. (g/L)	I.Z (mm)		T.S (kg.f)	E (%)	CRA (Deg)	Wett. (sec)	AP $Cm^3/Cm^2.sec$
	S. aurous	*E. coli*					
Blank	0	0	70	18	95	2	26.5
0	11	9	59	17	238	2	28.3
10	20	21	59	17	240	2	28.0
20	26	25	58	16	247	3	26.5
30	25	24	58	16	245	3	25.6
40	26	25	58	16	246	2	25.0
50	24	24	57	16	245	3	27.6

Antimicrobial properties, G-ve (gram negative bacteria *E-coli*); G+ ve (gram positive bacteria – *St. aurous*); (T.S) tensile strength; (E) elongation at break; (CRA) crease recovery angle; (AP) air permeability; (WA) wettability; and (N%) nitrogen percentage.

Conditions used: Glyoxal, 30 g/L; $Al_2(SO4)_3$, 4 g/L; PVP, 30 g/L; drying at 80 °C for time 5 min; curing at 140 °C for 3 min.

Table III: Effect of glyoxal and aluminum sulfate concentrations on some physico-chemical and antibacterial properties of cotton fabric treated with PVP and sanitize®

$Al_2(SO_4)_3$ conc. (g/L)	Glyoxal conc. (g/L)											
	10				20				30			
	I.Z	T.S	CRA	AP	I.Z	T.S	CRA	AP	I.Z	T.S	CRA	AP
0	0 (0)*	69.9 (18)**	95	26.4	0 (0)	69.9 (18)	95	26.4	0 (0)	69.9 (18)	95	26.4
2	18	58	212	29	22	51	218	27	24	52	220	26

	(18)	(17)			(20)	(17)			(23)	(15)		
4	19 (19)	48 (15)	230	27	22 (20)	48.2 (15)	239	29	26 (25)	50 (15)	245	27
6	21 (20)	48 (15)	232	27	22 (20)	46 (14)	240	26	25 (25)	41 (14)	246	26
8	20 (22)	42 (14)	234	27	22 (20)	41 (14)	245	25	26 (24)	40 (13)	248	26

*** Values in brackets represent the antimicrobial properties of the cotton fabrics against gram negative bacteria *(E. Coli)*.**
** **Values in brackets represent the elongation at break of the cotton fabrics**

(A) Antimicrobial activity against G+ve(*S. aureus*.) Values in brackets represent the antimicrobial properties of the treated cotton fabrics against G-ve *(E. Coli)* bacteria. (T.S) tensile strength; (E) elongation at break; (CRA) crease recovery angle; (AP) air permeability.

Conditions used: PVP, 30 g/L; Sanitize®, 20 g/L, drying at 80 °C for time 5 min; curing at 140 °C for 3 min.
However, higher catalyst concentration exerts, fabric tendering due to hydrolysis of cotton cellulose under the effect of heat and acidic conditions during the curing process, thus providing depolymerization of cotton cellulose at higher reaction temperature **or** catalyst concentration **[17]**. This would account for the slight decrement in fabric tensile strength as the concentration of the acid catalyst increases. On the other hand the decrement in elongation at break of the treated cotton fabric is attributed to rigidity conferred on the cotton structure by crosslinking **[12]**.

3.3. Effect of pre-carboxymethylation of cotton fabric (CMC)

Cotton fabrics were subjected to partial carboxymethylation followed by crosslinking with glyoxal in presence of PVP and Sanitize®. Partial carboxymethylation of cotton fabrics enhance the anionic character of the cotton fabrics and consequently, it facilitates the ionic interaction of PVP, Sanitize® with CMC.

Table IV shows the effect of pre-carboxymethylation of cotton fabric on its performance properties after crosslinking with glyoxal in presence of Sanitize® and PVP. It is seen from table VI that, the antimicrobial activity of both CMC and cotton fabric remains the same. This is evidenced by approximately equal inhibition zone of the treated cotton fabric and CMC fabric towards *S. aureus* and *E. coli*. However, both CRA and T.S are slightly higher for the CMC fabrics compared with cotton fabrics. This could be attributed to the extra ionic interaction in case of CMC **[14]**.

3.4 FTIR analysis

Figures 1-6 show FTIR analysis of untreated cotton fabrics, CMC fabric, PVP, Sanitize®, cotton fabrics crosslinked with glyoxal in presence of PVP and Sanitize®, fabrics crosslinked with glyoxal in presence of PVP and Sanitize®, respectively. Peaks assignment of each figure is explained in Table V-VII. It could be concluded from Figures 1-6 and Tables V-VII that; the characteristic peak of PVP and Sanitize® are observed in FTIR of cotton and CMC fabrics after crosslinking with glyoxal in presence of PVP and Sanitize®.

Table IV: Effect of pre-carboxymethylation of cotton fabric on its performance properties afttreatment with glyoxal in the presence of with Sanitiz and PVP

Substrate	I. Z (mm)		CRA (deg)	T.S (kg.f)	Elongation at break (%)
	S. aureus	*E. Coli*			
Cotton	**26**	**25**	**245**	**50**	**17**
CMC	**27**	**26**	**255**	**60**	**21**

(I.Z) Inhibition zone, (T.S) tensile strength; (CRA) crease recovery angle;

Treatment conditions: PVP, 30 g/L; Sanitize®, 20 g/L, glyoxal, 30 g/L; $Al_2(SO4)_3$, 4 g/L, drying at 80 °C for time 5 min; curing at 140 °C for 3 min.

Table V: Peaks assignment of cotton and CMC fabrics

Cotton (Fig. 1)			CMC (Fig. 2)		
No.	**Peak cm^{-1}**	**Assignment**	**No.**	**Peak cm^{-1}**	**Assignment**
---	---	------	1	3382	Carboxylic –OH stretching
1	3417	Alcoholic –OH stretching			
2	2901	Aliphatic $-CH_2$ stretching	2	2902	Aliphatic $-CH_2$ stretching
	-----	-----	4	1731	-C=O stretching of carboxylic acid
5	1640	Absorbed water and hydrogen bond	5	1638	Carboxylate anion- symmetric stretching
	----	-----			
6	1428	Aliphatic $–CH_2$ bending	6	1425	Aliphatic $–CH_2$ bending
7	1367	-CH bending (deformation stretching)	7	1373	-CH bending (deformation stretching)
8	1338	-OH in plan bending	8	1338	-OH in plan bending
9	1161	- C-O-C - asymmetric bridge stretching	9	1156	- C-O-C- asymmetric bridge stretching
10	1110	- C-O-H bending of secondary alcoholic	10	1113	- C-O-H bending of secondary alcoholic
11	1057	Asymmetric in plan ring stretching	11	1058	Asymmetric in plan ring stretching
14	1033	-C-O stretching	14	1030	-C-O stretching
15	902	Asymmetric out-of-phase ring stretch $–C_1$-O-C_4 β – glucosidic bond	15	896	Asymmetric out-of-phase ring stretch – C_1-O-C_4 β – glucosidic bond
	---	------	16	707	-O-C=O bending in carboxylic acid

Table VI: Peaks assignment of PVP and and Santitize®

PVP (Figure 3)			Santitize (Figure 4)		
No	**Peak Cm^{-1}**	**Assignment**	**No**	**Peak Cm^{-1}**	**Assignment**
1	3442	-NH and $-^{\oplus}NH_3$ stretching	1	3400	-NH and $-^{\oplus}NH_3$ stretching
2	2955	Aliphatic $-CH_2$ stretching	2	2924	Aliphatic $-CH_2$ stretching
4	1652	-C=O stretching	7	1644	$-^{\oplus}NH_3$ deformation
5	1495	–C–N– stretching vibration	8	1462	–C–N– stretching vibration
6	1462	–C–N– bending vibration	11	1246	Si-CH_3 symmetric deformation

Table VII: Peaks assignment of cotton fabrics and CMC fabric crosslinked with glyoxal in presence of PVP and Santitize®

Cotton fabrics crosslinked with glyoxal in presence of PVP and Santitize® (Figure 3)			CMC fabrics crosslinked with glyoxal in presence of PVP and Santitize® (Figure 4)		
No	**Peak Cm^{-1}**	**Assignment**	**No**	**Peak Cm^{-1}**	**Assignment**
1	3415	-NH and $-^{\oplus}NH_3$ stretching	1	3411	-NH and $-^{\oplus}NH_3$ stretching
4	1648	$-^{\oplus}NH_3$ deformation	4	1652	$-^{\oplus}NH_3$ deformation
8	1283	Si-CH_3 in Sanitize	8	1283	Si-CH_3 Sanitize
11	1058	Si-O-Si stretching	12	1057	Si-O-Si stretching

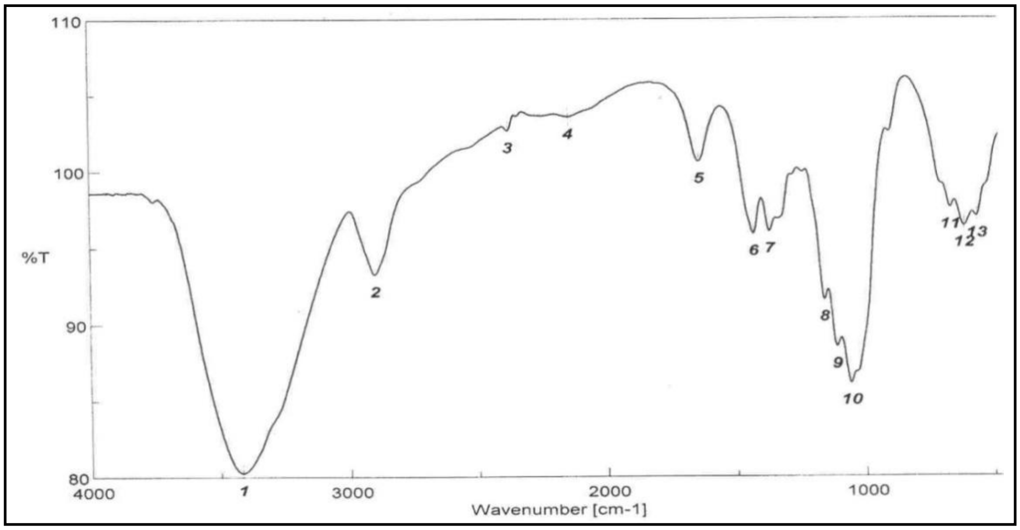


Fig. 1: FTIR of untreated cotton fabrics

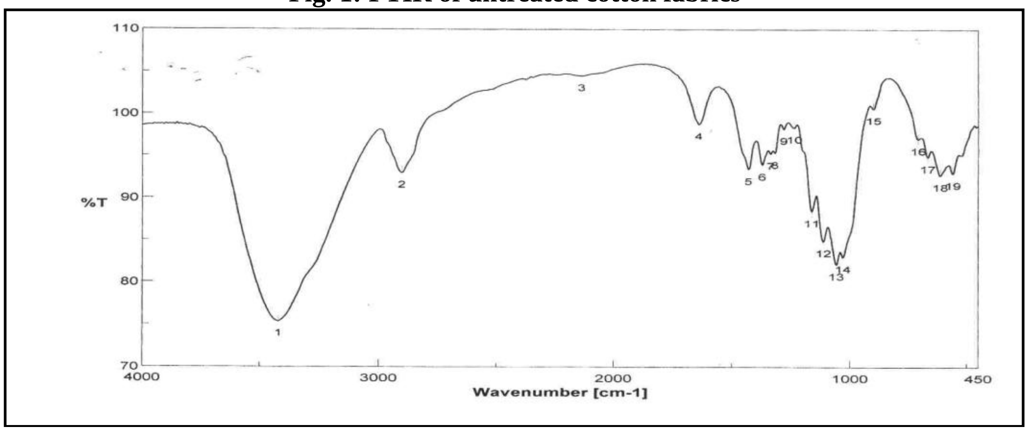


Fig 2: FTIR of CMC fabric

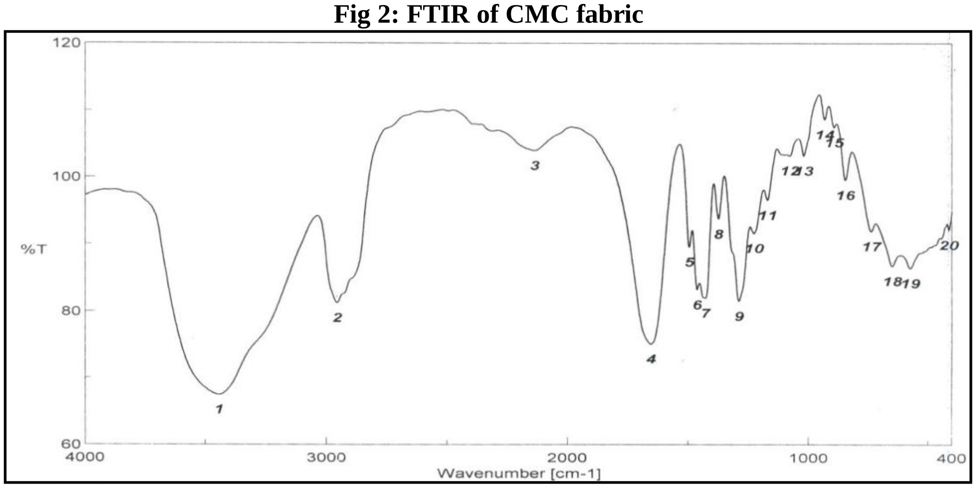


Fig. 3 : FTIR of PVP

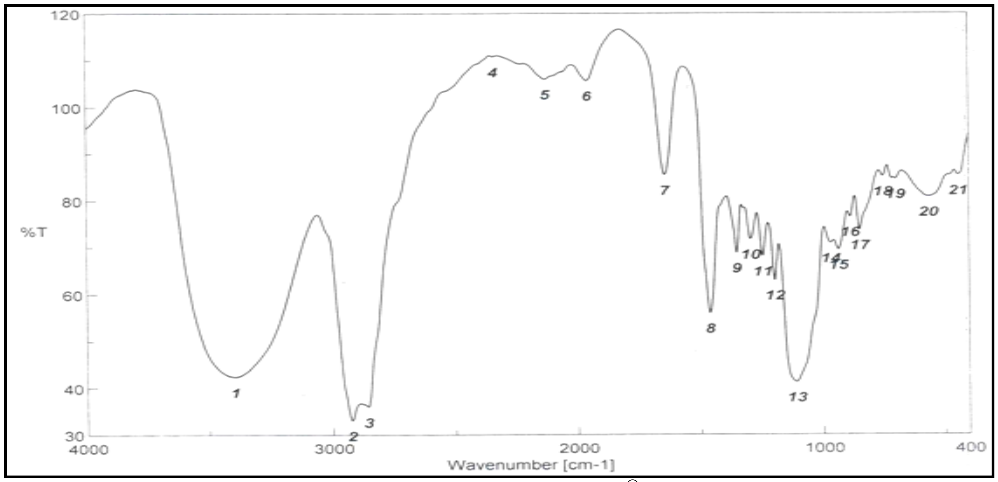


Fig. 4 : FTIR of Santize®

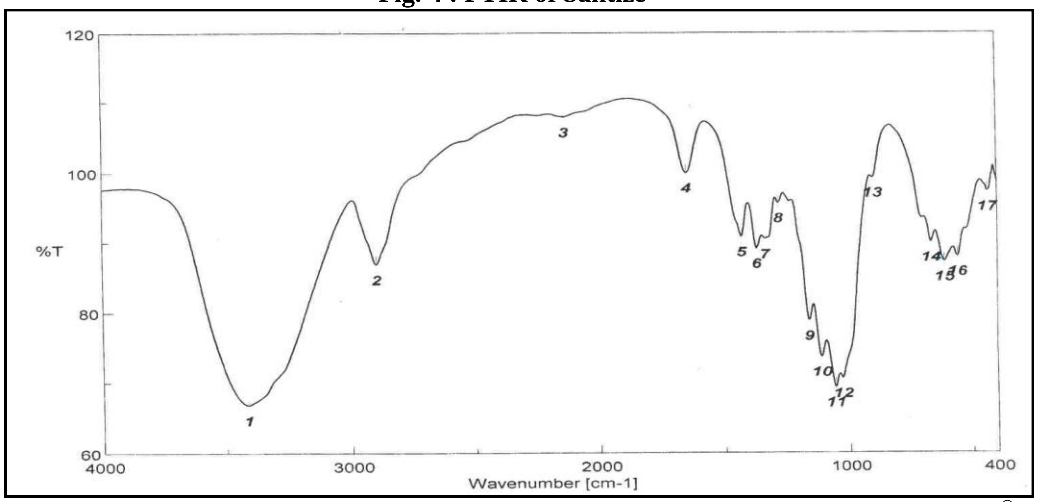


Fig. 5: FTIR of cotton fabrics crosslinked with glyoxal in presence of PVP and Santize®

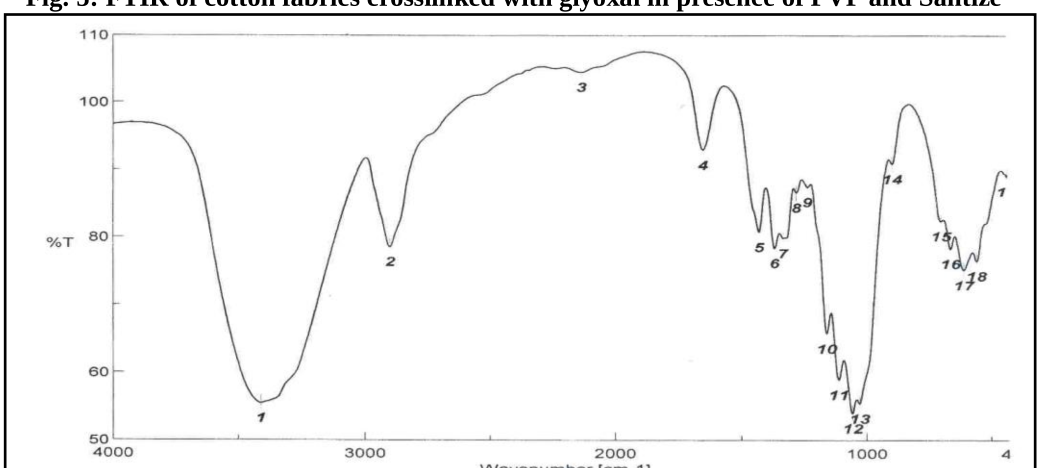


Fig. 6: FTIR of CMC fabrics crosslinked with glyoxal in presence of PVP and Sanitize®

3.5 Scanning electron micrograph (SEM)

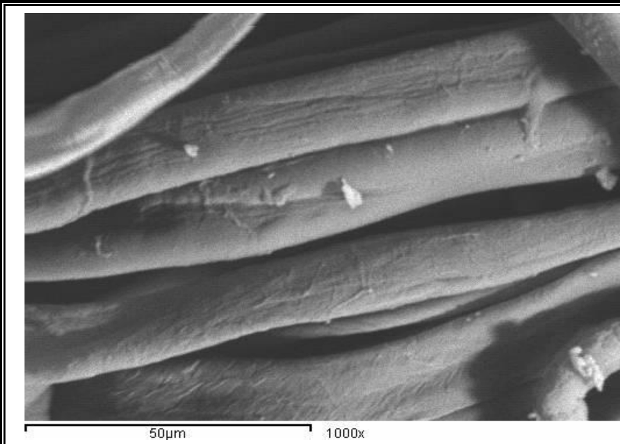
In this study, we investigated the effects of easy care finishing of cotton or CMC fabrics with glyoxal in presence of PVP and Sanitize® on the change in the fabrics morphology. Examination of bleached cotton fabric micrograph (Fig. 7A)

Shows typical fibers with twisted, wrinkled and harsh surfaces that are produced when fibers from the boll dehydrate upon boll opening **[18]**.

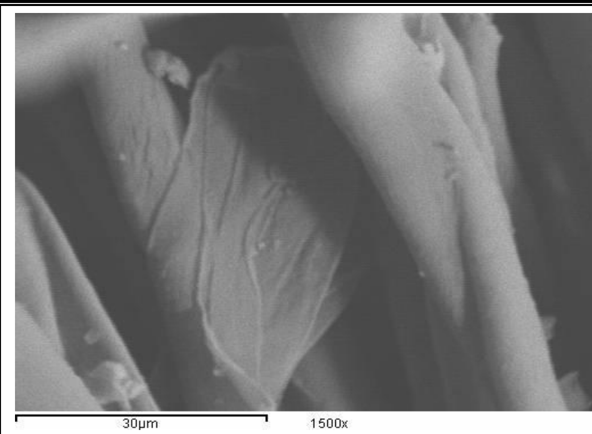
Figure 7 (B) shows scanning electron micrograph of cotton fabric after easy care finished with glyoxal in presence of PVP and Sanitize®. It is evident that the fibers shows smooth ridges, concave grooves, smooth surface, PVP homo-polymer and Sanitized form thin film on the fabric surface and between inter fibres.

Figure 7 (C) shows SEM of CMC. It is evident that, the fibers are swelled and show flat ridges, concave grooves, few protruding fibrils and harsh fiber surface. The change in the structure of fibers surface is comparatively notable. After carboxymethylation treatment the surface of cellulose fibers become rough, loose and striated. The external fibrillation of modified cellulose fibers was also exfoliated partially. In addition, the surface of cellulose fibers shows some helical ditches orientated along the direction of micro-fibril. Fiber swelling is due to the higher sodium hydroxide concentration used during the carboxymethylation reaction.

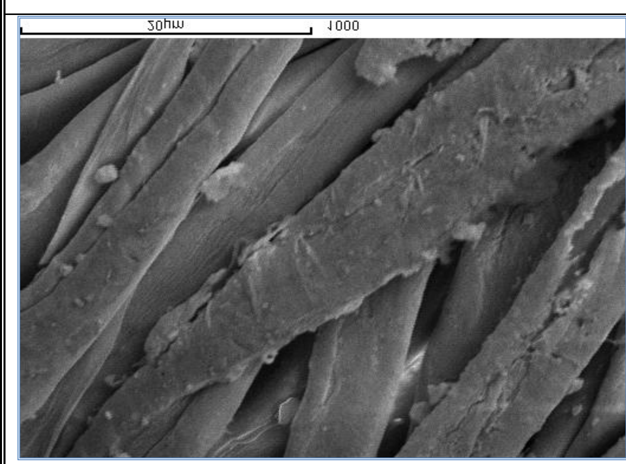
Figure 7 (D) shows SEM of CMC fabric after easy care finished with glyoxal in presence of PVP and Sanitize®. It is evident that, PVP homo-polymer and Sanitize® form thin film on the fabric surface and between inter fibres.



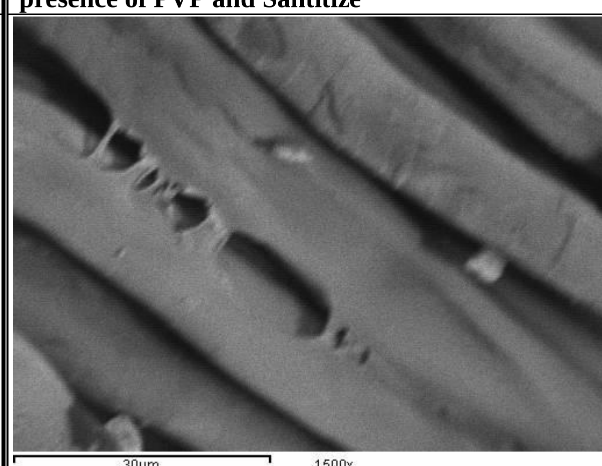
(A) Untreated cotton fabrics



(B) Cotton fabrics crosslinked with glyoxal in presence of PVP and Santitize®



(C) CMC



(D) CMC fabrics crosslinked with glyoxal in presence of PVP and Santitize®

Fig 7: SEM of cotton and CMC before and after crosslinked with glyoxal in presence of PVP and Sanitize®

Conclusion

Incorporating of dimethyltetradecyl-[trimethyl silane-propyl] ammonium chloride (DTPAC), (commercially available as Sanitize® T99-19) and poly (vinylpyrrolidone) (PVP) as antimicrobial agents in easy care finishing formulation along with glyoxal enhances the easy care and functional properties of the treated fabrics. Whereas both antimicrobial activities against *E. coli* and *S. aurous* are marginally improved compared with those fabrics treated with Sanitize® only. Moreover, crosslinking of cotton cellulose with glyoxal using pad dry cure techniques in presence of PVP and Sanitize® increases the crease recovery angle (CRA) of the treated fabric whereas both air permeability, wettability and tensile strength are kept unaltered. Pre-carboxymethylation of cotton fabric increase both CRA and tensile strength. FT-IR analysis shows that; the characteristic peak of PVP and Sanitize® are observed in FTIR of cotton and CMC fabrics after crosslinking with glyoxal in presence of PVP and Sanitize®. SEM of fabric after easy care finished with glyoxal in presence of PVP and Sanitize® shows a thin film from PVP homo-polymer and Sanitize® on the fabric surface and between inter fibres.

References

1- Y. Gao and R. Granston, *"Recent Advances in Antimicrobial Treatment of Textiles", Textile Research Journal*, **78** (1) 60-72 (2008).

2- P. Bajaj, "*Finishing Textile Materials* "*Journal of applied polymer Science*, **83** (3) 631-659 (2002).

3- M. C. Thirty, "*Antimicrobial take the field" AATCC Review*, **1** (11) 11-17 (2001).

4- E. Menezes, "*Antimicrobial Finishing for Specully "Textiles International Dyes*, **187** (12) 13-16 (2002).

5- R. Perumlray," *single-stage antimicrobial and crease proof finishing of cotton materials*" *Journal of Industrial Textiles*, **42** (4) 376-391 (2012).

6- W. Wei, G. Yang, "*Polymeric Carboxylic acid and citric acid as a non formaldehyde DP finish*" *Textile Chemical and Color*, **32** (2) 53-57 (2000).

7- A. El-Shafei, R. Refaie and A. Hebeish," *Improving non formaldehyde easy care finishing of cotton using glyoxal-chitosan combination*",*3rd International Conference of Textile Research Division*, April 2-4 (2006).

8- R. Refai, M. Hashem and A. Hebeish, "*Inducing durable press in ionically crosslinked cotton fabric", Research Journal of Textile and Apparel*, **9** (2) 47-63 (2005).

9- Sanitized AG and Clarinet International. Back up information of sanitized T99-19, Product Nr.T5302 E08n, www.sanitize.com.

10- ASTM, Standard Test Method. (1994). *Breaking load and elongation of textile fabric*, D-1682-94.

11- O. Irob, Y. Moo and W. Anderson, "*Antimicrobial activity of annatto extract*",
International Journal Pharmacy, **3**, 84-96 (1996)

12- M. Rowe, *Textile Chemistry and Colorist*, **10**, 207 (1978)

13- K. Yamanoto**,** *Crease-Resistance Treatments of Cotton Fabrics with Non-Formaldehyde Crosslinking Agents Textile Research Journal, 52, 357 (1982).*

14-- M. Hashem, R. Refaie, K. Goli, B. Smith and P. Hauser, *Enhancement of Wrinkle FreeProperties of Carboxymethylated Cotton Fabric via Ionic Crosslinking with Poly(vinylpyrrolidone), Journal of Industrial Textiles*, **39**, 1, 57-80 (2009).

15- H.M. Fahmy "Activities Fabrics to Improve their Performance Properties and Antibacterial Utilization of Poly(N-vinyl-2-pyrrolidone) in Easy Care Finishing of Cotton**.** JOURNAL OF INDUSTRIAL TEXTILES, *39: 109-122* (2009)

16- Schindler, W. and P. Hauser, *Antimicrobial Finishes, Chemical Finishing of Textiles*, Woodhead Publishing Ltd, Cambridge, England, 2004, p165. **17- M. Hashem, M. El-Shakankery, S. Abd El-Aziz, M. G. Fouda, H. Fahmy,**

Improving easy care properties of cotton fabric via dual effect of ester and ionic

Carbohydrate Polymers, **56**, 1692-1698 (2011).

18- Y. Li and I. Hardin, "*Treating cotton with cellulases and pectinases: Effects on cuticle and fiber properties*", *Textile Research Journal*, **68** (9) 671–679 (1998).