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**RESEARCH ARTICLE**

# Development of Medical Reusable Gowns Using Eco-Friendly Finishing Agents

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## *Abstract*

In the domain of medical textiles, production of reusable gowns required for bedridden patients as well as therapeutic staff is of prime importance. To realize this objective, a plan was designed to functionalize cotton fabrics with eco-friendly finishing agents. A plenty of experiments have been carried out to prepare cotton fabrics bearing antibacterial character essential for hospital bed sheeting, surgical articles and other medical and nursing purposes. Triclosan, β-Cyclodextrin and glyoxal are the main modifying agents for twill-weave cotton throughout this investigation and under a variety of conditions. To achieve this target, step-wise and simultaneous processes for preparation have been extensively studied. Tentative reaction mechanisms are suggested to clarify chemical reactions involved and the products are characterized by FTIR measurements and SEM micrographs. A study of fabric performance and antibacterialproperties of finished products is undertaken and an elucidation of the findings is given in some details. Antibacterial activity is evaluated according to the standard Agar Diffusion Method and its durability to washing is also reported. Results revealed that finished fabrics reserved most of their performance involving strength properties in addition to enhanced air and water permeability. Finished cotton fabrics prepared under these conditions, exhibited an efficient and durable antibacterial characteristic and thus can be categorized as multipurpose medical textiles.



## Introduction

In view of the need to produce textile materials with antibacterial properties, voluminous research has been achieved on the preparation of antibacterial agents including quaternary ammonium compounds, chitosan, triclosan, nanoparticles of noble metals, bioactive plant – based matters and several other products. Choice of an antibacterial agent for application is a function to its activity, toxicity, durability, good washing resistance and ecological acceptability [3].

Triclosan, (commercially known as Irgasan DP 300) also chemically defined as 5-chloro-2-(2,4-dichlorophenoxy) phenol or 2,4,4\-trichloro-2\-hydroxy diphenylether (Figure 1).

Triclosan is an antibacterial finishing agent that can be incorporated into many skin-care products, tooth pastes, liquid soap, carpets, children's toys and plastic kitchenware and many other consumer products [3-5]. Owing to its character as a broad spectrum antibacterial agent in addition to its low toxicity and bacteriostatic activity towards a wide range of both gram-positive and gram-negative bacteria, triclosan inhibits the growth of microorganisms by an electrochemical mode of action. The biocidal agent blocks the active site of the protein reductose enzyme (ENR) which is an essential enzyme for fatty acid synthesis in bacteria [5, 6].

Researchers showed that triclosan is fairly harmless to humans and is a very potent inhibitor to building of ENR enzyme and only a small amount is needed for powerful antibiotic action. In addition, since the agent is not water soluble, it does not leach out and it continuously inhibits the growth of bacteria in contact with surface [4].
To achieve a more durable antibacterial finishing on cotton textiles, triclosan has been inserted into the hydrophobic cavity of β-Cyclodextrin to form an inclusion complex which was then embedded in a polymer film or fibre, or encapsulation in microspheres which were subsequently attached to a polymeric substrate [4].
β-Cyclodextrin (β-CD) is an important biodegradable and nontoxic textile auxiliary composed of a cyclic oligosaccharide produced via enzymatic degradation of starch. Seven pyranose units are bound into a toroidal-shaped cyclic structure (Figure 2). Its chemical structure is characterized by a hydrophilic outer surface and an internal hydrophobic hollow interior, which render it capable of holding other molecules to form inclusion compounds, a property which can be exploited in textile finishing producing extraordinary results [7].

Recently, an intensive research work has been focused on surface treatment of cotton using β-Cyclodextrin or some of its derivatives in order to produce functionalized cotton textiles bearing desirable properties. This can be exemplified by the treatment leading to decrease of the rate of volatile fragrance substances [8], anchoring β-Cyclodextrin to retain fragrances on cotton using hetero bifunctional reactive dyes [9] and multi finishing of cotton to impart antibacterial in addition to anticrease characteristics on cotton fabrics [10].

It is known that some pharmaceutically active compounds, fungicidal and bactericidal substances are able to form complexes with Cyclodextrins. Moreover, a number of applications based on this character are reported.This includes removal of nicotine and tar from smoke, reduction of bad odour from different sources by inclusion in cavities of Cyclodextrin attached to the textile fabric [11]. The comfort of wearing is positively influenced by Cyclodextrin for the textiles used directly in contact with the body.
This project is mainly meant to help prevention of formation of bedsores facing bedridden patients at hospitals and centres of health care through preparation of specially treated gowns characterized by their antibacterial properties. In addition to antibacterial characteristics, gowns produced under should exhibit acceptable abrasion resistance, liquid penetration and thermal insulation properties for prevention of bedsores development.

To achieve this objective, it is planned to treat cotton fabric with a powerful antibacterial agent (triclosan) under the influence of a catalytic action and following a defined experimental scheme. The character of Cyclodextrin to form inclusion complexes will be employed in the gown preparation to increase wear comfort through improvement of wetability and antistatic properties together with gradually and continuously releasing previously encapsulated triclosan. The slow release of the triclosan, if achieved, would offer antibacterial agent depletion resistance with washings [12].

Gown preparation was carried out using two different processes; a step wise process and a simultaneous process. The efficacy of each of the two processes in producing well treated fabric sample are to assessed and compared in following steps in order to highlight the best method for fabric preparation.

**Experimental**

**Materials**

**Cotton fabric**

Twill weave fabric applied in this project was kindly supplied by Misr Company for Spinning and Weaving, Mehalla El-Kobra, Egypt, mill scoured and bleached with the following specifications; nominal wt 145 g/m$^2$ and yarn count 30/1 for warp and 24/1 for weft.

**Fabric preparation**

The chosen fabric was further laboratory purified by washing at 100 $^oC$ for 60 minutes using a solution containing 2 g/l sodium carbonate and 1 g/l Hostapal CV-ET (nonionic wetting agent).The fabric was then washed with hot water and left to dry at ambient conditions

**Chemicals**

- Glyoxal (40% aqueous solution) as crosslinking agent
- β-Cyclodextrin (β-CD) was kindly provided by Wacker-Chemie, GmbH, München, Germany
- Triclosan 2,4,4\-trichloro-2\-hydroxy diphenylether, commercially known as Irgasan DP 300, M.W. 289.54 g/mol was purchased from Sigma – Aldrich Co.

- Aluminum sulphate, $Al_2(SO_4)_3.18H_2O$ and ethyl alcohol were of laboratory-grade chemical reagents.

**Methodology**

***Step wise process:***

Finishing with glycoxal and β-CD (Step A)

Fabric samples were impregnated for 5 minutes in a padding solution containing glycoxal, β-CD and $Al_2(SO_4)_3$. The sequence of addition was as follows; a small amount of water added to the β-CD and the resulting solution was warmed up until complete dissolution. The solution was then cooled down to room temperature and the pad bath concentration was adjusted along with a concurrent addition of glycoxal and the catalyst. The cotton fabric was padded twice, and then squeezed to a wet pick up of ca 100 % on a laboratory padder. The fabric was dried at 80$^oC$ for 5 minutes and cured at 120 $^oC$ for 3 minutes. Finally the fabric was rinsed with warm water at 45 – 50 $^oC$ and left to dry at room temperature.

Treatment with triclosan as antibacterial agent (Step B)

Fabric samples previously treated with glyoxal and glyoxal / β-CD in presence of catalyst were impregnated in a solution containing different concentrations of triclosan (10 g/L -50 g/L) in water / ethanol separately at 60 $^oC$ for 30 minutes on a water bath.

***Simultaneous process:***

Equimolar amounts of triclosan and β-Cyclodextrin were mixed together and then manually grinded using a mortar and a pestle for 15 minutes. These conditions lead to the best yield and to the most stable complexes. The triclosan and β-CD complexes were further dissolved in water; this was followed by addition of glycoxal and aluminium sulphate. Cotton fabrics were impregnated in the above solution; containing the β-CD complexes, glycoxal, and aluminium sulphate. Cotton was padded twice to a wet pick up ca. 100 %. The fabric was then dried at 85$^oC$ for 5 minutes and cured at 120$^oC$ for 3 minutes

**Characterization**

**Evaluation of the finished fabric**

1- Tensile strength (TS) was tested in the warp direction according to ASTM: D 1296 – 98.

2- Tear strength measurements were conducted as per ASTM: D2261 – 96.

3- Air permeability assessment was performed according to ASTM: D737-75. It is expressed as the quantity of air, in cubic centimetre, passing through square centimetre of fabric per second ($cm^3/cm^2/sec$).

4- Water vapor permeability (Moisture transmission rate, MVTR or breathability) is a physical assessment describes the materials ability to be wet by water. It is determined with the HRN: EN 20811:2003.

5- FTIR Measurements:
The structures of resulting products due to interaction between β-CD, glyoxal and triclosan were monitored using Fourier Transform Infrared Spectroscopy (FTIR) as per a procedure described elsewhere [12]. FTIR spectra were recorded using S-100 FT-IR spectrometer (Perkin Elmer) and scanned from 4000 to 400 $cm^{-1}$ in ATR mode and using potassium bromide as supporting material.

6- Scanning Electron Microscopy (SEM)
The morphology of untreated cotton , cotton treated with glyoxal and catalyst), β-CD and triclosan in presence of a catalyst under different reaction conditions were characterized by Scanning Electronic Microscopy technique (Electron prop micro analyzer (JEOL,JXA –840 A).

7- Antibacterial assessment
The antibacterial activity of the treated samples were screened in vitro against *Staphylococcus aureus* (G+ve bacteria), *and Escherichia coli* (G-ve bacteria) using the Agar Diffusion Method.according to AATCC test method 147 - 1988.

8- Durability
Assessment of the durability of treated textile fabric to washing was carried out using standard detergent free from optical brighteners after 20 laundry cycles and determined according to AATCC - test method 135 – 2000.

## .Results and Discussion

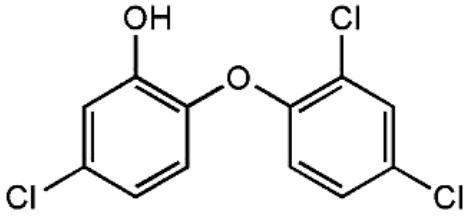
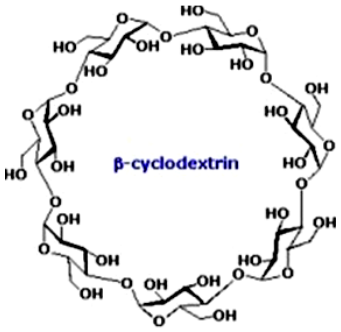



Figure 1: Molecular structure of Triclosan



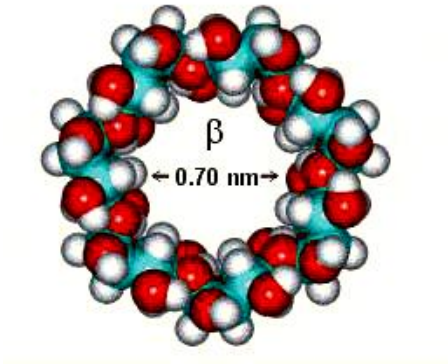
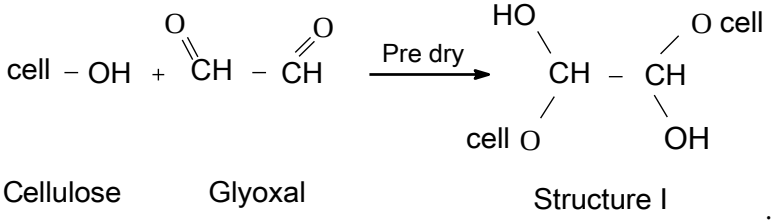




Figure 2: Schematic structure of β-Cyclodextrin

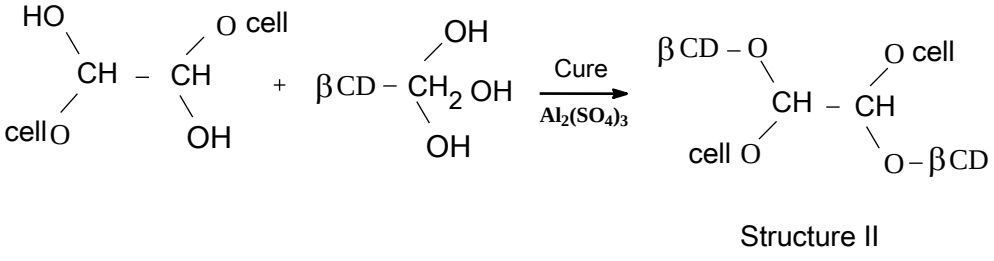
### 3.1. Tentative Mechanism

The reaction between β-CD and glyoxal, leads to the formation of ether linkage between the two reacting molecules. When cotton cellulose is reacted with β-CD/glyoxal mixture, further ether bond formation takes place between glyoxal and cellulose hydroxyl, and then, β-CD is linked to cotton cellulose through a strong ether bonding renders it insoluble and withstands washing [14].

The scheme illustrated below represents the chemical reaction between cellulose hydroxyl and glyoxal to yield "structure I".

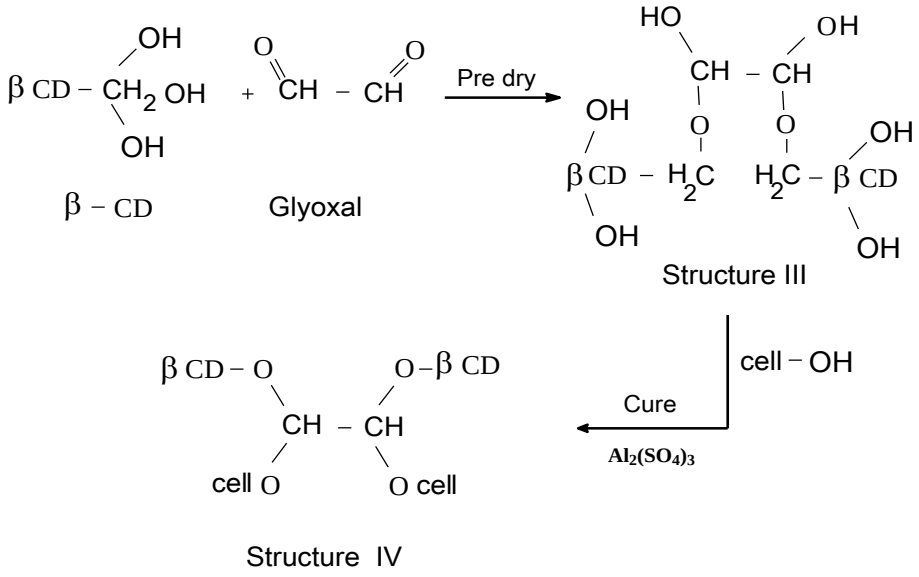



Structure I in turn, reacts with β-CD to form "structure II".




Alternatively, glyoxal reacts with β-CD to yield "structure III", upon reacting with cotton cellulose, the symmetrical molecule "structure II" is formed. Obviously, structures II and IV represent crosslinked cotton cellulose to which β-CD moieties are chemically bonded. These structures can function as guest molecules to receive or

release certain active agents in number of fields of applications, especially those bearing antibacterialproperties for health care of humans [12].




**Determination of β-CD concentration on the fabric using - UV-Vis spectrophotometry**

Phenolphthalein (Ph.Ph) as a visualizing agent was used as an indicator for measuring the concentration of β-CD on the fabric. The colour intensity of Ph.Ph is proportional to the amount of β-CD on the fabric at low concentrations.The glyoxal - β-CD treated fabric was cut to 2.5 cm X2.5 cm and accurately weighed. The fabric sample was immersed in 50 ml Ph.Ph solution (5 X $10^{-5}$ M )and mechanically shaken at room temperature for an hour. After removing the fabric sample the Ph.Ph solution absorbance was measured at a maximum absorption wave length of 551 mμ. The amount of β-CD on the fabric was calculated using the calibration standard curve.

UV-Vis spectrometry is an easily performed first test of the occurrence of complexation in particular in nonfluorescing systems. Moreover, the power of modern chemometric techniques allows valuable analytical applications of small effects of β-CD inclusion on UV-Vis spectra. The emphasis of absorption changes and absorption studies will be on the apparent changes in the chemical properties of guest molecules, such as acid–base equilibrium. For that purpose, Phenolphthalein-modified β-CD was synthesized for as a new type of guest-responsive color change indicator. UV–Vis spectrophotometer (Jasco V-530 uv/vis spectrophotometer) was used to measure the absorbance of phenolphthalein solutions (400 – 700 nm)

The most distinguished work in this field is report by Taguchi [15]. He has demonstrated that upon the binding of phenolphthalein to β-CD cavity in aqueous solution at pH 10.5, the red-colored dianion form is rapidly transformed into a colorless lactonoid form.

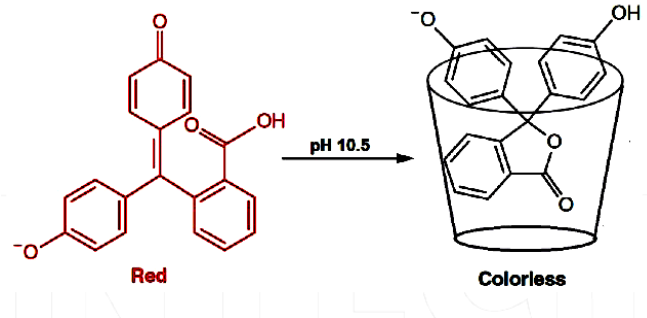



**Fig .3Proposed mechanism for the colour change of phenolphethalein in the presence of β-CD.**

The spectrophotometric technique involving phenolphthalein as the competing reagent appears to be the most promising test. It is based on the fact that in alkaline solutions a colourless 1:1 complex is formed between phenolphthalein and β-CD that the red phenolphthalein dianion is partially displaced by a competing reagent to an extent depending upon its affinity to form a complex with the β-CD host.

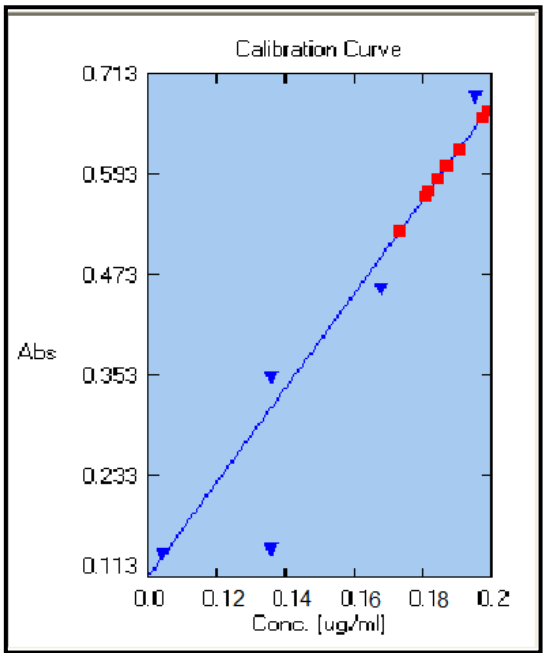



Fig.4: calibration curve of phenolphthalein

| Cotton | Abs | β-CD (mg/g of fabric) |
|---|---|---|
| **Untreated cotton** | 0.772 | 0 |
| **Cotton treated with 10 g/l β-CD** | 0.587 | 0.51 |
| **Cotton treated with 20 g/l β-CD** | 0.566 | 2.5 |
| **Cotton treated with 30 g/l β-CD** | 0.622 | 5.8 |
| **Cotton treated with 40 g/l β-CD** | 0.661 | 9.6 |
| **Cotton treated with 50 g/l β-CD** | 0.668 | 9.6 |

**Table 1 Amount of β-CD on treated cotton fabric**

Relation between The actual weight of β-CD and glyoxal in a liter of water and the amount of β-CD (mg)/g of fabric was shown in the table. As shown from the results, increasing concentration of β-CD in the treating bath increasing the amount of cyclodextrin add on (fixed) per gram fabric. The maximum add on (fixation) was achieved by treatment of cotton fabrics with 40 g/L β-CD(table1) and this result is consistent with the rest of the other results inferred that the ideal quantity for using β-CD on cotton fabric is 40 g/L

**.2.Factors affecting stepwise process (process A&B)**

**3.2.1 Effect of β-CD concentration on fabric performance properties**

A study of the effect of variation in β-CD concentration on the performance properties of treated cotton fabric revealed, as shown in table 2, that increasing the β-CD concentration in the treating bath leads to a remarkable decrease in strength properties including tensile properties, elongation at break and tear strength. The decrease in strength properties at higher concentrations of β-CD can be ascribed to extended molecular degradation of cellulose under both hydrolytic effect of the catalyst and deposition of rigid cellulosic segments within the fine structure of cotton cellulose.

**Table 2: Effect of variation in cyclodextrin concentration on performance of cotton fabric**

| β-CD concentration (g/L) | T.S | Elongation at break | Tear strength (gm) | Air permeability A.P L./$m^2$/min | Moisture vapor transmission MVTR g/$m^2$/24hrs. |
|---|---|---|---|---|---|
| Blank | 77 | 18 | 2000 | 309 | 420 |
| 20 | 43 | 13 | 900 | 380 | 627 |
| 30 | 42 | 13 | 800 | 445 | 867 |
| 40 | 42 | 12 | 800 | 588 | 1244 |
| 50 | 41 | 12 | 800 | 587 | 1243 |

*Glyoxal, 50 g/L; triclosan, 10g/L; $Al_2(SO_4)_3$, 20g/L; dry, 80 $^oC$/5 min.; curing, 120 $^oC$/3 min,M:L,1:10(owf).*

It can be stated that introduction of β-CD to the finishing bath and its subsequent inclusion in the chemical bonding of cellulose microstructure leads to act as plasticizer, reducing, to some extent, the rigidity caused by crosslinking and may also lessen the degree of acid hydrolysis which may occur due to presence of glyoxal and catalyst [16]. Data of elongation at break of treated fabric under these conditions seem to be unaltered.

**Air permeability**: The air permeability [17] of a fabric also influences its comfort behaviour. A material that is permeable to air is also ,in general ,likely to be permeable to moisture vapor. Thus, the moisture- vapor permeability and liquid moisture transmission are normally closely related air permeability so the changes in fabric air permeability as a function of relative humidity are important for chemical protective clothing point of view .Table 2 contains the values of air permeability for fabrics treated with different concentrations of β-CD in presence of glyoxal and triclosan Table 1 shows that values of air permeability are increases with increasing the concentration of β-CD up to 40g/l then levelled off. The use of twill fabric provides higher air permeability as compared to plain weave fabric. This property determines the air flow rate of the fabric which influences the comfort behaviour of the fabric

**Water vapor permeability i.e. breathability or moisture vapor transmission rate (MVTR):** Breathability (18,19) is the ability of textile construction to allow water vapor to pass out from the body through it, but not to allow liquid from the outside .The escape of body generated moisture vapor affects comfort significantly. Breathability akey factor to decide its comfort is measured in terms of moisture vapor rate (MVTR). (MVTR) represents the amount of moisture that passes through a clothing assembly such as a dressing during a given time period the higher (MVTR) the higher the comfort level of the fabric. This means that the perspiration (vapor state i.e. before condensation) generated by body due to physical exertion allowed to pass through the fabric by the presence of interstics in the finished fabric

Data in table 1 indicate an enhancement in water vapor permeability which expressed as (MVTR), when the concentration of β-CD is increased up to 40g/l then levelled off

### 3.2.2. Effect of β-CD concentration on the antibacterial activity of fabric treated with Triclosan

Cotton fabrics having the antibacterial properties were prepared by the action of β-CD onto cotton cellulose in presence of glyoxal as a crosslinker, and then loaded by triclosan in 10g/L concentration as an antibacterial agent. Samples were tested for this property according to standard diffusion disk method and the durability of the durability of the finish after 20 washing cycles is examined.

The results displayed in figure 5 show the inhibition zones diameter(IZD) observed with glyoxal–treated, samples treated with glyoxal in presence of β-CD and absence of triclosan and samples finished with β-CD in different concentrations in presence of glyoxal (50 g/L) using the pad - dry – cure technique followed by treatment with triclosan (10 g/L) and evaluated for antibacterial properties against (G +ve) Staphylococcus aureus) and (G–ve)Escherichia coli) bacteria according to the standard method for evaluation. It is evident from fig.5 that increasing β-CD concentration from 20 g/l to 40 g/l in causes an increase in antibacterial activity expressed as an inhibition zone diameter (IZD).

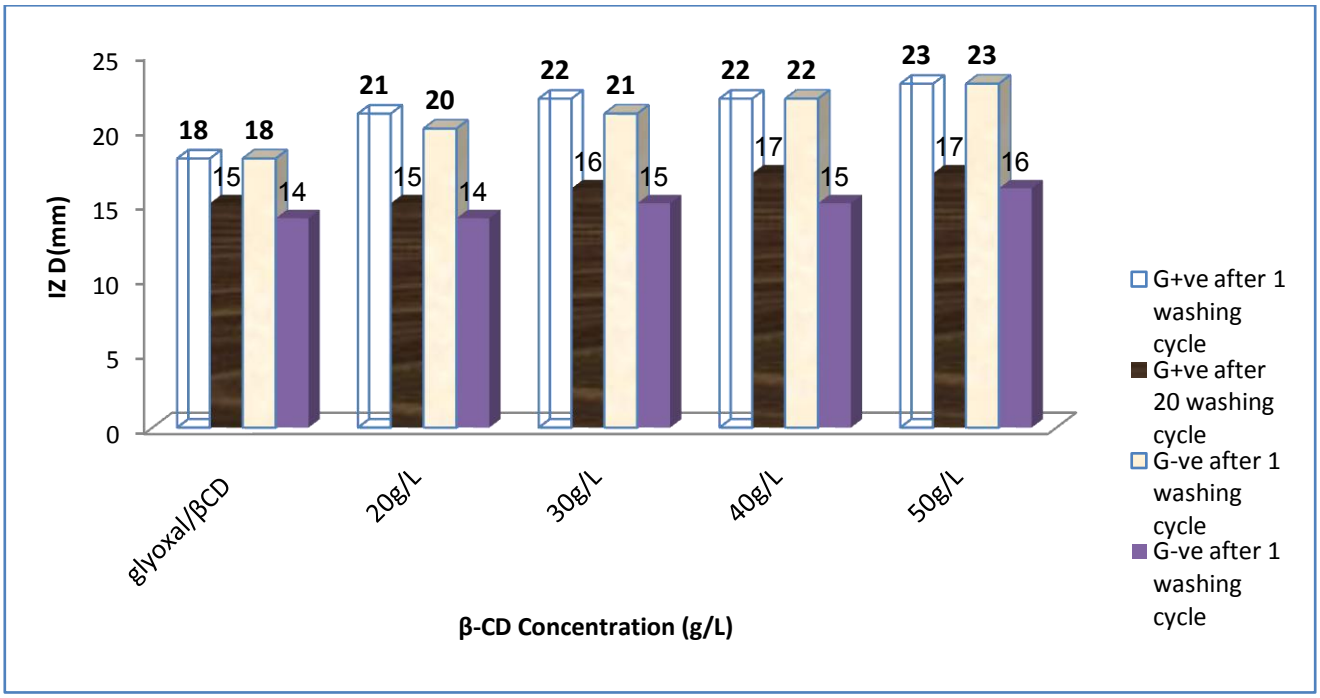



**Figure 5: Effect of β- cyclodextrin concentration in presence of glyoxal on the antibacterial properties of cotton fabric reacted with 10g/L triclosan,M:L 1:10(owf)**

**Condition used : [triclosan] ,10g/L;[glyoxal] 50g/L , [$Al_2(SO_4)_3$] 20g/L,**

**M:R,1:10(owf). blank sample:glyoxal/ β-CD in absence of triclosan**

Results revealed that IZD for samples treated with β-CD / glyoxal were in the 18 mm and 14 mm for both G+ve and G–ve bacteria after one washing cycle and 20 washing cycles respectively because of slight anti bacterial properties of glyoxal [20].

Fig 5,6 shows also that treatment of β-CD treated samples with triclosan produces finished fabrics with enhanced antibacterial properties expressed as IZD values with both G+ve and G–ve. This indicates the role of β-CD in hosting triclosan in the cavities and the antibacterial property is reserved even after 20 washing cycles

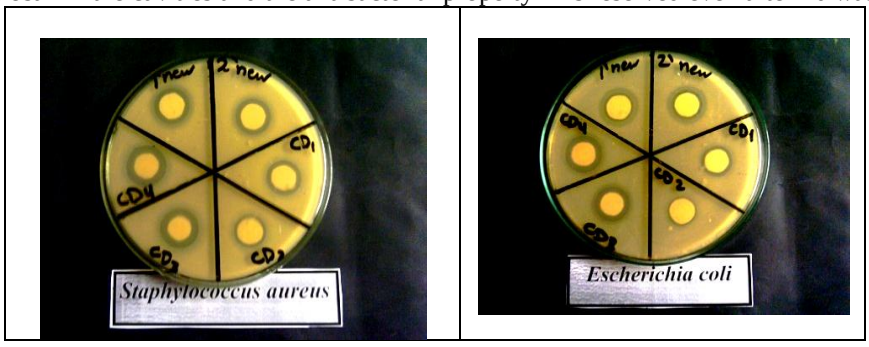



**Figure 6 : Effect of β- cyclodextrin concentration in presence of glyoxal on the antibacterial properties of cotton fabric reacted with 10g/L triclosan,M:L 1:10(owf)**

### 3.3. Effect of variation of triclosan concentration on the performance of cotton fabric treated with glyoxal and β-CD

The effect of variation in concentration of triclosan on the performance of cotton fabric pre-treated with glyoxal (50 g/L) and β-CD (40 g/L) and $Al_2(SO4)_3$ catalyst was thoroughly investigated. The results are listed in table 3 and elucidation of these results indicate that higher concentrations of triclosan are accompanied with a decrement of strength properties of treated fabric, probably due to the acidic conditions of reacting triclosan leading to deterioration of treated cotton fabric. Higher values of tensile strength are observed for fabrics treated in absence of triclosan and presence of β-CD, glyoxal and catalyst.

As mentioned before, β-CD seems to act as a plasticizer which in turn reduces rigidity of cotton and lessens acid hydrolysis encountered on crosslinking with glyoxal and catalyst [16]. Raising the triclosan concentration left the values of elongation at break unchanged. Results of table 3 indicate that increasing triclosan concentration from from 20 g/L to 50 g/L has no effect on tear strength of fabrics treated under these conditions.

**Table 3: Effect of variation in triclosan concentration on the performance of cotton fabric treated with glyoxal/ *β-CD***

| Triclosan concentration (g/L) | T.S | Elongation at break | Tear strength (gm) | Air permeability A.P L./$m^2$/min | Moisture vapor transmission MVTR g/$m^2$/24hrs. |
|---|---|---|---|---|---|
| Blank cotton | 77 | 18 | 2000 | 309 | 420 |
| Glyoxal / β-CD (g/L) in absence of triclosan | 38 | 13 | 800 | 380 | 1252 |
| 10 | 37 | 12 | 800 | 585 | 1245 |
| 20 | 37 | 12 | 600 | 620 | 1238 |
| 30 | 31 | 12 | 600 | 623 | 1240 |
| 40 | 28 | 12 | 600 | 621 | 1242 |
| 50 | 26 | 12 | 600 | 620 | 1239 |

*Glyoxal, 50 g/L; β-CD, (40 g/L); $Al_2(SO_4)_3$, 20g/L; drying, 80 $^oC$/5 min.; curing, 120 $^oC$/3 min.M:L,1:10(owf)*

Results of **air permeability** for cotton pre-treated with glyoxal and β-CD are slightly superior to that of untreated cotton. Incorporation of triclosan (10g/L) in the finishing bath causes a marked enhancement in air permeability of the treated samples. Higher concentrations of triclosan (30g/L to 50 g/L), had no significant effect on such property whereas results are comparable.

**Water vapor permeability, (Water vapor transmission rate (MVRT) )**

For the use of triclosan as an antibacterial finishing agent in the present investigation, it is advisable to examine the role of variation of its concentration on the level of water vapor permeability. Data in table 3 indicate that the lowest value of **MVRT** are observed with fabrics treated with glyoxal and β-CD in absence of triclosan and higher values are found upon introducing of 10 g/L triclosan to the treating bath. Then levelled off with further increase in concentrations of triclosan up to 50g/L

From the medical point of view, these findings are ideal for transmission of liquids, e.g. sweat or biological fluids away from skin surface. This is besides the hydrophilicity of cotton and the role of β-CD in forming complexes without affecting the hydrophilic character of treated cotton fabrics [10]. In addition, twill construction of fabric is considered to be in favour of water absorbency leading to immediate

transfer of moisture to outer layers giving dry feel. This property is essential to keep the patient dry and thus, avoid medical problems may be developed due to wet skin [21].

**3.3.1. Effect of variation in triclosan concentration on the antibacterial properties of cotton treated fabric with glyoxal and β-CD and its durability**

A thorough investigation of the effect of variation in triclosan concentration ranging from (10 g/L -50g/L) expressed as ($CD_1$-$CD_5$) on the antibacterial properties of cotton fabric treated with glyoxal (50 g/l) and β-CD (40 g/l) using the pad-dry-cure technique is carried out. Results represented in figures 7 shows that increasing triclosan concentration from 10 g/L to 50g/L brings about finished fabrics with considerably enhanced antibacterial characteristics, expressed as inhibition zone diameter (IZD) mm/mg sample with both G +ve and G -ve bacteria

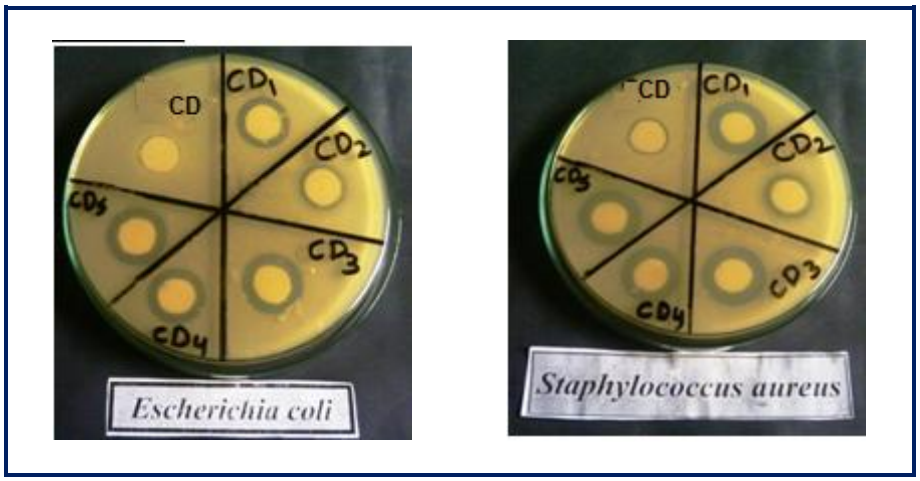



**Figure 7 : Effect of using different concentration of triclosan on treatment of cotton fabric pretreated with glyoxal and β-CD on the antibacterial test using diffusion method**

Experiments have been performed to examine how durable the antibacterial properties of treated fabrics can withstand washing up to 20 cycles Fig8. .Measurements revealed that these finished fabrics retain reasonable antibacterial activity after this number of washings. This retained activity can be attributed to the hosting capability of the cavities within the cyclodextrin moieties which confine antibacterial agent molecules and release them gradually [22].

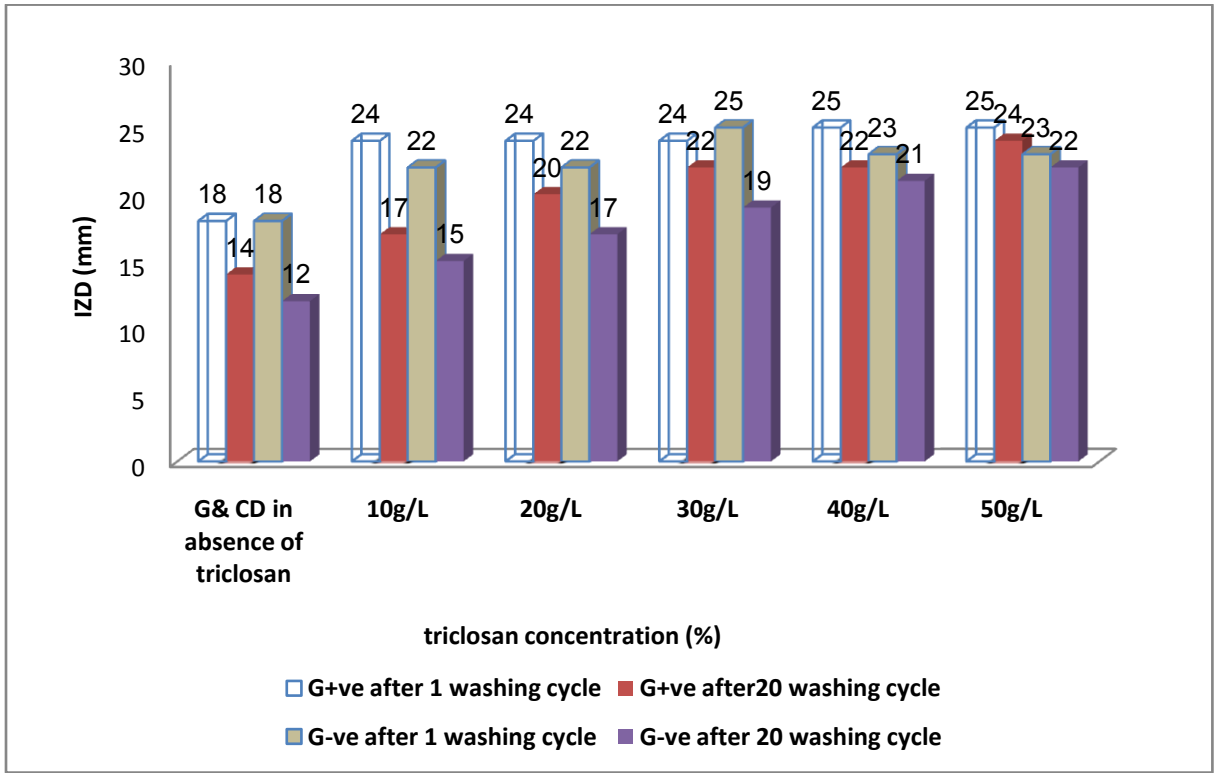



**Figure 8: Effect of triclosan concentration on the antibacterial activity of cotton fabric reacted with glyoxal/ β- CD.**
**Condition used : [triclosan] ,(10-50)g/L;[glyoxal] 50g/L , [β- CD] 40g/L,M:R,1:10(owf) [$Al_2(SO_4)_3$], *20g/L***

### 3.4. Simultaneous Process

Previous procedures adopted for preparation of cotton fabrics bearing antibacterial properties have been performed in Step-wise processes( A&B). These processes include treating fabric samples with glyoxal, β- CD and triclosan using assigned concentration of each agent and different stages of treatment. A systematic study of the effects of variation in concentration of such agents and the impact on fabric performance and antibacterial characteristics has been achieved in some details. In the present section, these processes have been combined to deal with what is known as "Simultaneous Process".

As previously described in the experimental section, equimolar amounts of triclosan and β- CD are mixed together and manually grind using a mortar with a pestle for 10 minutes. The resulting finely divided mixture is dissolved in water followed by addition of glyoxal and $Al_2(SO_4)_3$ .18 ($H_2O$) under constant stirring.

Finishing of cotton fabric involved impregnation of samples in the above mentioned treating solution, squeezed on a laboratory-padder and to a wet pick up of ca. 100 %. The padded samples are dried at 85 $^oC$ for 5 minutes and cured at 120 $^oC$ for 3 minutes.

The results displayed in table 4 are concerned with evaluation of fabric performance for samples simultaneously treated with a mixture of equimolar amounts of triclosan and β- CD in presence of glyoxal/ $Al_2(SO_4)_3$ for half an hour The sample was squeezed, dried and cured under the above mentioned conditions.

A remarkable decrement in tensile strength and elongation at break was observed, while values of tear strength indicate the same situation especially in case as compared with those for untreated cotton

**Air permeability** of the sample was found to be significantly improved as compared with that for untreated cotton. It is suggested that values of air permeability is dependent on the porosity of the fabric surface and thus, air is allowed to pass through it freely.

**Table 4: Effect of variation in triclosan concentration on the performance of cotton fabric treated with glyoxal**

| 1 : 1 β- CD : Triclosan molar ratio | T.S | Elongation at break | Tear strength (gm) | Air permeability A.P L./$m^2$/min. | Moisture vapor transmission (MVTR) g/$m^2$/24hrs. |
|---|---|---|---|---|---|
| Blank | 77 | 18 | 2000 | 309 | 420 |
| Sample | 33 | 15 | 1000 | 427.5 | 910 |

**Condition used:** ***[β-CD:triclosan] ,Mol:Mol, [Glyoxal],50g/L ; [$Al_2(SO_4)_3$ ], 20g/L,M:L,1:10(owf),time of impregnation 30 min.,temp.,room temp.(25°C)***

**Water vapor permeability** (**moisture vapor transmission rate MVTR)** of cotton fabric treated with equimolar amounts of triclosan and β-CD is improved as compared with that of untreated cotton. The level of water vapor permeability of the sample, prepared by impregnation for half an hour in the treating liquor was found to be superior to that of untreated sample

From the hygienic point of view, when the fabric exhibits good water vapor permeability expressed as (MVTR), transfer of moisture in the inner layers of finished takes place and provides the patient with a comfortable dry feel. This character is very essential for bedridden patients to be kept comfortably dry for a long time and to be protected from serious medical problems [ 24], probably leading to bedsores.

**3.4.1. Evaluation of antibacterial activity for finished cotton fabrics,and its durability**

Samples of cotton fabrics treated with triclosan, β- CD, glyoxal and catalyzed by $Al_2(SO_4)_3$ to produce fabric samples bearing the antibacterialcharacter were evaluated for their activity according to a test method 147-1988. Values are expressed as inhibition zone diameter (IZD). .Results as shown in figures 9 and 10 indicate that IZD for triclosan / β- CD in presence of glyoxal finished fabrics are 21 mm / mg sample and 20 mm / mg sample against G +ve bacteria and G–ve bacteria respectively after the first washing cycle this against 17 mm / mg sample and 14 mm / mg sample after 20 washing cycles .


**Figure 8: Effect of treatment of cotton fabric with equimolar amount of β- CD/triclosan in presence of glyoxal onThe antibacterial activity and its durability**

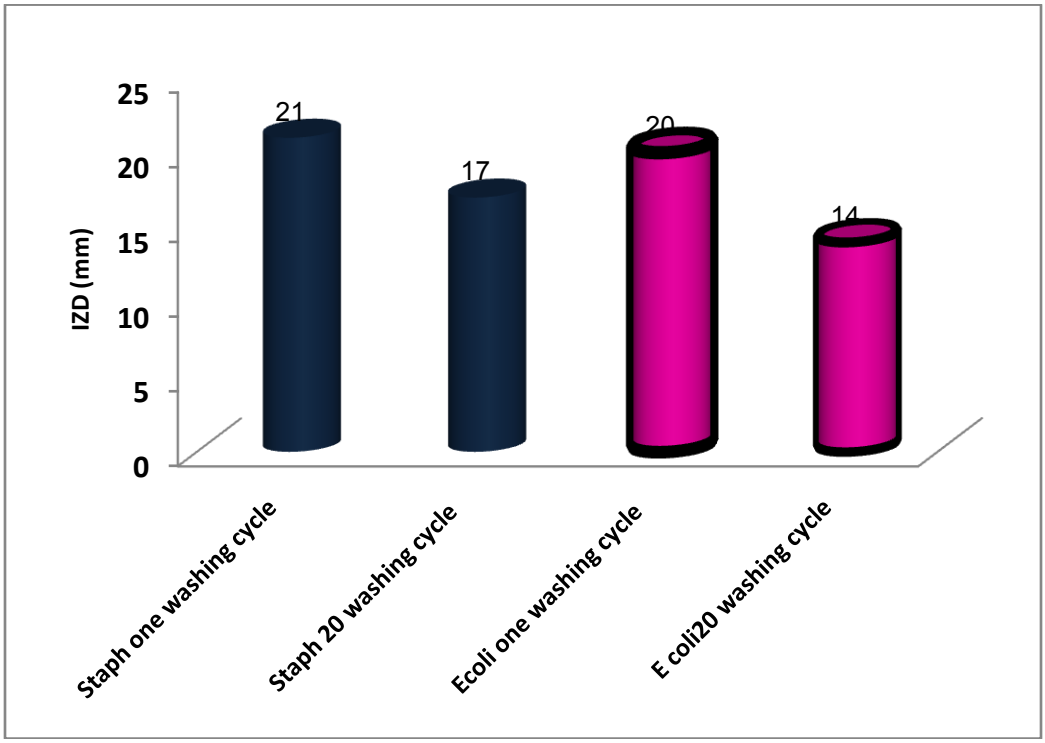



**Figure 10: Effect of treatment of fabric with equimolecular ratio of β- CD and triclosan in presence of glyoxal on its antibacterial activity**

The above mentioned IZD values reflect the roll of β- CD in hosting the antibacterial agent molecules, keeping them as inclusion complexes in the β-CD cavities and slow release of the finish over a long time and withstanding this number of washing cycles [25].

**3.5. FTIR Measurements**

Figures 11, 12 and 13 represent FTIR spectra for samples of cotton fabric treated with glyoxal /β-CD, β-CD / triclosan/glyoxal stepwise process(A&B) and also for the sample treated with equimolar ratio of β-CD / triclosan using simultaneous technique respectively

Absorption band of hydroxyl groups of cotton fabrics treated with glyoxal / β-CD, β-CD/triclosan and equimolar ratio of β-CD/ triclosan 3347 $cm^{-1}$, 3450 $cm^{-1}$, 3346 $cm^{-1}$ and 3418 $cm^{-1}$ with transmission intensity 51.9, 67.7, 37.37 and 56.26 respectively, the lower the transmission intensity the higher number of hydroxyl group is transformed into acetal and hemiacetal products. The lower intensity is observed in case of cotton fabric treated with β-CD followed by triclosan treatment.

The similarity between FTIR of cotton fabric treated with glyoxal and β-CD and that of cotton fabric treated by β-CD/triclosan and with equimolar ratio of β-CD/triclosan indicated that the basic structure of units of β-CD was preserved.

Absence of bands characterized for triclosan (i.e. 1597, 4503. 1470, 750) benzene ring in the last two figures indicate inclusion of triclosan in the cavity of β-CD.

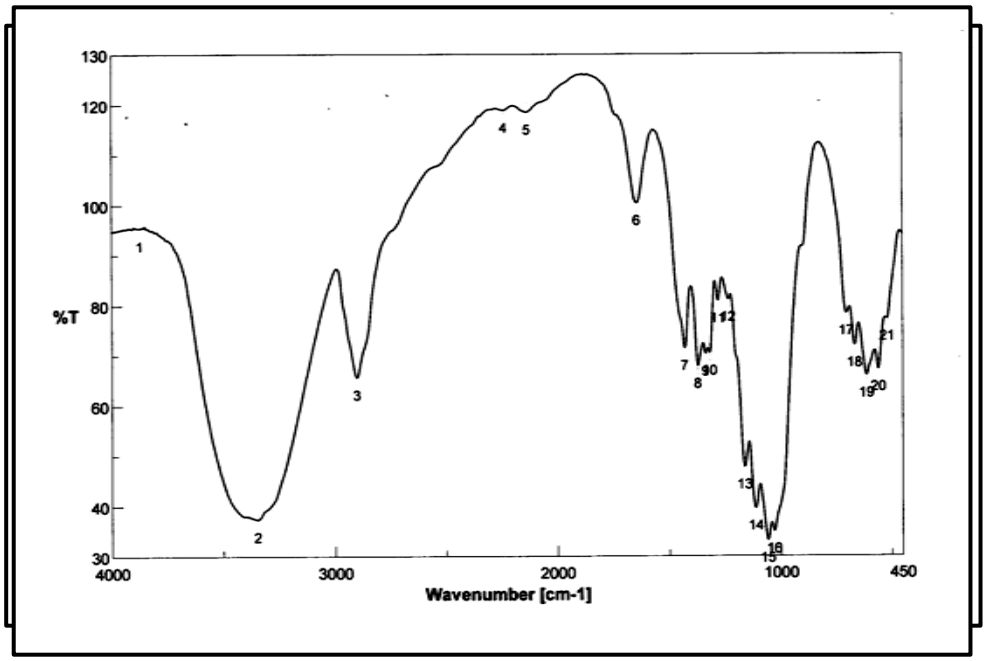



**Figure 12: FTIR of cotton fabric treated with glyoxal/βCD /triclosan (stepwise technique)**

**Figure 11: FTIR of cotton fabric treated with glyoxal and β-CD**

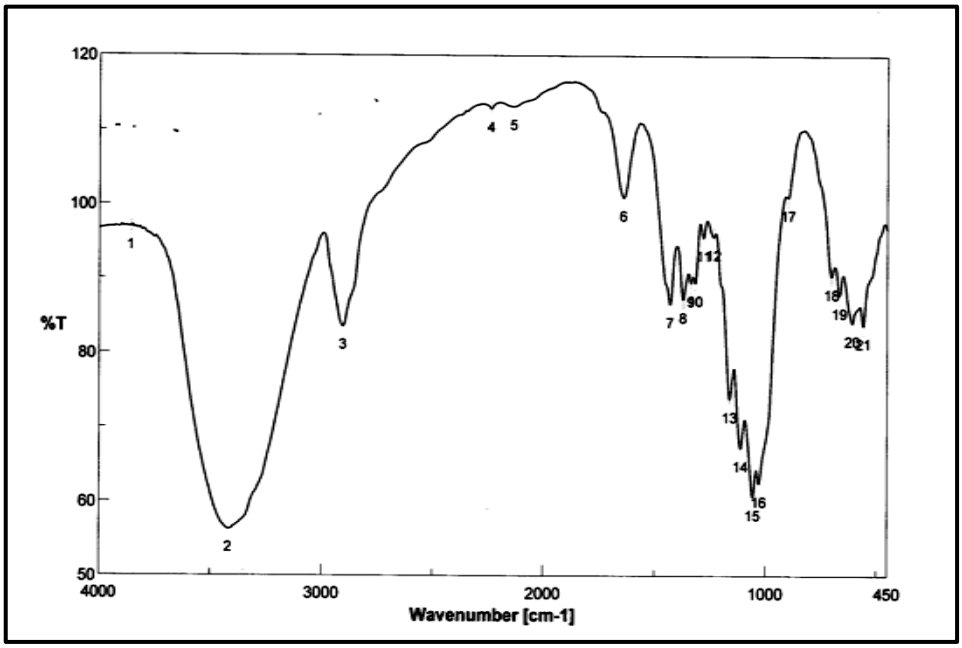
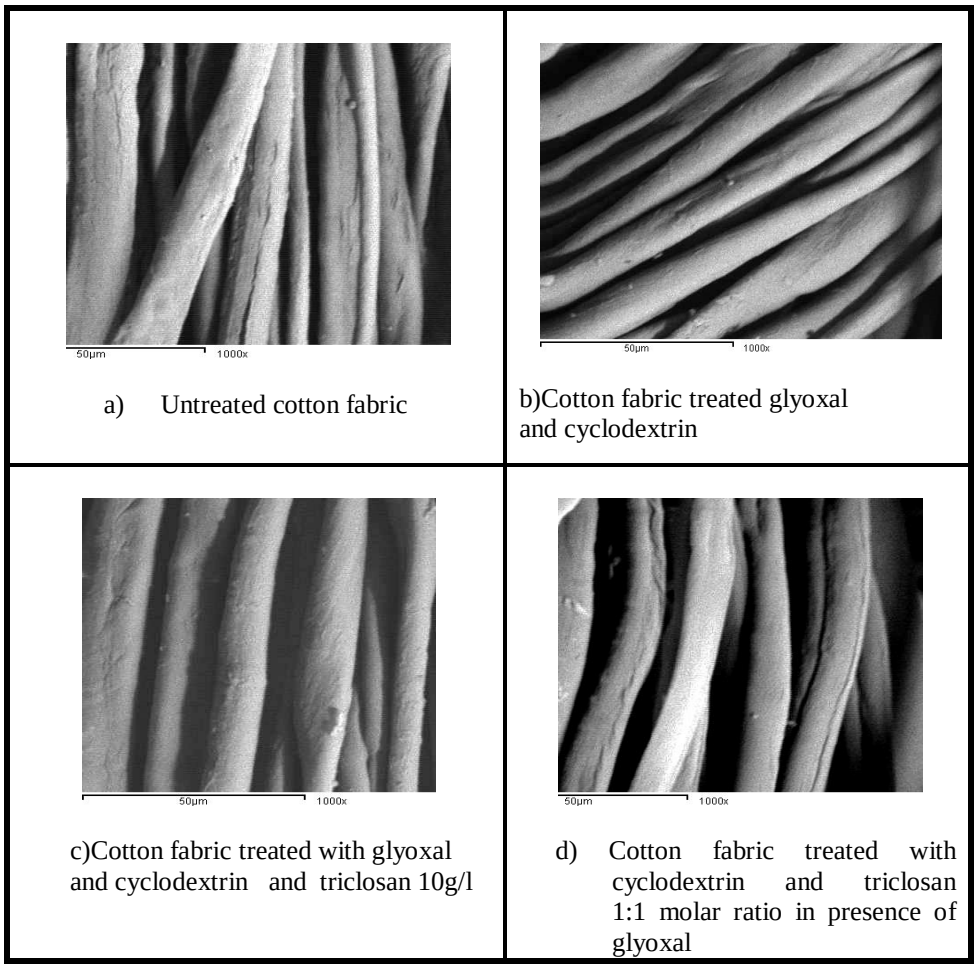



**Figure 13: FTIR of cotton fabric treated with equimolar amounts of βCD /triclosan in presence of glyoxal (simaltanus technique)**

### 3.7. Scanning Electron Microscopy (SEM)

Surface morphology of untreated and treated cotton fabrics were characterized by scanning electron microscopy (SEM). Figure 14 shows the surface morphology of untreated cotton fabric (figure 14a), treated cotton fabric with glyoxal and β cyclodextrin (figure 14b), glyoxal / β cyclodextrin / triclosan 10g/L (figure 14c) and β cyclodextrin and triclosan 1:1 molar ratio (figure 14d).




a) Untreated cotton fabric

b)Cotton fabric treated glyoxal and cyclodextrin

c)Cotton fabric treated with glyoxal and cyclodextrin and triclosan 10g/l

d) Cotton fabric treated with cyclodextrin and triclosan 1:1 molar ratio in presence of glyoxal

**Figure 14: Scanning electron microscope photographs of treated and untreated cotton fabrics**

Glyoxal-β-CD compounds were coated on the surface of both cotton fibers. The glyoxal-β-CD treated cotton photograph Figure14(b) showed that there was white material coated film on their surfaces compared with the untreated ones. Using of triclosan by two different method not affecting the film coating the cotton fabric as shown in figure 14(c, d) and this is another method for proving the inclusion of triclosan into cyclodextrin moiety

Figure 15a shows Scanning electron microscopy of β-CD as large crystalline particles with no definite shape and the physical mixture of β-CD-triclosan is shown in figure 15(b) appeared as unmodified particles of β-CD covered by adhering small crystals of triclosan. The reported changes in morphology of β-CD-triclosan are an indication for complex formation.

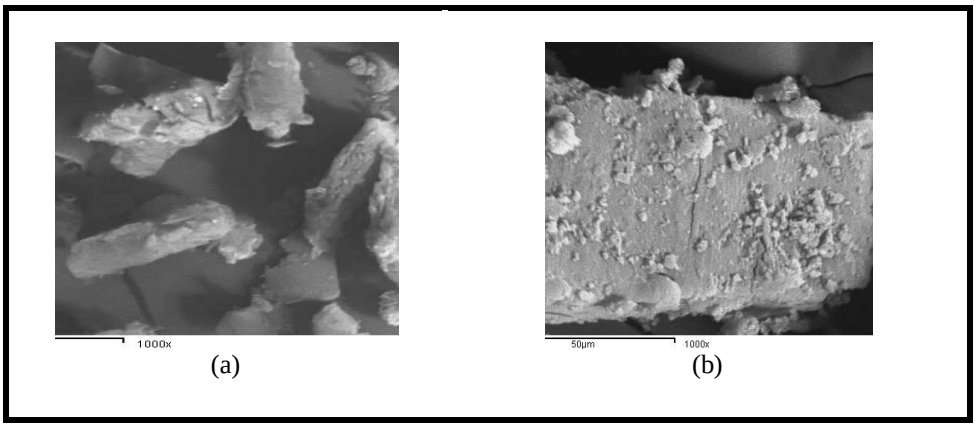



**Figure 15: SEM micrographs for (a) β cyclodextrin, (b) cyclodextrin loaded triclosan after grinding**

## Conclusions

A plan was designed to functionalize cotton fabrics with eco-friendly finishing agents. Several experiments are carried out to prepare cotton fabrics bearing antibacterial characteristics. Triclosan, β cyclodextrin and glyoxal are the main finishing agents for twill-weave cotton under a variety of conditions. Step wise and simultaneous processes for preparation are extensively studied. Products are characterized by FTIR measurements, SEM micrographs and evaluation of bactericidal activity. Results revealed that finished fabrics reserved most of their performance including strength properties and improved air and water vapor permeability. Finished fabrics exhibit efficient and durable antibacterial character up to 20 washing cycles potentially required for medical applications.

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