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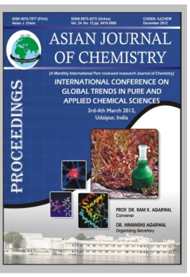

Antibacterial Activity of Magnesium(II) Ions Loaded Cyclodextrin-Grafted-Cotton Fabric†

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In this report β-cyclodextrin has been grafted onto cotton fabric and the magnesium ions were loaded onto the resulted grafted fabric to impart antibacterial activity. Both the prepared β-cyclodextrin grafted fabric and magnesium ions loaded grafted fabric were characterized by scanning electron microscope and Fourier transform infrared techniques. The antibacterial character of magnesium ions loaded fabric was tested by agar diffusion test against *E. coli* and *S. aureus*. The results show that Gram positive (*S. aureus*) bacteria shows better antibacterial character than Gram negative bacteria (*E. coli*).

Key Words: Cyclodextrin, Citric acid, Grafting, Antibacterial.

INTRODUCTION

The peculiar shape and the presence of a hydrophobic cavity in cyclodextrin produce the extraordinary capability of hosting species to include a large variety of different molecules and form the stable inclusion compounds and supramolecular adducts[1-3]. Cyclodextrins are non-reducing cyclic linked oligosaccharides produced through the enzymatic degradation of starch[4]. They are torodial-shaped cyclic oligosaccharides with a hydrophilic outer surface and an internal hydrophobic hollow cavity, which can entrap a vast number of active ingredients. The remarkable ability of cyclodextrins to include hydrophobic compounds has been exploited in several fields including antimicrobial textiles[5]. The growth of microorganisms on textile inflicts a range of unwanted effects on the textile and the wearer as well. Those effects include the generation of unpleasant odour, stains, discolouration of fabric and an increased like hood of contamination[6]. So it is highly desirable to minimize the microbial growth on the textiles. Usually these inclusion compounds can be crystallized and purified and are successfully exploited in different fields such as food manufacturing, cosmetics, pharmaceuticals, analytical and organic chemistry[7-17]. Several papers and patents has been reported on relevant applications of cyclodextrins for antimicrobial, insectifree, aroma finishing and in textile dyeing.

Cotton textiles are highly popular with people because they are like human skin, sweat absorbing and comfortable. Therefore cellulose fibres are the most promising dressing material and more preferred in biomedical applications such as wound and burn dressings[18]. However, cotton fabric provides an excellent environment for microorganism to grow because of their tendency to absorb moisture[19]. Therefore, the application of antimicrobial agents on cotton fabric is needed to impart biocidal action.

In textile field, a novel functional surface treatment of cotton, based on the permanent fixation of β-cyclodextrin on fabric, is receiving increased attention[20,21]. Due to increasing resistance of bacteria against metals such as copper, zinc, cobalt and silver due to their strong antibacterial properties[20]. It has been demonstrated that cyclodextrin fixation on cotton does not affect the hydrophilic property of cellulose and the immobilized cavity of cyclodextrin does not lose its complexing power to form inclusion complexes with other molecules[22]. In this study the cyclodextrin has been grafted onto cotton fabric and the resultant fabric has been loaded with Mg(II) ions. These ions occupy space within internal cavities of cyclodextrin molecules. The entrapment of magnesium ions into cavities of grafted cyclodextrin in the cotton fabric produces long lasting effective biocidal action and provides protection to the fabric against environmental encounters during handling, washing of fabrics, thus its shelf-life enhanced.

EXPERIMENTAL

Cyclodextrin, citric acid, sodium dihydrogen phosphate and magnesium nitrate were obtained from Balaji Scientific Chemicals, Mumbai, India. Cotton fabric was received as a

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gift from Tirupur textile mill. Double distilled water was used throughout the investigation.

Preparation of cyclodextrin grafted cotton fabric: The attachment of cyclodextrin onto pre-weighed cotton fabric was made in aqueous solution containing 0.10 % monomer cyclodextrin, 0.3 % crosslinker citric acid and 0.30 % sodium dihydrogen phosphate and esterification was carried out at 180 ºC for 3 min and then formed poly (cyclodextrin-co-citric acid) grafted fabric was placed at 40 ºC in a dust-free chamber until the fabric was completely dry. All the solutions were prepared in double distilled water.

The percent grafting (G) was calculated using the expression:

$$\% \text{ grafting (G)} = \text{Wg} - \text{Wo}/\text{Wo} \times 100$$

where, Wo and Wg are the sample weights before and after graft-copolymerization, respectively. In order to check the reproducibility of the grafting yield, the graft-copolymerization process was performed in triplicate and average value is reported.

Preparation of magnesium ions-loaded grafted fabric: Magnesium(II) ions loaded grafted fabric was prepared by the following method. A dry pre-weighed piece of grafted fabric was equilibrated in distilled water for 24 h. Thereafter, the swollen fabric was put in aqueous solution of magnesium nitrate, prepared by dissolving 30 mg $Mg(NO_3)_2$ in 25 mL of double distilled water, about 24 h at 27 ºC to incorporate magnesium ions into grafted fabric. The fabric was washed in distilled water for 20 min to remove unreacted salt.

Characterization of magnesium ions-loaded grafted fabric: FTIR studies [Perkin Elmer make model spectrum RX1 (Range 4000-400 cm^{-1}) were done by making KBr pellets with respective samples. Scanning electron microscope (SEM) images were taken with a JEOL JSM 6390 model operating at an acceleration voltage of 15 kV. The antibacterial studies were done by using agar diffusion method (Kirby bauer method). The antibacterial studies were done to the Mg (II) grafted fabric using gram positive and gram negative bacteria's.

Antibacterial test for magnesium-loaded grafted fabrics: The antibacterial activity of the fabrics was tested qualitatively, with *E. coli* species (gram-negative bacteria) and *S. aureus* species (gram-positive bacteria). For qualitative measurement of antimicrobial activity, the magnesium-loaded fabrics were placed at center of disc of 1 cm diameter and the antimicrobial activity was tested using modified agar diffusion assay (disc test). The plates were examined for possible clear zone after incubation at 37 ºC for 2 days. The presence of any clear zone around the circular disc on the plate's medium was recorded as an inhibition against the microbial species[21].

RESULTS AND DISCUSSION

Preparation of magnesium (II) ions loaded fabric (Fig. 1) depicts the formation of β-cyclodextrin grafted cellulose by the esterification of cellulose and β-cyclodextrin. According to Fig. 1, the citric acid, being bifunctional due to presence of -COOH groups, acts as a linker molecule between cellulose and cyclodextrin. One of the carboxyl a group of citric acid reacts with -OH group present in cotton cellulose, while the other carboxylic group is bonded to the cyclodextrin moiety through esterification. In this way, the internal cavity of cyclodextrin molecules, attached to the cotton cellulose, acts as a host to entrap foreign species.

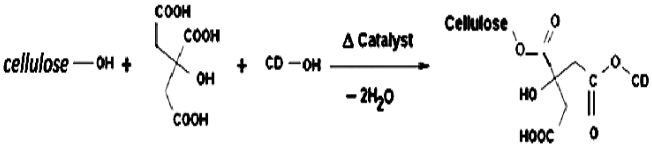

Fig. 1. Schematic reaction of β-cyclodextrin molecule with cotton cellulose

When cyclodextrin grafted fabric is equilibrated in the aqueous solution of $Mg(NO_3)_2$ magnesium ions enter into internal hydrophobic cavity and accumulate there. Since the diameter of internal cavity of β-cyclodextrin is around 7-8 Å, the probability of encapsulation of Mg(II) ions with hydrodynamic radius of 1.15 Å cannot be ruled out.

FTIR spectral analysis: A spectrum of β-cyclodextrin shows a peak of -OH stretching vibration observed at 3597 cm^{-1} and a strong and complex band at 1260-1000 cm^{-1}, which is due to stretching vibration of -C-O. Spectrum of reference fabric shows a broad peak corresponding to -OH stretching of hydroxyl group of cellulosic cotton fabric is observed in the range 3800-3400 cm^{-1} and sharp peak for -OH bending vibrations of cellulosic fabric is observed at 1559 cm^{-1}.

A broad peak of -OH in-plane bending vibration occurred in the general region of 1352-1000 cm^{-1} for the plain fabric. The asymmetric and symmetric C-H stretching are obtained in the range of 2906 cm^{-1} and 2860 cm^{-1} respectively. The FTIR spectrum of cyclodextrin-grafted-fabric, a broad peak at 3600-3400 cm^{-1} is observed, which is due to the presence of -COOH group of citric acid and -OH group of cellulose and in addition, sharp peak of -OH bending of cellulosic fabric at 1651 cm^{-1} is also observed at 1599 cm^{-1}. A broad peak of -OH in-plane bending vibration usually occurred in region of 1420-1000 cm^{-1} is observed at 1360-1042 cm^{-1} due to the attachment of cyclodextrin with oxygen moiety of hydroxyl group of cellulosic fabric in cyclodextrin grafted fabric. However, in the spectrum of Mg(II) loaded grafted fabric, a broad band of 3600-3400 cm^{-1} disappears, of cyclodextrin grafted fabric, which may probably be due to the binding of magnesium ions with electron rich oxygen atoms present in the internal cavity of β-cyclodextrin.

Scanning electron microscopy: The surface morphology of the reference and cyclodextrin-grafted fabrics. Fig. 2 indicates that the surface of plain or ungrafted fibres has relatively rough texture while after grafting of cyclodextrin a smooth surface is obtained. This is an indication of presence of micrometer sized grafted thin layer onto the fibre surface. The EDAX image confirms the loading of Mg ions onto cyclodextrin grafted fabric of cellulosic fabric.

Antibacterial properties of Mg(II) ions loaded fabric: β-cyclodextrin grafted fabric and Mg (II) ions loaded grafted fabrics were tested for *S. aureus* and *E. coli*. The results depict the zone of inhibition in fabrics. β-Cyclodextrin grafted fabric shows minimum of inhibition due to the presence of citric acid as crosslinker, while comparing Mg(II) ions loaded fabric with β-cyclodextrin grafted fabric. The former shows excellent inhibitions against *S. aureus* and *E. coli*. Inhibitory effect is more pronounced in *S. aureus* when the fabrics are repeatedly

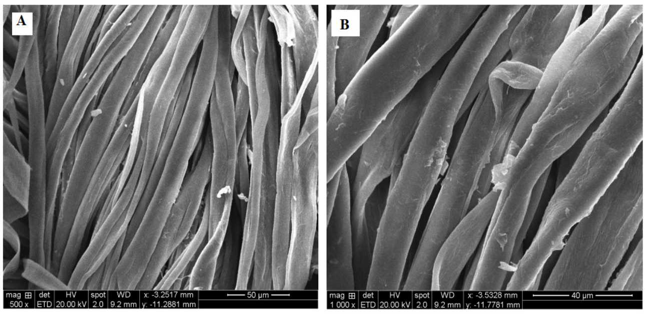

Fig. 2. Scanning electron micrograph of (A) reference and (B) Mg ions-loaded grafted fabric

washed and their activity was measured (Table-1). It is observed that the antibacterial efficiency is retained after repeated washing cycle due to entrapping of Mg(II) ions in β-cyclodextrin cavity.

TABLE-**1**
ANTIBACTERIAL ASSESSMENT BY AGAR DIFFUSION METHOD

Microorganimsm	*S. aureus* (Gram positive)	*E. coli* (Gram negative)
	Zone inhibition diameter (mm)	
Standard	17	17
Grafted fabric	32	26
Mg (II) ions grafted fabric	36	32

Conclusion

It is concluded that Mg(II) ions can be conveniently loaded into the internal cavities of cyclodextrin-linked cotton fabric for antibacterial applications. FTIR results confirmed the grafted cyclodextrin fabric at 1360-1042 and disappears in magnesium grafted fabric which may probably due to the binding of magnesium ions. SEM studies reveals the loading of magnesium ions in to the grafted fabric and the EDAX image confirms the presence of entrapped Mg(II) ions. The grafted fabric shows antibacterial action against both *E. coli* and *S.aureus* bacteria's. However gram positive (*S. aureus*) bacteria exhibit much much antibacterial character than gram negative one.

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