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Preparation and Characterization of Nanocomposite Cotton Fabrics (NCCFs) With in Situ Generated Silver Nanoparticles

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Abstract — Nanocomposite cotton fabrics (NCCFs) embedded with in situ generated silver nanoparticles (AgNPs) were successfully prepared using an eco-friendly green synthesis approach. Lemon leaf extract was employed as a natural reducing and stabilizing agent to functionalize cotton fabrics and facilitate the formation of AgNPs within the cotton matrix. The incorporation of phytochemicals from the extract enabled effective reduction of silver ions and strong adhesion of nanoparticles onto the cellulose fibers. The synthesized NCCFs were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM) coupled with energy dispersive X-ray analysis (EDX), Fourier transform infrared spectroscopy (FTIR), and thermogravimetric analysis (TGA). XRD results confirmed the presence of crystalline silver nanoparticles along with cellulose structure, while SEM analysis revealed uniformly distributed spherical AgNPs with sizes increasing with higher precursor concentration. EDX analysis verified the elemental composition of silver within the fabric. FTIR studies indicated that the fundamental structure of cotton remained intact, with interactions between AgNPs and functional groups occurring primarily through electrostatic forces. TGA results showed a slight reduction in thermal stability of NCCFs compared to the untreated cotton matrix. The antibacterial activity of NCCFs was evaluated against Gram-positive (Staphylococcus aureus, Bacillus subtilis) and Gram-negative (Escherichia coli, Pseudomonas aeruginosa) bacteria using the agar diffusion method. The prepared nanocomposites exhibited significant antibacterial performance, with inhibition zones ranging from 11 to 25 mm, whereas untreated cotton fabrics showed no antibacterial activity.

Keywords— Nanocomposite Cotton Fabrics (NCCFs), Silver Nanoparticles (AgNPs), In Situ Synthesis, Cotton Fabric, Nanocomposites, Fabric Characterization, Antibacterial Activity, Antimicrobial Textiles, Surface Modification, Nanotechnology.

I. INTRODUCTION

Metal Nanoparticles (MNPs) have intent of many significant and branch out many applications [1,2]. The general physical and chemical methods of synthesis of nanomaterials are not eco-friendly in nature [3]. Nanotechnology has been revolutionizing many fields of research including photonics, medicine, catalysis, photo electricity and industrial manufacture etc. The necessity for the progress of materials which withstand antimicrobial properties has focused different studies around Metal Nanoparticles (MNPs) and their incorporation into Cotton fabrics.

Pure Ag exhibits the thermal conductivity and highest electrical among all the metals and the lowest contact resistance [4]. The preparation of Ag based nanoparticles (AgNPs) is offering a range of applications. The significance of silver nanoparticles keeps on increasing, due to their broad range of applications in diversified fields like medicine, sensors, antibacterial agents and catalysts and so on. AgNPs have different physical, chemical and biological properties compared to their bulk counterpart. The properties of metal nanoparticles pertaining to optical, thermal and catalytic properties are size dependent in general and for silver nanoparticles in particular. In addition, the AgNPs have a significant antibacterial activity against wide spectrum of bacterial and fungal species. Due to this, the AgNPs systems are most extensively used as sterilizer in medical products, textiles, food storage bags and personal care products. They have been synthesized by different methods such as chemical [5], electro chemical [6], radiation [7], photochemical [8], Langmuir – Blodgett [9] and biological methods [10]. In this study, an attempt was made to build up NCCFs @ AgNPs by using cotton matrix infused with Lemon leaf extract as reducing and stabilizing agent.

II. MATERIALS

1. Preparation of Lemon Leaf extract

Citrus lemon belongs to Rutaceae which is widely grown commercially worldwide. The fresh green Citrus lemon leaves were collected in early hours of morning. In order to get rid of non-adhered impurities on leaves, thoroughly washed using deionised water then dried in shade.

A quantity of 100 grams of the dry leaves was soaked in 900 ml of preheated deionised water (80˚C) by water bath method, for 20 minutes duration. After cooling to ambient temperature, extract was filtered which may be readily usable or can be stored at 5 oC till used to avoid fungus formation. This lemon leaf extract as plant extract contains phytochemicals consisting of active alkaloids, phenols, terpenoids, quinines, amides, flavonoids, proteins and alcohols etc., [10].

2. Preparation of Cotton Matrix

The pristine cotton fabrics had been procured from local market (Hyderabad, India). The cotton fabric was washed with deionised water and dried in shade. The cleaned cotton fabric was cut into dimensions of 150mm ×100mm. These pieces of cotton fabrics were immersed along the inside walls of glass beakers which contain lemon leaf extract. These systems were then placed on magnetic stirrer at room temperature and maintain stirring rate 300rpm for 24 hours at room temperature. A fine layer of citrus lemon leaf extract i.e., phytochemical layer, strongly adhere to cotton fabric by molecular forces [11]. During this process, cotton fabrics colour turned into light greenish which representing infusion of phytochemicals of lemon leaves extract. Then cotton fabric was separated and rinsed many times with deionised- water until all non-adhered organic-impurities of leaf extract were removed and subjected to shade dry. The lemon leaves extract infused Cotton Fabrics were employed as Cotton Matrix.

3. Ag Source Solution Preparation (AgSS)

AgNO3 source solutions of 1mM-5mM were prepared as per standard procedures. AgNO3 (M.W.169.87) crystals are readily dissolve in de-ionized water. The prepared Ag source solutions (AgSS) may be employed to form in situ AgNPs. To prevent photo-oxidation of silver, beakers containing AgSS were covered with a tin foil lid.

4. Cotton Matrix-In situ generation of AgNPs

The systems of Ag source solutions with different concentrations i.e., 1mM, 2mM, 3mM, 4mM and 5mM were prepared in separate beakers. In each beaker a piece of Cotton Matrix was immersed in the Ag source solution along the inner side walls of beaker. These systems were then placed on magnetic stirrer at room temperature and maintain stirring rate 300rpm for 24 hours. During this process, the cotton matrix colour turned from light greenish to ochre, primarily which may be due to incorporation of in situ generated Ag Nanoparticles onto Cotton Matrix which is designated as NCCFs with in situ formed AgNPs (NCCFs @ AgNPs). In NCCFs, infused lemon leaf extract reduce Ag ions into AgNPs, i.e., in situ generated AgNPs, and impregnated onto the cotton fabric by electrostatic interaction between the AgNPs and - OH groups of cellulosic chain of cotton matrix [12] . The NCCFs having in situ formed AgNPs were thoroughly washed with deionised-water and dried. NCCFs colour was retained even after repeated washings.

III. RESULTS AND DISCUSSIONS

Characterization

The physical appearance of Cotton Fabric, Cotton Matrix, NCCF @ AgNPs were recorded by using Sony digital Camera. The X-ray diffractograms were recorded using an X -ray diffractometer (Brucker D8 Advance at 40 kV & 25 mA) in the range of 2θ = 100 to 800. The SEM images of the NCCFs, were recorded employing Zeiss EVO 18 instrument operated at 10kV and also EDX recorded for confirmation. The spectra of Cotton fabric, matrix and NCCFs were recorded employing a FTIR spectrometer (Smart iTR ATR Nicoet iS10) 4000-600 cm-1 in the wavenumber region (32 scans and 4 cm-1 resolution). For the study of thermal stability of matrix, NCCFs @ AuNPs, thermograms were obtained employing Thermo gravimetric analyzer (Perkin Elmer Model STA 6000) in 300-7000C range at 100C per minute heating rate in nitrogen inert atmosphere. The antibacterial activity of NCCFs was studied by an agar diffusion method [16]. The clearance of zones formed against Gram-positive (Staphylococcus aureus, Bacillus subtilis) as well as Gram-negative (Pseudomonas, Escherichia coli) bacteria were photographed. Diameters of zones of inhibition were measured and tabulated.

Physical appearance - Digital images of NCCFs @ AgNPs

To notice physical appearance of NCCFs prepared using different concentrations of source solutions, their digital images were recorded. For comparison, the images of the cotton fabric and the cotton matrix were recorded. The digital images of the cotton-fabric, cotton-matrix and NCCFs using 1mM, 2mM, 3mM, 4mM and 5mM Ag-source solutions are presented in Fig.1 (a), (b), (c), (d), (e), (f) and (g) respectively.

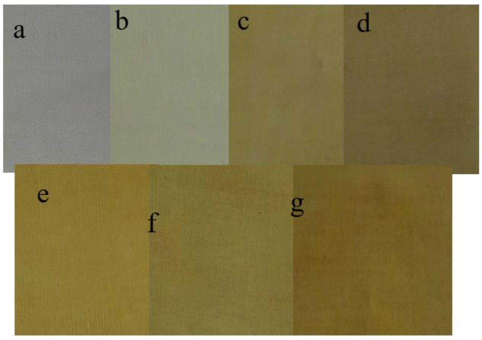

Fig.1 Physical Appearance of digital images of cotton fabric (a), cotton matrix(b),

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NCCFs@AgNPs with AgSS of 1mM(c), 2 mM(d), 3 mM(e), 4 mM(f) and 5 mM(g)

Fig.1 revealed that the colour of matrix is light greenish, while the colour of the NCCFs changed to ochre. The change in colour of NCCFs when compared to that of matrix primarily indicates in situ generation of silver nanoparticles.

2. X-ray diffraction Studies

To find effect of silver nanoparticles on crystalline stage of cotton matrix, X-ray diffraction studies were conducted. X- ray diffraction study of cotton-fabric, cotton-matrix and NCCFs @ AgNPs with 1mM,2mM,3mM,4mM and 5mM Ag source solutions(AgSS) were depicted in Fig.2(a), for lucidity, diffractograms of cotton-fabric, cotton-matrix and NCCFs with in situ formed silver nanoparticles by means of 5mM source solutions were depicted in Fig. 2(b)

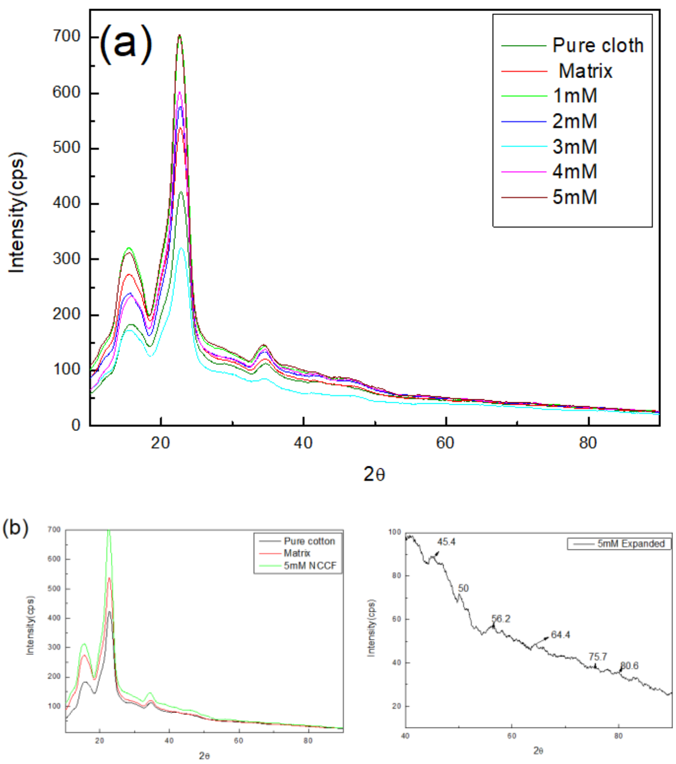


Fig. 2(a) X-ray diffractions of Cotton fabric, Cotton matrix, NCCFs@AgNPs with AgSS of 1mM-5mM, Fig. 2(b) Cotton fabric, cotton matrix and NCCFs@AgNPs with AgSS of 5mM

Fig. 2(a) and (b), reveals three intense peaks at 2θ= 15.5o, 22.6o and 34.5o which are correspond to cellulose cotton fabric arose owing to the diffractions from (110), (200) planes of cellulose-I structure [10]. The other peak at 2θ= 34.5o arose owing to the diffraction from (004) plane of cellulose structure-II [11].

In addition to those main peaks, there exist very indistinct peaks which are concealed by the intense peaks. In order to observe these peaks, diffractogram of NCCF with in situ formed silver nanoparticles using 5mM AgSS was extended in 2θ= 40o to 90o. In those peaks observed at 2θ= 45.4o, 64.4o and 77.3o correspond to the diffractions from (200), (220) and (311) of silver nanoparticles [12]. While other found at 2θ= 56.2o arose due to the diffraction (220) plane of AgO nanoparticles [13]. Thus, the NCCFs had both AgNPs and AgO. This is predictable as silver is a strong oxidizing agent, some of them oxidized to AgO.

3. SEM & EDX Analysis

To observe the in situ formed AgNPs in the NCCFs using different concentrated Ag source solutions, scanning electron microscopy analysis was made. Scanning electron micrograms of the NCCFs using 1mM to 5mM of silver source solutions were shown respectively in Fig.3 (a)-(e). Corresponding EDX spectra of the NCCFs employing 1mM and 5mM solutions are presented respectively in Fig. 4 (c) and (d).

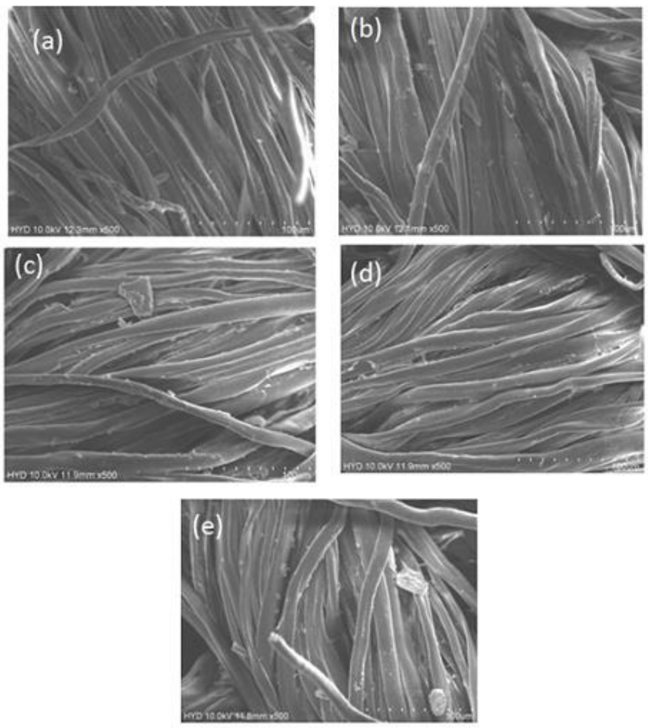
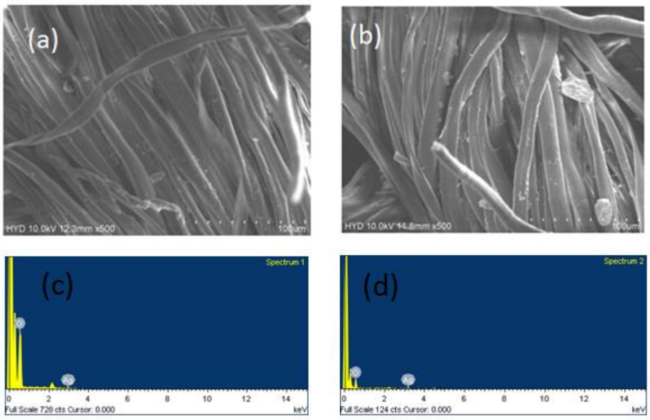
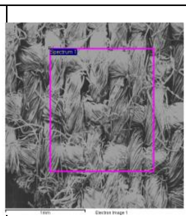


Fig.3 SEM images of AgNPs due to different concentrations of Ag source solutions (a) 1mM, (b) 2mM, (c) 3mM, (d) 4mM and (e) 5mM



Element	Weight%	Atomic %	
			
O K	51.83	92.71	
Ag L	2.45	0.65	
Au M	45.73	6.64	
Totals	100.00		

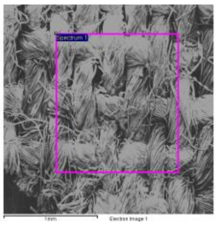
Element	Weight%	Atomic%	
			
O K	94.56	99.15	
Ag L	5.44	0.85	
Totals	100.00		

Fig.4 SEM images of AgNPs due to (a) 1mM, (b) 5mM Ag Source solutions and EDX spectra and Elemental Composition of NCCFs with in situ formed Ag NPs using (c)1mM and (d) 5mM Ag Source solution.

It was estimated that the NCCFs prepared using 1mM to 5mM source solutions had the spherical nanoparticles in the size of 70nm to 120nm range. The average size of formed AgNPs increased with augment in concentration of silver source solution and more AgNPs were generated with increased concentration of Ag SS. The presence of the Ag element in NCCFs was established by EDX spectrum. Thus, the SEM analysis indicated that the prepared NCCFs on employing lemon leaf extract act as effective bio-reductant, formed spherical AgNPs.

4. FTIR Spectroscopic Studies

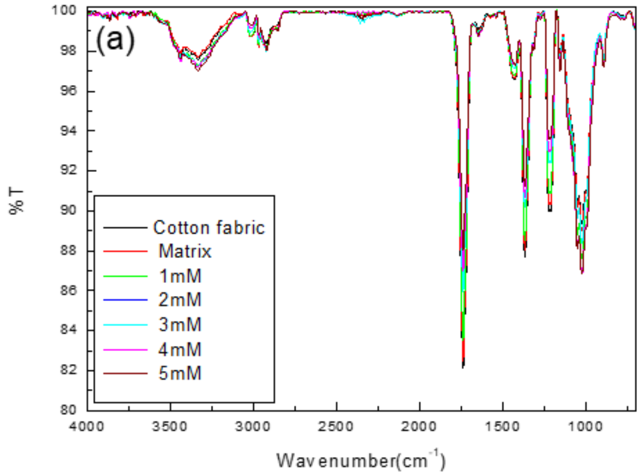


Fig. 5(a) The FTIR spectra of cotton fabric, matrix and the NCCFs with in situ formed silver nanoparticles using 1- 5mM AgSS

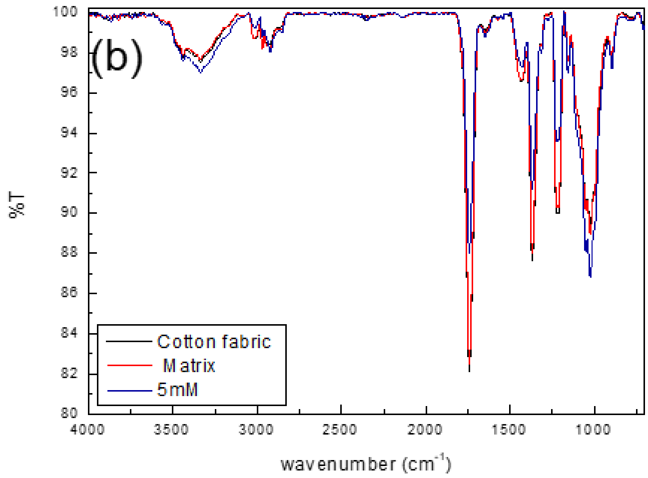


Fig. 5(b) The FTIR spectra of cotton fabric, matrix and the NCCFs with in situ formed AgNPs using 5 mM AgSS.

To explore the interactions between the formed silver nanoparticles and cotton matrix within NCCFs, FTIR studies were conducted. The FTIR studies on cotton fabric, cotton matrix and NCCFs with insitu formed silver nanoparticles using 1mM, 2mM, 3mM, 4mM and 5mM of AgSS were presented in Fig.5(a)

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For Clarity, FTIR studies of cotton fabric, cotton-matrix and NCCF @ AgNPs with 5mM AgSS were depicted separately in Fig. 5 (b). The FTIR spectra of cotton-fabrics, matrix and NCCF @ AgNPs were similar but variations in intensity persist. From Fig. 5 (b), it is apparent that intensity of cotton-fabric and cotton-matrix is same. The peaks observed at 3334 cm-1 and 1739 cm-1 were assigned to the intermolecular -OH and C=O groups. The other three bands appearing at 1434, 1367 and 661cm-1 were owing to CH vibrations. The other two bands appearing at 1223cm-1 and 1018 cm-1 were owing to =C-O-C vibrations and other band at 881 cm-1 to C-C out of plane vibrations. Thus, formed silver nanoparticles did not show any change in structure of cotton matrix but exhibiting electrostatic interactions among components. These interactions of electrostatic nature assist to stabilize and cap formed silver nanoparticles within NCCFs.

5. Thermogravimetric studies

To study effect of generated AgNPs on thermal stability of NCCFs, the thermogravimetric studies were conducted. The primary thermograms of cotton-matrix and NCCFs were shown respectively in Figure 6(a) and 6(b).

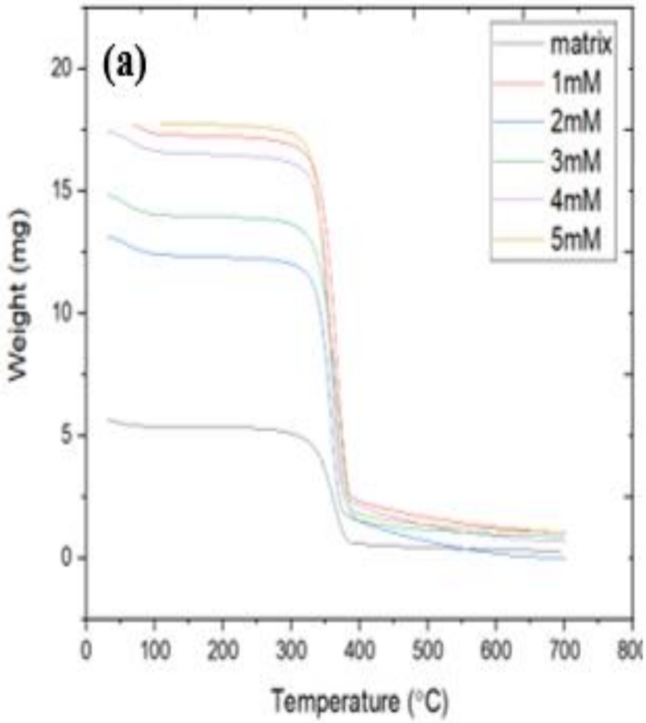

Fig. 6(a) Overlapped Primary thermograms of Matrix and NCCFs with different AgSS and

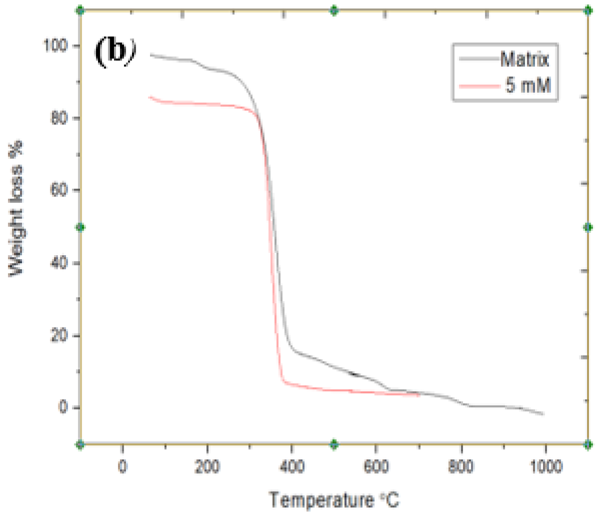

Fig. 6(b) Primary thermograms of matrix and NCCF with 5mM source solution.

The inflection temperature of the cotton matrix is 3880C and inflection temperature of 5mM of AgSS is 3750C. The thermal stability of NCCFs @ AgNPs using 5mM AgSS was found to be lower than that of cotton matrix.

6. Antibacterial activity analysis

Silver annihilates disease causing pathogenic bacteria [15,16]. To investigate antibacterial activity of NCCFs with in situ formed silver nanoparticles against Gram-positive ((Bacilussubtilis and Staphylococcus aureus) and Gram-negative (Escherichia coli and Pseudomonas aeruginosa) bacteria was carried out. The antibacterial activity were also done for cotton fabric (C1) and (C2). The NCCFs made using the different concentrations of Ag source solutions are indicated as 1mM (1), 2mM (2), 3mM (3), 4mM (4), and 5mM (5).

The inhibition zones of NCCFs against gram-positive and gram-negative bacteria were presented in Fig.7. The measured diameters of inhibition zones were tabulated. From Fig.7 and Table 1, it is obvious that cotton-fabric and cotton-matrix did not reveal any clear zones demonstrating their incapacity to inhibit the growth of the bacteria. However, NCCFs with in situ generated AgNPs exhibit clear inhibition zones with diameters ranging from 11 mm to 25 mm indicating good antibacterial activity. Hence, generated Ag NPs on NCCFs showed fine antibacterial activity and were in accordance with published literature[17,18].

Hence, antibacterial NCCFs with in situ formed AgNPs are useful for making medical fabric for bandage, wound cleaning, dressing, surgical aprons, bed materials for hospitals and napkins etc.

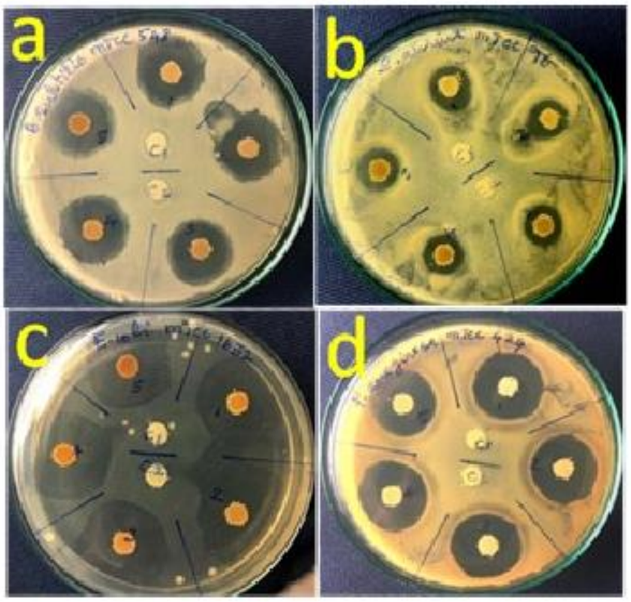

Fig.7 Zones clearance of the NCCFs with in situ formed AgNPs using Lemon leaf extract as a reducing agent against (a) Bacilussubtilis and (b) Staphylococcus Aureus and (c) Gram negative (Escherichia) and (d) Gram Positive (Pseudomonas aeruginosa) bacteria.

Table 1: Diameters of the clear zone of inhibition for white cloth, matrix and the NCCFs made using 1mm, 2mM, 3mM, 4mM and 5mM Ag source solutions against different bacteria

Sample	Diameter of Zone of Inhibition (mm)			
	B. Subtilis (mm)	*S. aureus* (mm)	*E. coli* (mm)	*P. aeruginosa* (mm)
White cloth (C1)	0	0	0	0
Matrix (C2)	0	0	0	0
NCCFs@AgNPs 1	19	12	24	19
2	19	11	25	21
3	17	11	25	20
4	18	11	24	20
5	19	12	22	19

IV. CONCLUSIONS

The studies of XRD, SEM with EDX confirms Nanocomposite Cotton Fabrics within situ formed Ag nanoparticles by lemon leaf extract as bioreductant. The FTIR studies of cotton matrix and NCCFs @ AgNPs were similar except intensity changes among spectra of matrix and NCCFs. The AgNPs formed in NCCFs were with size between 51 to 60nm. The NCCFs @ AgNPs showed fine antibacterial activity against Gram-positive and Gram-negative and these NCCFs @ AgNPs can be find applications of antibacterial apparels and hospital bed materials. Thus, antibacterial NCCFs with in situ formed AgNPs are useful for making medical fabric for bandage, wound cleaning, dressing, surgical aprons, bed materials for hospitals and napkins etc.

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