Chiang Mai J. Sci. 2012; 39(4) : 712-722
http://it.science.cmu.ac.th/ejournal/
Contributed Paper

Preparation Characterization and Antimicrobial Activity of Electrospun Nanofibers from Cotton Waste Fibers

Piyaporn Kampeerapappun
Division of Textile Chemical Engineering, Faculty of Textile Industries
Rajamangala University of Technology Krungthep, Bangkok 10120, Thailand.
Author for correspondence; e-mail: piyaporn.k@rmutk.ac.th

Received: 16 November 2011
Accepted: 24 May 2012

ABSTRACT

In this study, cellulose electrospun nanofibers were prepared directly from cotton waste fiber solution via electrospinning at room temperature. The effects of solution concentration and applied voltage on morphology of electrospun nanofibers were investigated. It was observed that the fiber diameter was strongly affected by the concentration of polymer solution and applied voltage. Analysis of the fourier transform infrared spectroscopy (FT-IR) spectra demonstrated that cotton fibers and cellulose electrospun nanofibers showed the same characteristic functional groups. X-ray diffraction (XRD) revealed that the crystallinity of the cellulose was reduced after electrospinning. Then, an antimicrobial agent solution, 3-(trimethoxysilyl) propyldimethyl octadecyl ammonium chloride (AEM), was treated on cellulose electrospun nanofiber mats. All treated electrospun nanofiber mats showed better uniformity and % extraction compared with untreated mats. These electrospun nanofiber mats exhibited excellent antibacterial activities against *Staphylococcus aureus* (*S. aureus*) from 2% w/w antimicrobial agent.

Keywords: electrospinning, cotton, antimicrobial property, nanofibers

1. INTRODUCTION

Electrospinning is one of the methods used to create nanofibers from polymer melts and solutions. Electrospinning uses electrostatic forces instead of mechanical forces to produce fine fibers. When a high voltage is applied to a polymer droplet, it becomes an electrically charged polymer jet, which solidifies and collects as an interconnected web of small fibers. Both natural and synthetic polymers can be used as raw material to electrospun into nanofibers. Electrospun nonwoven fabrics are used in variety applications such as specialty filters [1], composite reinforcement [2], protective clothing [3], tissue engineering [4], wound dressing [5] and catalytic application [6].

Cotton is the most popular natural fibers used textile fiber in the world today. It is harvested from the cotton plant composed of 88-96% cellulose; the remainder is waxes, protein, and pectin materials [7]. This fiber is spun into yarn and used to make soft and breathable textile.

Several step processes are required for converting cotton plants to cotton fabric such as opening, blending, carding etc. During step processes, over 25% cotton fibers are lost as waste [8]. Typically, cotton waste is used for low value products such as cotton balls and cotton batting. In this research, we tried to convert cotton waste into higher-value products by using electrospinning technique.

Generally, cellulose cannot be dissolved in common solvents due to its highly crystalline structure [9-10]. However, several research groups studied the solvents for cellulose electrospinning and found it required a high temperature [11], concentrated salts [12] and need post-spun treatment of nanofibers [13]. In addition, with increasing public awareness of the potential threat of spreading diseases, demand for antimicrobial fabrics in domestic and international markets has grown significantly in recent years. Bio-protective clothing such as medical, protective garments, institutional and hygienic textiles are the main applications of antimicrobial fabrics [14]. The aim of the work is to study effect of electrospinning parameters on the morphology of cellulose nanofibers by using trifluoroacetic acid as a solvent. Moreover, antibacterial, chemical, and physical properties of cellulose electrospun nanofibers were investigated.

2. MATERIALS AND METHODS

2.1 Materials

Cotton waste fibers were supplied by Noparat Handicraft, Thailand. Trifluoroacetic acid was purchased from Fluka Chemika, Switzerland. Antimicrobial agent, 3-(trimethoxysilyl) propyldimethyl octadecyl ammonium chloride (AEM 5707: 7% in water) was donated from Union Compound Co. Ltd., Thailand. Two non-ionic wetting agents, Diadavin® and DOW CORNING® Q2-5211, were supplied by Bayer Thai Co., Ltd and Dow Corning (Thailand) Limited, Thailand, respectively. All other chemicals were of analytical reagent grade and used as received, without further purification.

2.2 Pretreatment of Cotton Waste Fibers

Cotton waste fibers were scoured and bleached with a solution containing hydrogen peroxide (50%) 6 g/L, sodium hydroxide 5 g/L, sodium silicate as a stabilizer 2 g/L, and Diadavin non-ionic wetting agent 1 g/L with liquor ratio of 1:30. The condition was used at 100 C for 30 min. Then, the fibers were rinsed with hot and cold water for 15 minutes each, respectively. The fibers were dried at 60 C for 12 h before preparing cotton waste solution.

2.3 Preparation of Untreated and AEM Treated Cellulose Elctrospun Nanofiber Mats

The concentration of cotton waste solution was prepared from 0.5-2% w/w by using trifluoroacetic acid as a solvent. The mixture was stirred with magnetic bar at room temperature until it was completely dissolved. Each solution was subjected to viscosity measurement using RV-7 Brookfield viscometer (Brookfield Engineering Laboratories, Inc). After that, prepared solutions were transferred to 5 ml syringe equipped with 21 gauge stainless steel needle (outer diameter (OD) = 0.82 mm and inner diameter (ID) = 0.51 mm) as an electrode. The syringe needle was connected to the positive output of a CRT50-10P high voltage power supply operating in the range of 15 to 25 kV (Gamma High Voltage Research, USA) and the solution was fed into the needle at fixed

rate of 0.4 ml/h by a NE-300 syringe pump (National Direct Network Co., Ltd, USA). The counter electrode was connected to a plate covered by aluminum foil to collect the electrospun nanofibers. The distance between the needle and plate was 15 cm. The electrospinning experiment was performed at room temperature.

To prepare AEM treated cellulose electrospun nanofiber mats, the 1.25% w/w cotton solution in trifluoroacetic acid was used for electrospinning. The electrospinning process was carried out at room temperature at a fixed electrical potential of 20 kV over a fixed distance of 15 cm. The solution feed rate was also fixed at 0.4 ml/h. A 2 ml of AEM solution was sprayed on cellulose electrospun nanofiber mats, area 23×28 cm^2, by HD-470 air brush spray gun with nozzle diameter of 0.2 mm (Guangzhou Pengcheng Cosmetology Firm, China). The concentration of AEM solution was varied from 0-7% w/w in water. Additionally, the distance between the spray gun and electrospun nanofiber mats was 120 cm. After that, the electrospun nanofiber mats were dried in oven at 40□C for 20 min.

2.4 Scanning Electron Microscope (SEM)

The morphologies of electrospun nanofibers were obtained using a 4510-Jeol scanning electron microscope (Jeol Ltd., Japan). Fiber diameters of electrospun nanofibers were measured by means of the ImageJ software from the SEM micrographs in their original magnificent.

2.5 Fourier-transform Infrared Spectroscopy (FT-IR)

The chemical structures of cotton fibers and cellulose electrospun nanofibers were characterized by Spectrum One Fourier-transform infrared spectroscopy (PerkinElmer Ltd., USA). The transmittance of each sample was recorded between 4000 and 450 cm^{-1}.

2.6 X-ray Diffraction (XRD)

X-ray diffraction patterns of cotton fibers and cellulose electrospun nanofibers were performed with a Bruker D-8-Discover diffractometer operating with Cu-Ká radiation source (l= 0.1542 nm). The scanning range and the scanning speed were 5-40% and 5 deg/s, respectively.

2.7 Uniformity and % Extraction

To test uniformity and % extraction, cellulose electrospun nanofiber mats were dyed with bromophenol blue (BPB) according to direct stain assays and BPB extraction. For uniformity, the 0.5 g of mat was dyed with 0.025% w/v bromophenol blue in water for 20 min at ambient temperature. The specimens were rinsed several times with water. The blue stain was then compared with pre-determined color standard. For % extraction, a mixture of the 0.001% w/v bromophenol blue in water and 0.01% w/v Q2-5211 wetting agent solution was prepared and measured at 595 nm with Aquamate UV-Vis spectrophotometer (Thermo Fisher Scientific Inc., USA) as the initial absorbance. A 0.5 g of mat was dyed with the solution for 20 min at ambient temperature. After that, the mat was taken out and remaining solution was tested at 595 nm absorbance as a final absorbance. The % extraction was calculated as followed:

$$\left[\frac{\text{Initial Absorbance-Final Absorbance}}{\text{Initial Absorbance}}\right] \times 100 = \text{\% Extraction}$$

2.8 Antimicrobial Test

The electrospun nanofibers mats were tested according to SN 195920-1994. Briefly, mats were placed on nutrient agar plates, which were inoculated with

Staphylococcus aureus (*S. aureus*) as the intended test bacteria. An antimicrobial efficiency of the mat was then recognized if the zone on the nutrient agar plate in contact with the specimen (contact zone) was free of bacteria. If necessary, additionally a bacterial free zone (inhibition zone) was formed at the edge of the mat. The antimicrobial efficiency results were compared with evaluation scheme.

3. RESULTS AND DISCUSSION

3.1 Effect of Electrospinning Parameters on Morphology of Cellulose Electrospun Nanofibers

Cotton, a natural cellulosic fiber, contains cellulose and non-cellulosic substances i.e. wax, pectin, and protein. To remove the non-cellulosic materials from cotton fibers, cotton pretreatment are required [15-16]. As a result of pretreatment, cotton fibers contained 99% cellulose. This result can be explained that non-cellulosic substances were removed from cotton fibers and these fibers were suitable for solution preparation.

Prior electrospinning, viscosity of the cellulose spinning solutions were measured and the results are shown in Figure 1. Moreover, to obtain the electrospun nanofibers condition with optimized diameters, the cotton solutions with various concentrations and applied voltages were electrospun. Table 1 shows SEM images of the cellulose electrospun nanofiber mats from the cotton solutions that had been fabricated under solution concentration in the range of 0.5-2.0% w/w and applied voltage in the range of 15-25 kV with a fixed flow rate and collection distance of 0.4 ml/h and 15 cm, respectively.

From Table 1, it was found that the bigger diameter of the nanofibers was obtained by increasing solution concentration. For example, at the applied voltage of 20 kV, the average fiber diameter increased from 78 ± 24 to 127 ± 73 nm for 1.25 to 2.0% w/w cotton solution concentration, respectively. Concentration and molecular weight of the dissolved polymer are related with viscosity of a polymer solution. An increase of the polymer concentration results in increasing the viscosity of polymer solution (Figure 1). During electrospinning, it was obtained that beads were found along fibers at low concentration (from 0.5 to 1% w/w). As

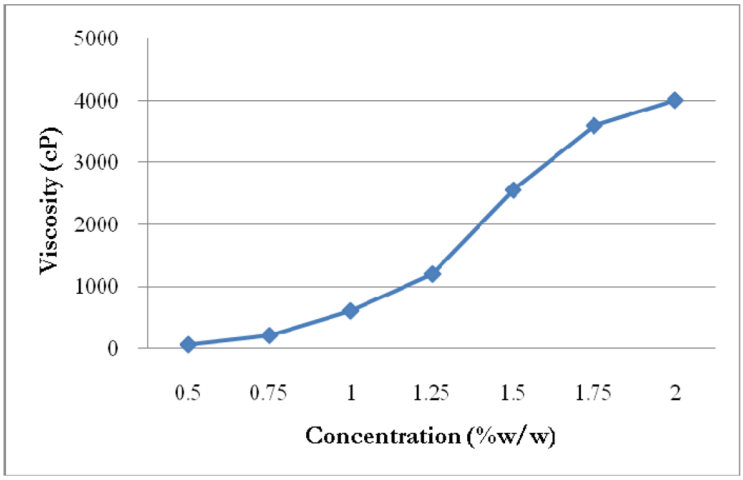
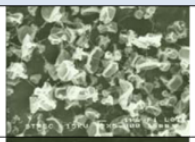
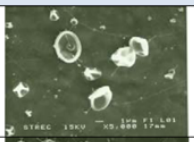
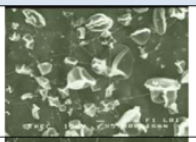
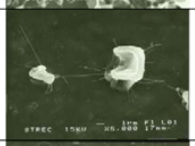
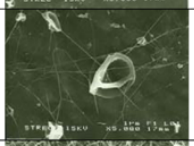
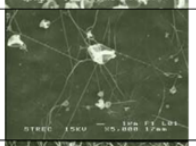
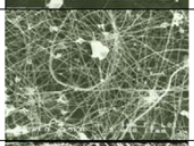
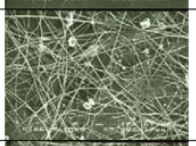
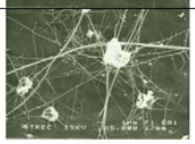
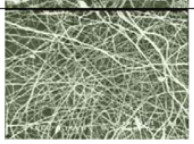
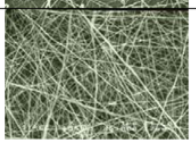
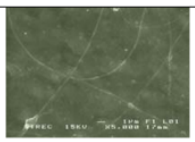
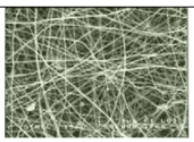
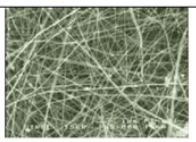
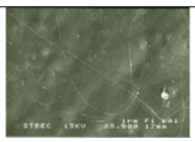
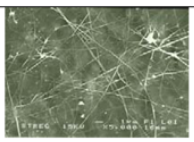
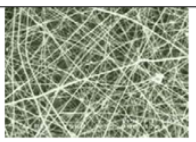
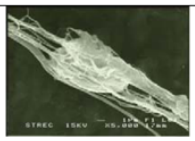
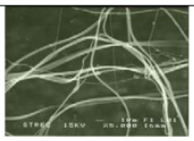
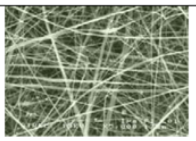


Figure 1. Viscosity values of cellulose solution.

the concentration increases (from 1.25 to 2% w/w), there was a gradual change in the shape of beads from spherical to spindle-like until a smooth fiber was obtained [17-18]. An increase in the solution concentration resulted in the formation of larger diameter fibers. These results were explained that at low concentration, there were few polymer molecule chains in solution resulting in too low entanglement of molecule chains to prevent the electrically driven jet from breaking up thus the discrete spherical droplet was formed. When the concentration of cotton solution was increased, the beads were disappeared due to enough chain entanglement to form a fiber.

At the same solution concentration but vary voltage, smaller diameter of fibers were obtained by increasing the supply voltage (see Table 1). This result indicated that a higher voltage would lead to greater

Table 1. Effect of solution concentrations and applied voltages on electrospun nanofibers morphology.

Conc (% w/w)	Voltage (kV)		
	15	20	25
0.5			
0.75			
1.00			
1.25			
1.50			
1.75			
2.00			

stretching of solution causing reduced diameter of the fibers and encouraged faster solvent evaporation to yield drier fibers. As the results, solution concentration at 1.25% w/w, at which the viscosity values of 1200 cP and supply voltage of 20 kV were found to be the optimal electrospinning conditions for cellulose electrospun nanofibers because they were generated the small and more content of fiber.

3.2 Characterization of Cellulose Electrospun Nanofibers

Cotton waste fibers and cellulose electrospun nanofibers were studied crystallinity and functional group by XRD and FT-IR, respectively. The results were shown in Figure 2-3.

The crystallinity of cotton waste fibers and cellulose electrospun nanofibers was investigated by X-ray diffraction technique. The diffraction of cotton waste fibers showed only three peaks at 2q = 15, 17, and 22.7° which derived from cellulose I [19-20] (Figure 2), while no peak was observed on diffractogram of cellulose electrospun nanofibers. It was indicated that crystalline cellulose was transformed into amorphous cellulose. This phenomenon can be explained that cotton is one of the common sources of cellulose containing a large number of hydroxyl groups [21]. These hydroxyl groups on cellulose unit can form hydrogen bond between the chains resulting in very tightly packed crystallites. In the case of cellulose electrospun nanofibers, cotton fibers would be hydrolyzed with trifluoroacetic acid leading to cellulose decrystallization. The decrease of the degree of crystallinity resulted in the decreased modulus and tensile strength of nanofibers [22].

The FT-IR spectra of both cotton fibers and cellulose electrospun nanofibers are similar (Figure 3). In the spectra of the cellulose, the O-H stretching band at 3550-3100 cm^{-1} was found. Other spectra bands were found at 2917 for C-H stretching, at 1617 cm^{-1} for symmetrical

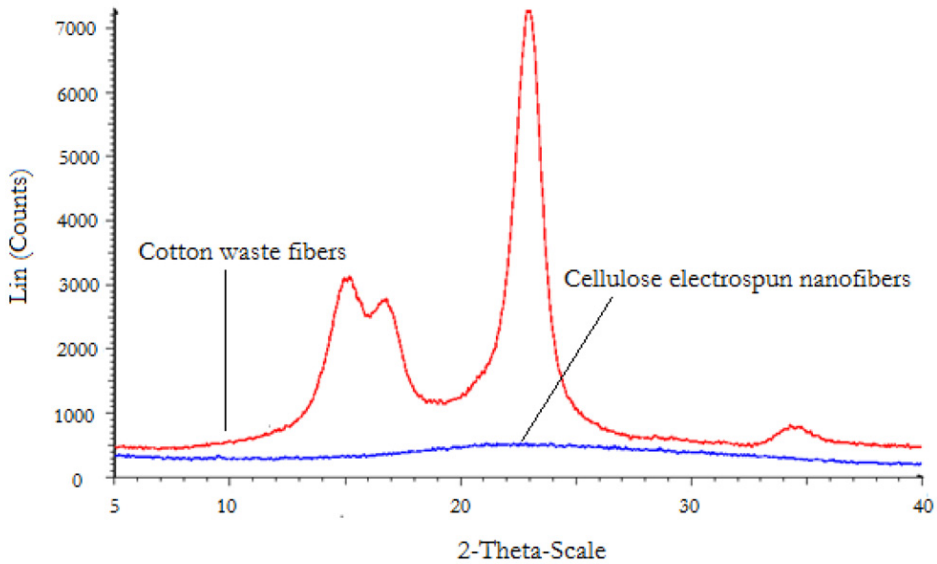


Figure 2. XRD patterns of cotton waste fibers and cellulose electrospun nanofibers.

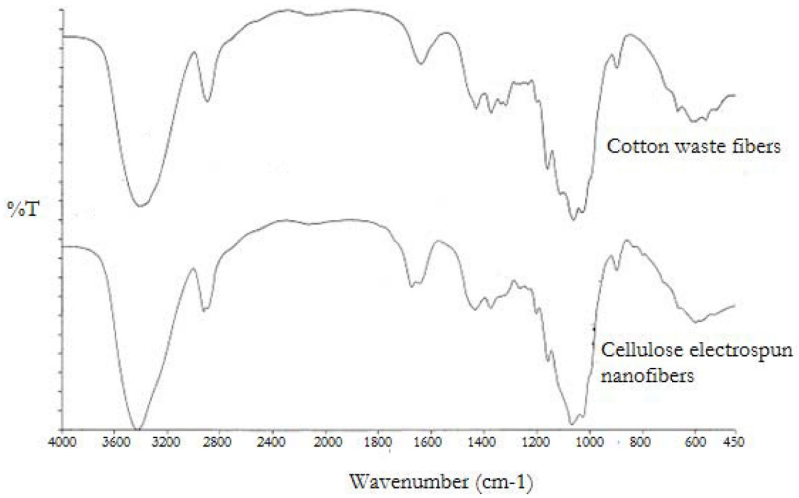


Figure 3. FTIR spectra of cotton waste fibers and cellulose electrospun nanofibers.

COO stretching, at 1316 cm^{-1} for C-H wagging, and at 1060 cm^{-1} for C-O-C absorption [23-24]. The C-F and C=O stretching peaks at 720 cm^{-1} and around 1800 cm^{-1}, respectively [25] was not observed in the spectrum of cellulose electrospun mats. This result implied that cellulose electrospun mats is composed of pure cellulose and TFA did not change functional groups of cellulose.

3.3 Morphology of AEM Treated Cellulose Electrospun Nanofiber Mats

A concentration of 1.25% w/w cotton waste solution was electrospun into nanofiber mats at applied voltage of 20 kV. After that, the electrospun nanofiber mats were treated with 2 ml AEM, 3-(trimethoxysilyl) propyldimethyl octadecyl ammonium chloride, solution at concentration of 0-7% w/w. Morphology of untreated and AEM treated cellulose electrospun nanofiber mats was investigated as shown in Figure 4.

After cellulose electrospun nanofibers were treated with 1-3% w/w AEM solution (Figure 4 (b)-(d)), they all gave the same fiber morphologies compared with untreated electrospun nanofibers (Figure 4 (a)). With an increased concentration of AEM solution (4-7% w/w), the fiber morphologies of electrospun nanofibers

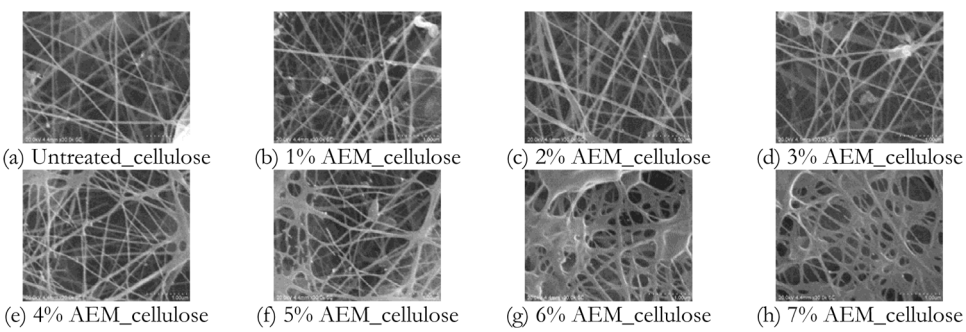


Figure 4. Morphology of untreated and AEM treated cellulose electrospun nanofibers, magnification 30,000×.

were changed. The explanation of these results is based on the fact that cellulose electrospun nanofibers are amorphous cellulose and show a diameter so small (average diameter ~ 78 nm) causing decreased fiber strength compared with original cellulose. The viscosity of AEM solution was found to increase with increasing solution concentration. The higher concentration of AEM solution caused the larger sizes of the droplets [26]. Therefore, when the droplet of AEM solution was dropped on electrospun nanofibers, some nanofibers was fused together; however, it can still be fibers.

3.4 Uniformity and % Extraction

The AEM treated cellulose electrospun nanofiber mats were investigated for uniformity and % extraction by dyeing with bromophenol blue. After dyeing, they were tested using a UV/Vis spectrophotometer. Table 2 shows the results of uniformity and % extraction of the antimicrobial electrospun mats.

From Table 2, it was observed that untreated cellulose electrospun nanofiber mats showed less uniformity than AEM treated cellulose electrospun nanofiber mats, while a comparison among cellulose electrospun nanofiber mats with AEM displayed very good uniformity except at 1% w/w of AEM solution. AEM, a quaternary ammonium salts, is an amphiphilic compound that can chemically bonded on surfaces of nanofiber mats. When chemical covalent was formed, the long chain hydrophobic group of AEM was outward from the surface. Then, the positively charged nitrogen ions of AEM can interact with negatively charged groups such as the sulfonate group of bromophenol blue, an anionic dye [27].

In the case of percent extraction, percent extraction of untreated cellulose electrospun nanofiber mats was a lower percentage than observed in cellulose electrospun nanofiber mats with AEM. A higher percent extraction value corresponded with an increased treatment efficiency of sample. The conclusion for percent extraction was that the higher the amount of AEM, the higher treatment efficiency a sample has.

3.5 Antimicrobial Efficiency

Untreated and AEM treated cellulose electrospun nanofiber mats were tested according SN 195920-1994 standard. The efficiency of antimicrobial electrospun mats against *S. aureus* is shown in Table 3.

From Table 3, free of growth of S. *aureus*

Table 2. Uniformity and % extraction of untreated and AEM treated cellulose electrospun nanofiber mats.

Description	Uniformity	% Extraction
Untreated_cellulose	Poor	32%
1% AEM_cellulose	Fair	73%
2% AEM_cellulose	Very good	95%
3% AEM_cellulose	Very good	97%
4% AEM_cellulose	Very good	>99%
5% AEM_cellulose	Very good	>99%
6% AEM_cellulose	Very good	>99%
7% AEM_cellulose	Very good	>99%

was inhibited with 2-7% w/w AEM treated cellulose electrospun mats. However, bacteria can grow on untreated and 1% w/w AEM treated cellulose electrospun mats. At 2-7% w/w AEM treated cellulose electrospun mats, all samples did not exhibit a zone of inhibition because AEM is a non-leaching antimicrobial agent. Non-leaching antimicrobial agent inhibits or destroys bacteria growth resulting in a durable and specific area that is non-toxic to the environment [28]. Antimicrobial ability of AEM is a result of its amphiphilic structure and surfactant properties. First, the long alkyl groups face away from the cell. Then, the charged nitrogen molecule and long hydrophobic alkyl chains interact with the phospholipid bilayer of the cytoplasmic membranes causing a phase separation of the hydrophobic and polar regions. As a result, weakness and leakage of the cytoplasmic membrane is caused by these compounds, which leads to a disruption of the permeability and instability of the membrane [29]. During inactivation, AEM remains intact and retains all original properties as long as they are attached to polymers [30].

Table 3. Efficiency of antimicrobial electrospun mats against *S. aureus*.

Sample	Results
Untreated_cellulose	NZ/NI
1% AEM_cellulose	NZ/NI
2% AEM_cellulose	I/Iz < 1 mm
3% AEM_cellulose	I/Iz < 1 mm
4% AEM_cellulose	I/Iz < 1 mm
5% AEM_cellulose	I/Iz < 1 mm
6% AEM_cellulose	I/Iz < 1 mm
7% AEM_cellulose	I/Iz < 1 mm

Note: NZ = No clear zone
NI = No Inhibition of growth under the sample
I = Inhibition of growth under the sample
Iz = Inhibition zone

4. CONCLUSION

Cotton waste fibers were successfully electrospun into nanofibers using trifluoroacetic acid as the solvent. This process occurred at room temperature and was not required concentrated salts and post-spun treatment. An increasing the concentration of cotton waste fibers solutions caused changing morphology from discrete droplets to a combination of beaded and smooth fibers and finally to completely smooth fibers. In the case of applied voltage, the diameter of nanofibers tended to decrease with increasing electrospinning voltage. The cellulose electrospun nanofibers was decrystalized to amorphous cellulose. However, the functional groups of electrospun nanofibers are the similar to cotton fibers. The electrospun nanofibers treated with AEM solution showed very good uniformity and % extraction compared with their untreated. The more AEM that was added, the more uniformity and % extraction observed. The antimicrobial efficiency of electrospun

nanofibers against S. aureus was very good despite the electrospun containing only 2% w/w antimicrobial agent. In addition, future studies should investigate the mechanical properties of cellulose electrospun mats, such as tensile strength and tear strength.

ACKNOWLEDGEMENTS

This research was supported in part by a grant from Rajamangala University of Technology Krungthep, Thailand. In addition, I would like to thank Mr. Somchai Udon for non-ionic wetting agent.

REFERENCES

[1] Dotti F., Varesano A., Montarsolo A., Aluigi A., Tonin C. and Mazzuchetti G., Electrospun porous mats for high efficiency filtration, *J. Ind. Text.*, 2007; **37**: 151-162.

[2] Zucchelli A., Focarete M.L., Gualandi C., and Ramakrishna S. Electrospun nanofibers for enhancing structural performance of composite materials, *Polym. Adv. Technol.*, 2011; **22**: 339-349.

[3] Lee S. and Obendorf S.K., Developing protective textile materials as barriers to liquid penetration using melt-electrospinning, *J. Appl. Polym. Sci.*, 2006; **102**: 3430-3437.

[4] Lannutti J., Reneker D., Ma T., Tomasko D. and Farson D., Electrospinning for tissue engineering scaffolds, *Mater. Sci. Eng.*, 2007; **C27**: 505-509.

[5] Khil M.S., Cha D.I., Kim H.Y., Kim I.S. and Bhattarai N., Electrospun nanofibrous polyurethane membrane as wound dressing, *J. Biomed. Mater. Res. Part B: Appl. Biomater*, 2003; **67B**: 675-679.

[6] Luepong K., Koombhongse, P. and Kongkachuichay P., Ceria fibers via electrospinning process: the effect of co-solvent, *Chiang Mai J. Sci.*, 2010; **37(1)**: 85-91.

[7] Jeihanipour A. and Taherzadeh M.J., Ethanol production from cotton-based waste textiles, *Bioresource Technol.*, 2009; **100**: 1007-1010.

[8] Yuen C.W.M., Cheng Y.F., Li Y. and Hu J.Y., Preparation and characterization of nano-scale cotton powder, *J. Text. I.*, 2009; **100**: 165-172.

[9] Zhou J., Zhang L. and Cai J., Bahavior of cellulose in NaOH/Õurea aqueous solution characterized by light scattering and viscometry, *J. Polym. Sci. Pol. Chem.*, 2004; **42**: 347-353.

[10] Xu S., Zhang J., He A., Li J., Zhang H. and Han C.C., Electrospinning of native cellulose from nonvolatile solvent system, *Polymer*, 2008; **49**: 2911-2917.

[11] Kulpinski P., Cellulose nanofibers prepared by the N-methylmorpholine-N-oxide method, *J. Appl. Polym. Sci.*, 2005; **98**: 1855-1859.

[12] Kim C., Frey M.W., Marquez M. and Joo Y.L., Preparation of submicron-scale electrospun cellulose fibers via direct dissolution, *J. Polym. Sci. B: Polym. Phys.*, 2005; **43**: 1673-1683.

[13] Kim C.W., Kim D.S., Kang S.Y., Marquez M. and Joo Y.L., Structural studies of electrospun cellulose nanofibers, *Polymer*, 2006; **47**: 5097-5107.

[14] Abo El Ola S.M., Kotek R., White W.C., Reeve J.A., Hauser P. and Kim J.H., Unusual polymerization of 3-(trimethoxysilyl)-propyldimethyloctadecyl ammonium chloride on PET substrates, *Polymer*, 2004; **54**: 3215-3225.

[15] Mitchell R., Carr C.M., Parfitt M., Vickerman J.C. and Jones C., Surface chemical analysis of raw cotton fibres and associated materials, *Cellulose*, 2005; **12**: 629-639.

[16] Karahan H.A. and özdoğan E., Improvements of surface functionality of cotton fibers by atmospheric plasma treatment, *Fiber Polym*., 2008; **9**: 21-26.

[17] Doshi J. and Reneker D.H., Electrospinning process and applications of electrospun fibers, *J. Electrostatics*., 1995; **35**: 151-160.

[18] Fong H., Chun I. and Reneker D.H., Beaded nanofibers formed during electrospinning, *Polymer*, 1999; **40**: 4585-4592.

[19] Jain R.K., Lal K. and Bhatnagar H.L., Thermal, morphological, x-ray and spectroscopic studies on cellulose and its esters, *J. Anal Appl. Pyrol*., 1985; **8**: 359-389.

[20] Oliveira R.L., Barud H.S., Assunção R.M.N., Meireles C.S., Carvalho G.O., Filho G.R., Messaddeq Y. and Ribeiro S.J.L. Synthesis and characterization of microcrystalline cellulose produced from bacterial cellulose, *J. Therm. Anal. Calorim*., 2011; CBRATEC7 Conference Special Issue: 1-7.

[21] Voronova M.I., Petrova S.N., Lebedeva T.N., Ivanova O.N., Prusov A.N. and Zakharov A.G., Changes in the structure of flax cellulose induced by solutions of lithium, sodium, and potassium hydroxides, *Fibre Chem+*., 2004; **36**: 408-412.

[22] Wu S., Zhang F., Yu Y., Li P., Yang X., Lu J. and Ryu S., Preparation of PAN-based carbon nanofibers by hot-stretching, *Compos. Interface*., 2008; **15**: 671-677.

[23] Kataoka Y. and Kondo T., FT-IR microscopic analysis of changing cellulose crystalline structure during wood cell wall formation, *Macromolecules*, 1998; **31**: 760-764.

[24] Ivanova N.V., Korolenko E.A., Korolik E.V. and Zhbankov R.G., IR spectrum of cellulose, *J. Appl. Spectrosc*., 1989; **51**: 847-851.

[25] Ito F., Infrared matrix-isolation spectroscopy of trifluoroacetic acid hydrates, *Chem. Phys*., 2011; **382**: 52-57.

[26] Jung D.S. and Kang Y.C., Droplet size control in the filter expansion aerosol generator, *J. Eur. Ceram. Soc*., 2008; **28**: 2617-2623.

[27] Lu Y., Wang Y. and Zhu X., The removal of bromophenol blue from water by solvent sublation, *Separ. Sci. Technol*., 2001; **36**: 3763-3776.

[28] Elshafei A. and El-zanfaly H.T., Application of antimicrobial in the development of textiles, *Asian J. Applied Sci*., 2011; **4**: 585-595.

[29] Franklin T.J. and Snow G.A., Biochemistry and molecular biology of antimicrobial drug action, Springer Science + Business Media, Inc., 2005.

[30] Kim Y.H. and Sun G., Dye molecules as bridges for functional modifications of nylon: antimicrobial functions, *Text. Res. J*., 2000; **70**: 728-733.