

# Cellulose Submicron Fibers

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## ABSTRACT

In order to manufacture cellulose submicron fibers, electrospinning of cellulose was tried with different solvents. Alpha-cellulose did not dissolve in 6% (w/w) sodium hydroxide/4%urea aqueous solution. Alpha-cellulose solution in 85% phosphoric acid was not spinnable at an applied voltage between 15kV to 25 kV and at a spin length of 4 to 6 inches. Electrospinning of alpha-cellulose in N-methylmorpholine-N-oxide/N-methyl-pyrrolidinone/water solvent mixture could be performed at an applied voltage of 28 kV and at a spin length of seven inches during spinning and at an ambient temperature of 38<sup>0</sup>C. The degree of crystallinity of the cellulose submicron fibers was found to be 37.88%. The number average fiber diameter of cellulose submicron fibers from 1.25% (w/w) cellulose solution in the N-methylmorpholine-N-oxide/N-methyl-pyrrolidinone/water solvent was found to be 207 nm and the number average fiber diameter of cellulose submicron fibers from 2.5% (w/w) cellulose solution in the N-methylmorpholine-N-oxide/N-methyl-pyrrolidinone/water solvent mixture was found to be 243 nm.

## INTRODUCTION

Due to depleting fossil fuels and stress on our ecosystem caused by synthetic polymers, more and more attention is being paid to plant based biopolymers. Cellulose is one such biopolymer, which is the most abundant renewable source of biodegradable polymer. Cellulose is found in the cell wall of almost of all the plants [1]. Cellulosic materials can be recycled and are completely biodegradable. Cotton fiber is one such cellulosic material. Cotton fiber is very soft, absorbs moisture easily, keeps the skin cool and is comfortable to wear.

If cellulose could be made into submicron fibers, it would have greater strength, flexibility, higher surface to volume ratio and will also absorb greater quantity of moisture [2-5]. Cellulose submicron fibers could be used as wound dressing by acting as scaffolds for tissue engineering and reinforcement for

making high strength biodegradable nanocomposites [2-3]. Cellulose submicron fibers are very fine and so a submicron fiber web can act as a filter out the bacteria from air and water.

A fiber having diameter less than 1000 nm is defined as submicron fiber. Submicron fibers have a very high specific surface area e.g. specific surface area for a 500 nm submicron fiber is 10 m<sup>2</sup>/g and specific surface area for a 50 nm submicron fiber is 100 m<sup>2</sup>/g. Incorporation of catalysts into the submicron fibers enhances the reactivity of the catalysts. U.S. Army and the Natick Solider Center recently reported that submicron fiber webs containing catalysts like POM, nano magnesium Oxide, modified β-Cyclodextrin, enzymes like OPAA, and biocides like N-Halamines, have greater oxidative/hydrolyzing activity against wide spectrum of chemical and biological warfare agents [6-9]. Impregnated activated carbon may not be able to remove hazardous low-molecular weight chemical vapors like TICs, but metal organic compounds impregnated in submicron fibers can absorb these vapors to a much greater extent than activated carbon. Submicron fibers may be also used as sorbents, as scaffolds for tissue engineering and as wound dressing [10-12].

Submicron fibers definitely hold great advantage over the microfibers in filtration because of lower drag force, lower pressure drop, higher permeability, higher interception and inertial impaction efficiency, higher particle capture efficiency, higher filter life, good cleanability, and hence, much higher filtration efficiency [13-14]. Aerosol barrier, permeability modeling and testing confirmed the performance advantages of submicron fiber for aerosol barrier materials. Integrating submicron fiber layer to current filter media improves current threshold of filterable particle size barrier [15]. In the fields of filtration, decontamination, and tissue engineering, submicron fiber materials have tremendous advantages over micro-fibrous materials.

Cellulose nanofibers is being produced by *Acetobacter xylinum* strain of bacteria grown in a culture medium containing carbon and nitrogen. The culture medium normally used is Hestrin-Schramm medium but some other mediums too have been tried [16-17]. The bacterial cellulose formed is very fine. They form a random network of fibrils. These fibrils are having a width less than 100nm. The bacterial cellulose has high mechanical strength, is highly biocompatible and is easily biodegradable. Bacterial cellulose can be used for making ultra fine filters, as reinforcement for high strength composites and wound dressings [17].

Researchers have tried to improve the yield, Young's modulus, alpha cellulose content, breaking stress, wet strength, antibacterial and bactericidal properties of bacterial cellulose [16]. Production efficiency of biosynthesized cellulose nanofibers is quite low and consequently, it never had commercial success because of higher cost of production.

Submicron fibers can also be manufactured by a process called electrospinning. In the past decade, electrospinning technology has become one of the most powerful tools to produce submicron fibers. The electrospinning involves supplying a precise constant amount of either a polymer melt or polymer solution, to a capillary or a spinnerette. A constant volumetric polymer flow rate is maintained to the syringe. The size and shape of the droplet depend upon the feed rate, tension and the gravitational forces. Exposing the droplet at the tip of a syringe, to a very high critical electrical voltage between the spinnerette and a grounded flat collector plate develops a charge on the surface of the droplet, which overcomes the surface tension of the drop to elongate the drop and form a very fine continuous polymer jet [18-22]. The polymer jet is stabilized by viscoelastic forces and is drawn down by acceleration towards a grounded collector of fibers [19, 23]. The polymer jet is initially straight due to longitudinal stress. But later on there is a charge relaxation, due to which there is development of instability. The jet near the grounded collector plate breaks in the instability region to form nanofibers [24]. The fibers lay in a haphazard array on the collection device so as to form non-woven fabric.

In order to manufacture cellulose submicron fibers through electrospinning, cellulose has to be dissolved in a solvent. There are many solvents for cellulose and the primary ones are N, N-dimethyl acetamide/lithium chloride (LiCl/DMA), cuprammonium hydroxide solution, tris (ethylenediamine) cadmium

dihydroxide, and dinitrogen tetroxide/dimethyl formamide system (N<sub>2</sub>O<sub>4</sub>/DMF) [25]. Most of these solvents pose danger to lives and environment. According to United States Patent 1787542, describes the use of at least 75% phosphoric acid to dissolve the cellulose at room temperature, whereas US patent 4058411 suggests that 85% concentrated phosphoric acid can fully dissolve cellulose at room temperature [26]. In 1969, Johnson patented N-methylmorpholine-N-oxide as a solvent for cellulose [27]. But the cellulose solution in N-methylmorpholine-N-oxide has tremendously high viscosity. Therefore, US Patent 3,508,941 described the use of N-methyl pyrrolidinone as a diluent to reduce the viscosity and improve the spinnability of the cellulose solution [28-29].

Sodium hydroxide and urea was found to be a non-derivatizing and nonpolluting solvent mixture [30]. The cellulose solution degrades very slowly. Cellulose solution was formed into a cellulose membrane [30]. Hattori et al. [31] found that ethylene diamine/thiocyanate salt solution is also a solvent for cellulose. They were able to dissolve up to 16% of the cellulose in 54/46 (w/w) ethylene diamine/sodium thiocyanate solution. The solution was found to be stable for 30 days. They observed using <sup>13</sup>C MAS NMR that ethylene diamine/sodium thiocyanate solution converts crystalline cellulose into amorphous structure during dissolution.

Frey et al. [2-3] electrospun 8% solution of cellulose in ethylene diamine/thiocyanate salt solution at an applied voltage of 30 kV. The cellulose solution was frozen-thawed three times before spinning. The diameter of the resultant fibers was found to be greater than 100 nm and the fibers were having collapsed spiral shape.

Cellulose nanofibers fibers were manufactured by a process called dry-wet electrospinning method [32]. Alpha-Cellulose solutions were formed in a 17.5% NaOH water solution and 50% water solution of NMMO. The electrospinning was carried out between 80 and 100°C and the nanofibers were collected on the surface of a grounded coagulation bath containing water.

In this study, electrospinning of cellulose was considered without using a bath, as a water bath could be hazardous at such high voltage of 9 to 10 kV. In order to make cellulose submicron fibers, cellulose has to be dissolved in a suitable solvent and that was a major challenge, and the solution has to be electrospun. The objectives of this study were:

Electrospin cellulose submicron fibers and to study the diameter and morphology of the submicron fibers.

## **MATERIAL AND METHODS**

### **Materials**

Alpha-cellulose was purchased from Sigma-Aldrich, St. Louis, Missouri. Solvents used in the study were sodium hydroxide/urea aqueous solution, concentrated phosphoric acid and N-methylmorpholine-N-oxide. Sodium hydroxide crystals, concentrated phosphoric acid and urea were purchased from Fisher Scientific.

### **Alpha-Cellulose Solution Preparation**

Concentration of alpha-cellulose in 6% (w/w) sodium hydroxide/4% (w/w) urea aqueous solution and concentration of alpha-cellulose in 12% (w/w) sodium hydroxide/8% (w/w) urea aqueous solution was 4% (w/w).

Concentration of alpha-cellulose in 85% phosphoric acid was varied between 3% to 5% (w/w). Cellulose was stirred in hot N-methylmorpholine-N-oxide/N-methyl pyrrolidinone/water mixture at 40°C for a period of fifteen minutes. The concentration of cellulose was altered between 1.25% (w/w) to 4.3% (w/w).

### **Electrospinning Of Alpha-Cellulose**

A gas tight syringe with a 22-gauge needle was used in the electro spinning. The applied voltage to the syringe was between 25kV and 28 kV. The positive lead from a high voltage supply equipment (Spellman High Voltage Electronics Corp.) was connected to the external surface of the needle of the syringe by an alligator clip. The distance between the two electrodes was altered between 2 and 7 inches. A flow rate of 0.1ml/hour was maintained during spinning. The submicron fibers were collected on a fabric. The collector used was a circular stainless steel plate. The submicron fibers were collected on a fabric.

### **Measurement Of The Fiber Diameter With Scanning Electron Microscope**

The diameter of submicron fibers was measured using images from scanning electron microscope. The submicron fibers samples were mounted on the aluminum mounts for SEM analysis. The number average fiber diameter of the submicron fibers were calculated based on measurement of diameter of twenty nanofibers per image.

### **Characterization of the Morphology of the Submicron Fibers**

The morphology of any fiber influences its mechanical, thermal, sorption, density and other properties. Morphology of the submicron fibers was studied with the help of powder X-ray technique on a BRUKER axS D8 X-ray machine. An electron beam was accelerated by a high voltage of 40 kV and a current of 50 mA. In powder X-ray technique, the samples were converted into a powder form and then samples were irradiated with an X-ray beam, having a wavelength of 1.54 Å in vacuum using a copper anode. The scattered intensity was measured between  $2\theta = 10^0$  to  $50^0$ . Crystallinity was determined by  $X_c = I_c / (I_c + I_a)$  where  $I_c$  and  $I_a$  are the total integrated intensities under all crystalline and amorphous peaks, respectively.

## **RESULTS AND DISCUSSION**

### **Dissolution And Spinnability Of Cellulose**

Alpha-cellulose did not dissolve in 6% (w/w) sodium hydroxide/4% urea aqueous solutions and in 12% (w/w) sodium hydroxide/8% (w/w) urea aqueous solution. Sodium hydroxide/urea solutions can dissolve natural cellulose having viscosity average molecular weight less than  $8.5 \times 10^4$  and regenerated cellulose having viscosity average molecular weight less than  $12 \times 10^4$  [30]. Alpha-cellulose has a molecular weight in the range of 324,000 to 2268,000 and the higher molecular weight of alpha-cellulose would be the reason for the non-dissolution [1].

Although, alpha-cellulose is soluble in 85% phosphoric acid, but when the solutions were electrospun at an applied voltage between 15kV to 25 kV and at a spin length of 4 to 6 inches, the collector fabric was found to be wet because of the presence of the solvent. It indicates that the solvent was not getting vaporized completely during the electrospinning and was collecting on the fabric. Concentrations higher than 7.5% (w/w) of cellulose yields liquid crystal solutions [33].

Solutions having concentrations 4.3%, 4%, 3.7%, 3.5%, 3%, 2.5%, 2%, 1.86%, 1.73% and 1.25% (w/w) of cellulose in N-methylmorpholine-N-oxide/N-methyl pyrrolidinone/water solvent mixture were soluble in the solvent mixture. Michael et al. (2000) observed using  $^{13}\text{C}$  MAS NMR that hot N-methylmorpholine-N-oxide converts crystalline ramie into amorphous solid by the initial penetration of solvents between the molecular sheets.

Subsequently, it breaks hydrogen bonds, to loosen individual chains and then dissolve it [34]. But the solution was solidifying and consequently clogging the needles during the electrospinning at 21°C. However, when the spinning temperature was increased from 21°C to 38°C, 1.25% (w/w) and 2.5% (w/w) solutions of cellulose in the N-methylmorpholine-N-oxide/N-methyl-pyrrolidinone/water solvent mixture were electrospinnable. The higher ambient temperature was maintained by heaters. Some of the submicron fibers were found to be beaded. The beading might be formed due to insufficient attenuation of the submicron fibers.

### Diameter Of Cellulose Submicron Fibers

The SEM image of the fibers produced from 1.25%(w/w) cellulose in N-methylmorpholine-N-oxide/N-methyl pyrrolidinone/water solvent mixture *Figure 1*. A web of submicron fibers is seen on all the images. Cellulose submicron fibers from 2.5%(w/w) cellulose solution in the N-methylmorpholine-N-oxide/N-methyl-pyrrolidinone/water solvent at an ambient temperature of 38<sup>0</sup> C were quite coarser as compared to fibers spun from 1.25% (w/w) cellulose solution in the N-methylmorpholine-N-oxide/N-methyl-pyrrolidinone/water solvent mixture at the same temperature *Figure 2*.

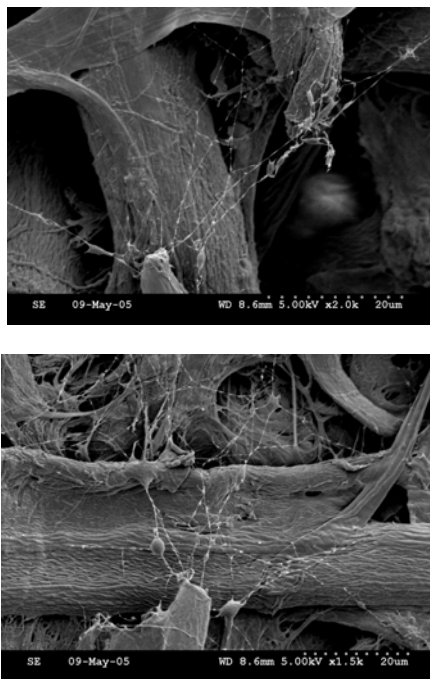


FIGURE 1. SEM images of cellulose submicron fibers 1.25% (w/w) and 2.5% (w/w) solutions of cellulose in the N-methylmorpholine-N-oxide/N-methyl-pyrrolidinone/water solvent mixture.

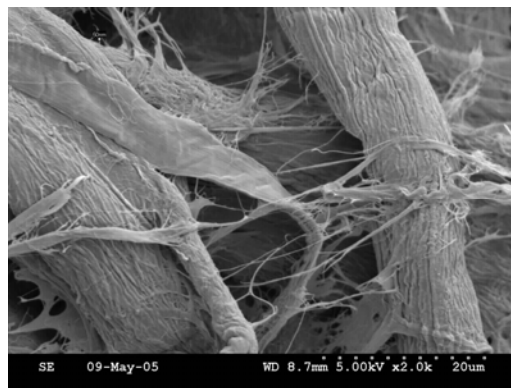


FIGURE 2. SEM image of cellulose submicron fibers from 2.5% (w/w) solutions of cellulose in the N-methylmorpholine-N-oxide/N-methyl pyrrolidinone /water solvent mixture.

The number average diameter and standard deviation of the submicron fibers were calculated based on measurement of diameter of twenty nanofibers per image are given in Table 1. The number average fiber diameter of cellulose submicron fibers from 1.25% (w/w) cellulose solution in the N-methylmorpholine-N-oxide/N-methyl-pyrrolidinone/water solvent mixture is 207 nm and the number average fiber diameter from cellulose submicron fibers from 2.5% (w/w) cellulose solution in the N-methylmorpholine-N-oxide/N-methyl-pyrrolidinone/water solvent mixture is 243 nm. Greater mass flow rate of the cellulose in the higher concentration solution yields coarser fibers, which is in agreement with the basic equation for mass balance Eq. 1.

$$W = A_1 V_1 \rho_1 C_1 = A_2 V_2 \rho_2 C_2 \quad (1)$$

where:

A = polymer jet cross section diameter;

V = linear polymer jet velocity;

$\rho$  = cellulose solution density;

W = mass throughput rate;

<sub>1</sub> = for parameters at the point of extrusion;

<sub>2</sub> = for parameters at the fiber stage in the nonwoven web on the collector.

It has been also found that diameter of electrospun fibers depend upon viscosity, which in turn is related to the solution concentration. So, the diameter of the electrospun fibers depends upon the solution concentration, as per the relation

$$d \propto C^\delta \quad (2)$$

where, d is the fiber diameter and C is the concentration of the polymer solution,  $\delta$  is a constant, which depends upon the polymers [35]. The

fiber diameter increase was observed with the increase in polymer solution concentration [36-37].

The histograms of number average fiber diameter of cellulose submicron fibers from 1.25% (w/w) and 2.5% (w/w) cellulose solution in the N-methylmorpholine-N-oxide/N-methylpyrrolidinone/water solvent mixture are shown in Figure 3.

TABLE I. Influence of concentration of the cellulose solution on the number average fiber diameter.

Solutions concentration	Fiber Diameter	
	Average (nm)	SD
1.25%	207	0.05
2.5%	243	0.06

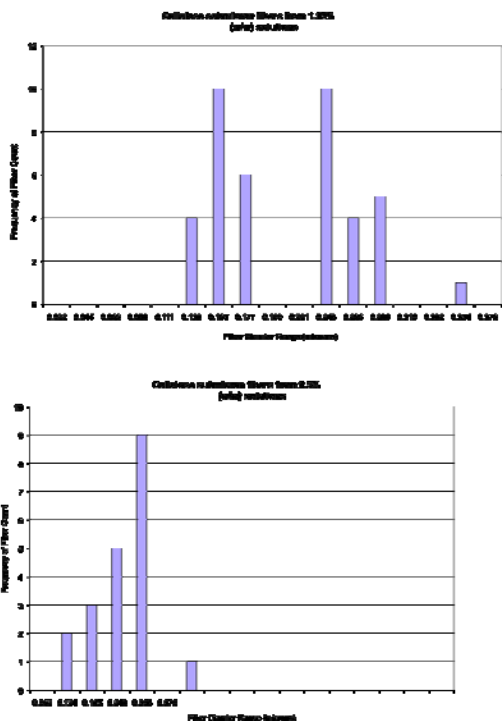


FIGURE 3. Fiber diameter histograms of cellulose submicron fibers from 1.25% and 2.5% (w/w) cellulose solutions.

### Degree Of Crystallinity Of The Cellulose Submicron Fibers

The morphology of any fiber influences its mechanical, thermal, sorption, density and other properties. A web of cellulose submicron fibers was used to study the morphology of the fibers using wide-angle x-ray diffraction technique. The scattered intensity on the x-ray machine was measured between  $2\theta = 10^0$  to  $50^0$ . Degree of crystallinity was

determined by  $X_c = I_c / (I_c + I_a)$  where  $I_c$  and  $I_a$  are the total integrated intensities under all crystalline and amorphous peaks, respectively. Degree of crystallinity of the cellulose submicron fibers was 37.88%. It is known that the cellulose fibers from NMMO/NMP/water solution will crystallize on solidification [38-40]. The degree of crystallinity of electrospun fibers is influenced by various process conditions such as flow rate, applied voltage and spin length, which have an effect on elongational stress. Degree of crystallinity of electrospun cellulose fibers from NMMO/water was observed in the range of 40 to 60% [39].

### CONCLUSIONS

The electrospinning of cellulose was a challenge for researchers for quite some time due to difficulty of the dissolution of the cellulose in the solvents. A lot of difficulties in electrospinning cellulose using sodium hydroxide/urea aqueous solution and concentrated phosphoric acid were encountered. However, N-methylmorpholine -N-oxide/N-methylpyrrolidinone/water mixture proved to be a good solvent. Degree of crystallinity of the cellulose submicron fibers was 37.88%. The number average fiber diameter of cellulose submicron fibers from 1.25% (w/w) cellulose solution in the N-methylmorpholine-N-oxide/N-methylpyrrolidinone/water solvent mixture is 207 nm and the number average fiber diameter from cellulose submicron fibers from 2.5% (w/w) cellulose solution in the N-methylmorpholine-N-oxide/N-methylpyrrolidinone/water solvent mixture is 243 nm.

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