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Short Communication

Preparation Ultra-fine Fibrillated Lyocell Fiber and Its Application in Battery Separator

Wang Xiwen[*], *Hu Jian, Long Jin*

State Key Lab of Pulp and paper engineering, South China University of Technology, 510640,Guangzhou, China
[*]E-mail: wangxw@scut.edu.cn

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Ultra-fine fibers were prepared from Tencel fiber by fibrillation. The fibrillation behaviour of Tencel fiber on different SEL condition with refiner is studied in this paper. The results show that Tencel fiber can be well fibrillated with long fiber length after refining at 0.4-0.8Ws/m SEL. The distribution of microfibers diameter are mostly between 200nm to 2000nm. The fibrillated fibers are applied in alkaline Zn/MnO_2 battery separator. The properties of the resulting separators in comparison to the commercial separator are much better especially in thickness and pore size.

Keywords: ultra-fine fiber; fibrillation; separator

1. INTRODUCTION

Lyocell fibres are 100% cellulosic fibers, solvent spun from purified woodpulp. They have been commercially manufactured and supplied under the trade name Tencel into a wide range of nonwovens applications for more than 15 years.

During this time, Tencel fibers have become well established in the manufacture of speciality papers for filtration applications ranging from beverage filters, through industrial air and liquid filters, to automotive fuel and oil filters and even in cigarette filter papers [1]. But the application in battery separator is little reported. Fine diameter fibers have always been of interest in battery separator applications. It is widely known that, in general, finer fibers are required to achieve good separation effect. Tencel fiber is unusual for its fibrillation character [2-4]. The fibrillated fiber is an ultra-fine fiber [5]. The fibrillation behaviour of Tencel fiber with refiner and application in battery separator is studied in this paper.

2. EXPERIMENTAL

2.1. Materials

Tencel fiber is 1.7dtex×4mm provided by Lenzing, Austria.

2.2. Preparation wet-laid nonwoven separator

Battery separator was made by wet-laid nonwoven method. The fiber composition of separator is shown in Table 1. Separator A is a commercial sample Made in Japan.

Table 1. Fiber composition of separator

Separator	PVA	Viscose fiber	Wood fiber	Tencel fiber
A	√	√	√	——
B	√	√	√	5%
C	√	——	√	10%

2.3. Measurement

Pore dimensions were tested using a model CFP1100A Capillary Flow Porometer (CFP) manufactured by PMI. In a typical CFP experiment, separator is filled with Pore wick and subjected to gas pressure. The pressure on the separator is increased in controlled fashion while the flow rate of gas through the separator is simultaneously monitored. Pore size information can be obtained from these two values. The pore size was calculated based on Eq. (1):

$$D = {4\gamma Cos\theta}/{p} \qquad (1)$$

Where D is the diameter; γis the surface tension; θis the contact angle; p is the pressure difference. Porosity was determined by mathematical calculation by Eq. (2):

$$\text{Porosity (\%)} = {1-(W/\rho)}/{L_1 \cdot L_2 \cdot t} \qquad (2)$$

Where W is the weight of specimen; ρis the density of specimen; L_1 is the length of specimen; L_2 is the length of specimen; t is the thickness of specimen.

Surface morphology of separator was observed by the field emission scanning electron microscope (SEM) LEO1530VP from Germany.

The freeness of fiber was measured following the Tappi method T227 om-94. Fiber length and fines content were measured by Kajanni FS-300.

3. RESULTS AND DISCUSSION

3.1. Fibrillation

Tencel fiber is also characterized with its ordered microstructure. This structure is similar as the PPTA (para-phenylene terephthalamid) fiber [2]. These fibers can be fibrillated. The fibrils will be made by this method.

Tencel fibers are refining with KRK-2500Ⅱ（Japan）refiner at different SEL (special edge load) for its fibrillation. Fibers pass the refiner five times to get homogeneous fibers. The properties of refining fibers are shown in Table2.

Table 2. The properties of refining fibers

Sample	SEL (Ws/m)	CSF Value	Fiber length (mm)	Fines（%）		
				<200nm	200-1000nm	1000-2000nm
1	0.1	600	3.16	4	10	8
2	0.2	510	2.87	6	14	11
3	0.4	450	2.40	8	20	14
4	0.6	350	2.01	9	23	16
5	0.8	300	1.64	11	26	20
6	1.0	280	1.02	13	29	22
7	1.5	240	0.87	15	32	24
8	2.0	100	0.64	16	35	25

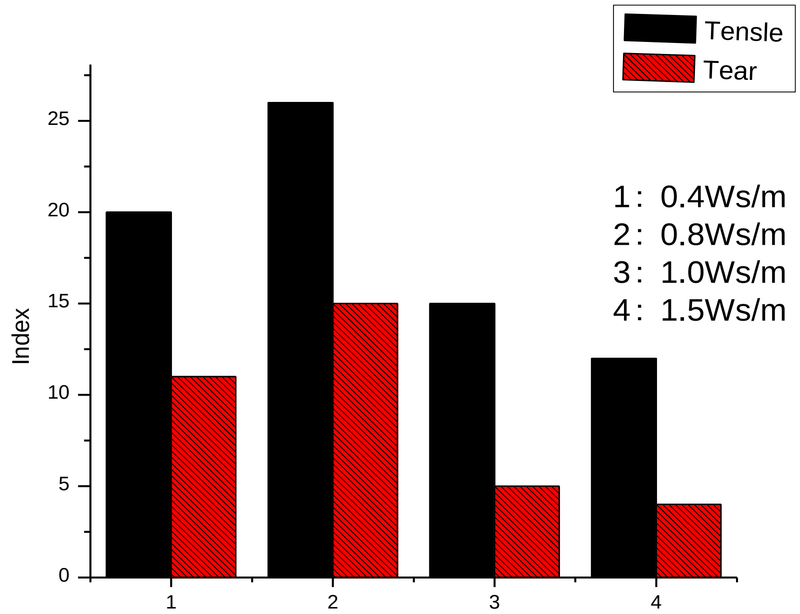
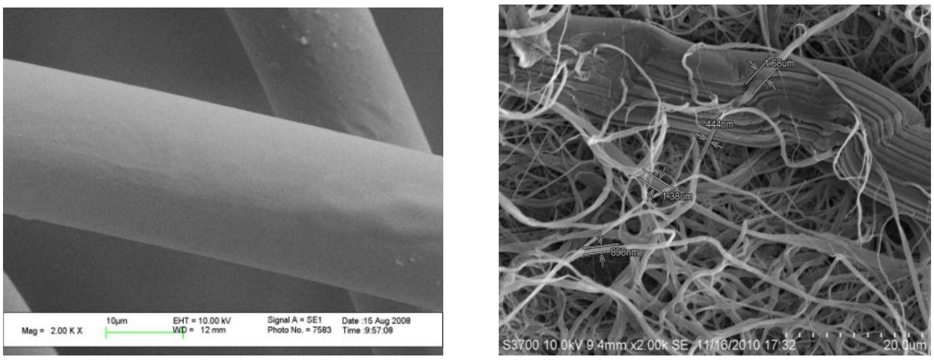


Figure 1. Paper properties of 100% refining Tencel fibers

The results indicated that fiber length and the CSF (Canada standard freeness) decrease with the SEL increased in Table2. But the fines content increased. The CSF of Tencel fiber is 100CSF at the SEL 2.0 Ws/m. Fibrills content of the fibrillated Tencel fiber is mostly between 200nm-1000nm. The 100% refining Tencel fibers paper properties are shown in Fig.1. The results show that the paper properties are much better when the SEL at 0.4-0.8 Ws/m. The fiber length is the key factors of the paper propeties. When fibers are very short the properties of the paper are poor. The fibers with short fiber length and fine diameter will run to the white water in the wet-laid process. So low specific edge loads are favoured to minimise fibre cutting and maximise fibril development. The optimal SEL is to 0.4-0.8 Ws/m.

Tencel fiber has a longer fiber length and enough microfibers at these conditions. The SEM photos of Tencel fiber and fibrillated Tencel fiber were shown in Fig.2. Tencel fiber before refining is very smooth and has no fibrills. When the fibers after refining, the body of the main fiber become soft and has a lot of microfibers on his surface.



A Before fibrillation B Fibrillated fibers

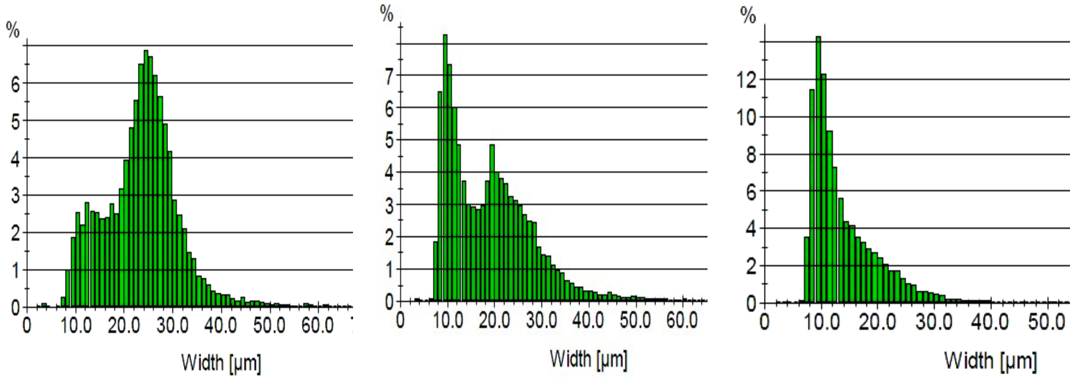
Figure 2. The SEM photo of Tencel fiber before and after fibrillation (at 0.4 Ws/m)

3.2. Application in separator

The fibrillated fibers have a diameters mainly range from 200nm to 2000nm. The fine pore structure can be made when these ultra-fine fibers to be used in papermaking process. The fine pore structure is very important for battery separators. The pore size must be smaller than the particle size of the electrode components, including the electrode active materials and the conducting additives. In practical cases, membranes with 10-20μm average pore sizes have proven adequate to block the penetration of particles since the tortuous structure of the pores assists in blocking the particles from reaching the opposite electrode in Zn/MnO_2 battery. The electrolyte is 35-40% KOH liquid in this battery [3]. So the material of the separator of the battery must has a good alkaline resistence, such as PVA fiber, viscose fiber and special wood fiber. Tencel fiber also has the same character. The refining Tencel fiber at 450CSF is added into the separator. The properties of different separators were shown in Table 3.

Table 3. The properties of separators

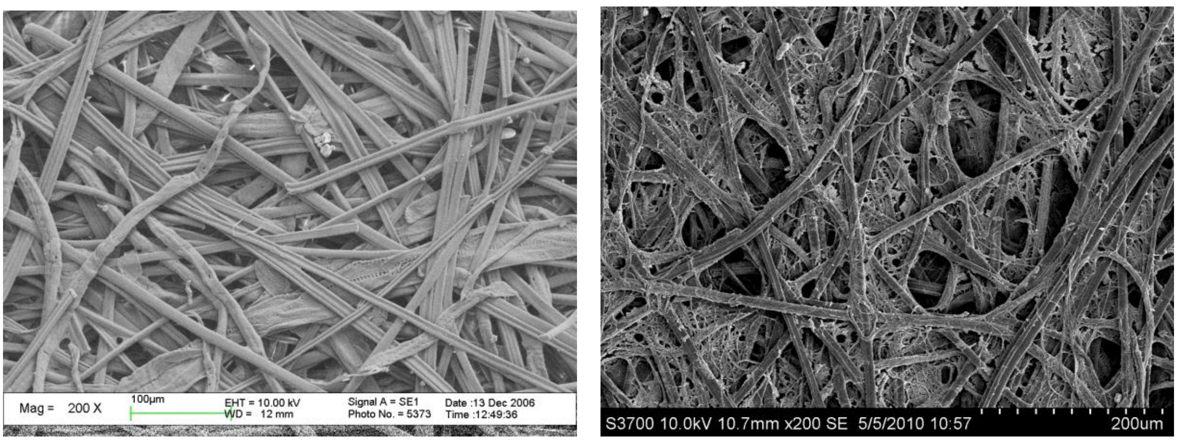
Separator	Weight (g/m^2)	Thickness （μm）	Tensile index (Nm/g)	Porosity (%)	Max pore size （μm）	Average pore size （μm）
A	35.1	90	65.2	37	45	25
B	35.0	85	72.5	36	35	18
C	35.2	80	76.8	35	30	12



A Separator A B Separator B C Separator C

Figure 3. Pore size distribution of separators

In Table3, it can be found that the pore size and the thickness decrease when fibrillated Tencel fiber is added. The high energy density can be got with a thin separator. But the porosities of the separators are almost the same in table 3. When fibrillated Tencel fiber added 10%, the average pore size is 12μm.



A Separator A B Separator C

Figure 4. The SEM pictures of separators

An appropriate porosity is necessary to hold sufficient liquid electrolyte for the ionic conductivity between the electrodes which ensure small internal resistance of batteries [6-8]. Typically, the commercialized battery separators have a porosity of 35%. The samller pore size and higher porosity has the best performance of the separator. The pore size distribution of separators was shown in Fig.3. It can be found that separator A's pore size distribution is from 10 to 50µm. Most of its pore dimensions are between20-30µm. The separator B's pore size distribution has two areas. One is near 10µm, another is between 20-30µm. But the separator C's distribution is mainly between 10-15µm. The tensile index also improved when Tencel fiber added. The fibrillated fibers increased the intertexture of the paper. So the fibrillated Tencel fiber can decrease the pore size and improve the tensile index with high porosity for separator.

Surface morphology of separators was observed by SEM. In Fig. 4, it can be found that the pores of separator C are more uniform than separator A. The separator A has some big pore. But in separator C, the big pore is filled by the microfibers from the fibrillated Tencel fiber in separator C.

4. CONCLUSION

Tencel fiber can be fibrillated to expose the fibrills with the diameter from 200nm to 2000nm. The optimal SEL is colse to 0.4-0.8 Ws/m for fibrillation in refining process. The fibrillated fiber can be used to the Zn/MnO_2 battery separator. The average pore size is 12µm much smaller than the commercial sample and with the porosity 35%. It indicated that the fibrillated Tencel fiber is a good material for alkaline battery separator to control the pore size and thickness and improve the tensile index.

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