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Preparation and characterization of a Lithium-ion battery separator from cellulose nanofibers

Hongfeng Zhang [a], **Xiwen Wang** [a,*], **Yun Liang** [a]

[a] *State Key Laboratory of Pulp and Paper Engineering, School of Light Industry and Food Sciences, South China University of Technology, Guangzhou, 510640, China*

* Corresponding author. Tel.:+86 13824480153.

E-mail address: wangxw@scut.edu.cn (X. Wang).

Abstract

Optimizing the desired properties for stretch monolayer separators used in Lithium-ion batteries has been a challenge. In the present study a cellulose nanofiber/PET nonwoven composite separator is successfully fabricated, using a wet-laid nonwoven (papermaking) process, which can attain optimal properties in wettability, mechanical strength, thermal resistance, and electrochemical performance simultaneously. The PET nonwoven material, which is fabricated from ultrafine PET fibers by a wet-laid process, is a mechanical support layer. The porous structure of the composite separator was created by cellulose nanofibers coating the PET in a papermaking process. Cellulose nanofibers (CNFs), which are an eco-friendly sustainable resource, have been drawing considerable attention due to their astounding properties, such as: incredible specific surface area, thermal and chemical stability, high mechanical strength and hydrophilicity. The results show that the CNF separator exhibits higher porosity (70%) than a PP (polypropylene) separator (40%). The CNF separator can also be wetted by electrolyte in a few seconds while a PP separator cannot be entirely wetted after 1 min. The CNF separator has an electrolyte uptake of 250%, while a PP separator has only 65%. Another notable finding is that the CNF separator has almost no shrinkage when exposed to 180 °C for 1 h, whereas a PP separator shrinks by more than 50%. Differential Scanning

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Calorimetry (DSC) shows that the CNF separator has a higher melting point than a PP separator. These findings all indicate that the CNF 29 separator will be more favorable than stretch film for use in Lithium-ion batteries.

Keywords: Materials processes, Natural product modification, Materials science, Nanomaterials

1. Introduction

In recent years, increasing concerns for the preservation of environment and sustainability of resources have been drawn. The utilization of natural sustainable fibers is receiving great interest as an alternative to non-renewable resources in material technology [1] [2] [3]. Each year approximately 100 billion metric tons of cellulose are produced [4], it makes this biopolymer as a resource that has been exploited for many decades and will still be important for the next ones.

Cellulose is a high-molecular weight linear homopolymer constituted of repeating β-D-glucopyranosyl units joined by (1-4) glycosidic linkages in a variety of arrangement [5] [6]. Cellulose nanofiber's astounding properties are: extremely large and active surface area [7], biodegradability, natural and renewable materials [8], coefficient of thermal expansion (CTE) of 0.1 ppm/K, high strength of 2–3 GPa and Young's modulus of 130–150 GPa, and good chemical durability [9] [10]. All these properties predict that cellulose nanofibers could be used in many applications in a diverse range of fields, including reinforcement agent in polymeric matrix [11] [12], thickening agent [13], optical transparent membrane [14] [15].

However, there are few reports about cellulose nanofiber applied in energy field. Lithium-ion battery is an ideal electrochemical energy system, which is characterized by high specific energy, high efficiency and long cycle life. Separator is considered to be a core component of the Lithium-ion batteries, its primary role is to maintain electrical isolation between the cathode and anode. Serving as electrolyte reservoir to ensure ionic transport is another important function.

At present, the commercial separator for Lithium-ion batteries are primarily stretch membranes, which are made of PE (polyethylene), PP (polypropylene) [16] [17] and their combinations. However, the fatal disadvantage of this current commercial separator is their relatively low melting temperature, 130 °C for PE and 160 °C for PP, respectively, which could cause direct contact of electrodes [18] and even lead to explosion of batteries when overheating or short circuit occurs. Additionally, hydrophobic character and low porosity of stretch film could affect electrolyte uptake and severely impede the ionic transportation.

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In order to overcome these drawbacks of commercial separator, among various approaches have been tried, nonwoven substrates have attracted extensive attention owing to their high porosity, excellent thermal properties, good chemical resistance and cost competitiveness [19] [20] [21] [22] [23] [24] [25]. However, the large pore size and excessive pore size distribution could lead to self-discharge, uneven current distribution and internal short circuit.In an attempt to meet the demand of Lithium-ion battery, studies on controlling the pore size and pore size distribution of nonwoven substrate have been reported by other research groups. Lee et al. [26] explored a new silica (SiO_2) nanoparticle/polyvinylidene fluoride-hexafluoropropylene coated polyethylene terephthalate (PET) nonwoven composite separator for a Lithium-ion battery as a promising alternative to a commercialized polyethylene (PE) separator. Thin Al_2O_3 over-layers are coated on both sides of a PI nonwoven separator produced by electrospinning via a dip-coating process by Il-Doo Kim [27]. In this study, as a new strategy to achieve this objective, cellulose nanofibers are employed to control pore size and its distribution of PET nonwoven produced by wet-laid process. Unlike SiO_2/Al_2O_3 nanoparticles, cellulose is an abundant and renewable biomass in the world and cellulose nanofiber can be successful applied as a separator in Lithium-ion battery [28], furthermore, cellulose nanofibers used in this study, different from cellulose nanofibers used by Lee, are fabricated by eco-friendly mechanical preparation without chemicals.

In this paper, the low-cost papermaking method is employed to prepare the wet-laid multi-layer separator comprising PET nonwoven and cellulose nanofibers.

2. Materials and methods

2.1. Materials

- PET fiber 0.03 dtex/3 mm, Teijin Japan.
- Tencel fiber 1.7 dtex/4 mm, Lenzing, Austria.
- Dispersing agent PEO (polyethylene oxide) molecular weight: 5 million, Guangzhou.
- Antifoaming agents (Organic silicon class) surface tension: 22.35 mN/m, Erosion point 39 °C, Guangzhou.

2.2. Preparation of PET nonwoven

Ultrafine PET fibers were dispersed by deflaker for 5000r, wet-nonwoven paper was prepared on a handsheet former (messmer 255 America) (TAPPI method T205 sp-02), in order to improve mechanical strength, the PET

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nonwoven paper was objected to hot pressing at 120 °C by calendar on a given pressure. Meanwhile, hot pressing can control the thickness of PET nonwoven paper [29] [30].

2.3. Preparation of Tencel cellulose nanofibers

Tencel fibers are regenerated from cellulose produced by Lenzing AG, Austria, through spinning process with N-methylmorpholine-N-oxide monohydrate as the solvent [31]. Tencel fiber has a unique nanofibril structure [32] and can be used as microfiber and nanofiber after beating. In the preparation process of Tencel cellulose nanofibers, different from acid hydrolysis and TEMPO-mediation, no chemicals were added.

The Tencel fiber was refined with High Consistency Disc Refiner at 5% pulp consistency, gradually we narrowed the gap of two discs and the gap in turn is 1.5, 1, 0.7, 0.5, 0.3, 0.1 mm. The final beating degree of refined Tencel pulp was about 86 °SR, then pulp was screened by five different net meshes in order to obtain nano-sized fibers. $1^{\#}$ pulp is the original pulp without screening, and different pulp numbers means that the pulp was filtrated through different net mesh numbers: $2^{\#}$-30, $3^{\#}$-50, $4^{\#}$-100, $5^{\#}$-120 and $6^{\#}$-200. $1^{\#}$ pulp separately mixing with $2^{\#}$, $3^{\#}$, $4^{\#}$, $5^{\#}$ and $6^{\#}$ pulp at the percentage of 3/2 were used to fabricate wet-laid multi-layer separator with both mean pore size and maximum pore size below 1 μm.

2.4. Separator preparation

Firstly, the PET nonwoven paper was spread out on the wire without wrinkle, then cellulose nanofiber solution after homogeneous dispersing was filtered onto the PET nonwoven paper in papermaking process, after drying, the PET layer and cellulose nanofiber layer could combine together tightly. The process preparation of wet-laid multi-layer separator is shown in Fig. 1.

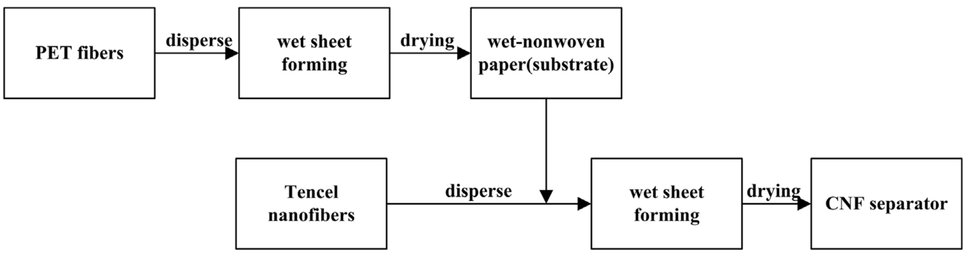

Fig. 1. Process of wet-laid multi-layer separator preparation.

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2.5. Properties test

2.5.1. Morphology observation

SEM (Scanning Electron Microscope) and FESEM (Field Emission Scanning Electron Microscope) were used to observe cellulose nanofiber and porous structure and surface morphology of the separator.

2.5.2. Physical properties

According to the GB451-79 standard, basic weight was tested by the balance with sensitive quality: 0.1 mg.

Thickness was examined by electrical thickness tester.

Mean pore size, maximum pore size and pore size distribution was tested by Capillary Flow Porometer from America.

According to the GB453-89 standard, tensile strength and breaking elongation were tested by LW tensile tester.

The electrolyte wettability was examined by observing the wetting area of the separator surface after dropping the liquid electrolyte onto separators.

The electrolyte uptake was determined by Eq. (1) with the weights of separators before and after soaking them in a liquid electrolyte for 10 min.

$$\text{Electrolyte uptaken (\%)} = (W_a - W_b)/W_a \times 100\% \quad (1)$$

where W_a and W_b are the weights of separator before and after soaking in the electrolyte.

The porosity was tested by Eq. (2)

$$\text{Porosity (\%)} = (W_a - W_b)/(\rho_e \times V_s) \times 100\% \quad (2)$$

where W_a and W_b are the weights of separator before and after soaking in the electrolyte, ρ_e is the density of electrolyte and V_s is the volume of separator.

The contact angles were tested by OCA40 Micro produced by data physics Germany.

The mechanical strength was measured by INSTRON 5565.

2.5.3. Thermal properties

Thermal shrinkage of separators was evaluated by calculating the dimensional change after exposure at, respectively, 160 °C, 170 °C, 180 °C, 200 °C for 1 h.

The melting point of separator was examined by DSC.

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2.5.4. Electrochemical performance

Coin cells (2032 type coin cell) were assembled in an argon filled glove box and a series of electrochemical performance were tested by MITS Pro Arbin Instruments.

3. Results and discussion

Table 1 illustrates the results how different pulp proportions affect pore sizes of CNF separator. It is noticeable that the separator from 1# and 6# pulp with the percentage of 3/2 achieved the goal of both mean pore size and maximum pore size below 1 μm. With higher screening degrees, we got higher proportion of nano-scaled fibers, so it is more likely to obtain nano-sized separator.

Before scrutinizing the characteristics of separator, the morphology of cellulose nanofibers were first observed. The morphology of Tencel nanofibers was shown in Fig. 2(a), which was produced by mechanical method, where the diameter of cellulose nanofiber is observed to be several hundred nanometers. During the intensively mechanical process, the severe sheer and fractional force is to be proved an effective method of fabricating nanofibrillation of cellulose fibers. Furthermore, Tencel fibers present many excellent properties as a raw material for Lithium membrane. Firstly, Tencel fibers possess high dry and wet mechanical strength, so it can be used to produce cellulose nanofibers through high consistency refining, other kinds of cellulose fibers would lose mechanical strength during intense beating action, and Tencel fibers is man-made from nature wood pulp, whole process is environmental friendly, because the recovery rate of dissolving agent is more than 99%.

The morphological uniqueness of the CNF separators, also compared to a commercialized PP separator and a pristine PET nonwoven, was elucidated. The pristine PET nonwoven exhibits excessively large-sized pores (approximately larger than 20 μm) that are irregularly formed between the PET fibers Fig. 2(b). From FESEM photographs Fig. 2(a), we can see that the PP separator have amounts of pores like slit (pore size < approximately 0.1 μm), where the pores are formed by uniaxially tensiling. By comparison, an intriguing finding presented by Fig. 2(b), three-dimensional network was constructed by cellulose

Table 1. Pore sizes of CNF separators at different pulp proportions.

	1#+ 2#	1#+ 3#	1#+ 4#	1#+ 5#	1#+ 6#
Mean pore size/μm	3.20 ± 0.41	2.4 ± 0.32	1.08 ± 0.27	0.6 ± 0.17	0.30 ± 0.1
Maximum pore size/μm	7.63 ± 1.86	5.42 ± 1.32	3.67 ± 0.89	1.73 ± 0.36	0.83 ± 0.15

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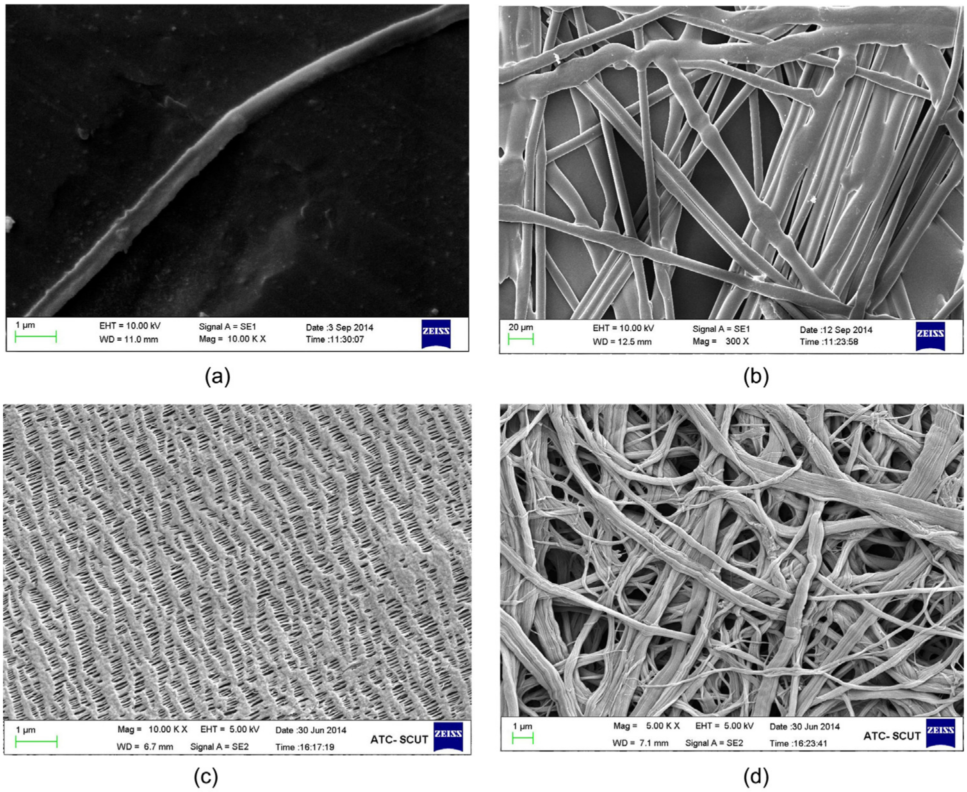

Fig. 2. FESEM/SEM photographs of: (a) Single Tencel nanofiber; (b) PET nonwoven; (c) PP separator; (d) CNF separator.

nanofibers, easily we can find that nano-sized pores are constructed by the compactly combined cellulose nanofibers, which was expected to play a vital role to prevent short circuit. Another notable finding is that the CNF separator has higher pore tortuosity, which can prevent the direct contact of electrode materials and is beneficial to avoid Lithium dentrite to puncture the separator. Table 2 also lists mechanical property of PP and CNF separator, although PP separator has better tensile strength than CNF separator, PET nonwoven acted as support offer adequate mechanical strength for CNF separator to be assembled in coin cells.

Yi Wang et al. [33] fabricated a potential Lithium-ion battery separator (PPTA/PET separator) by wet-laid process. Para-phenylene terephthalamide (PPTA) fibers and PET fibers were mixed in an aqueous suspension and randomly laid down on a screen belt. The wet-laid non-woven materials were calendered at 120 °C with different pressure for the bonding. Different from her way of preparation, we prepared PET nonwoven paper by wet-laid process as support layer for separator, not mixing with fibrillated fibers. As we all know, hot

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Table 2. Physical properties of the PET nonwoven, PP, PPTA/PET and CNF separators.

	Thickness /μm	Tensile strength/MPa	Modulus /MPa	Porosity /%	Electrolyte uptake /%	Mean pore size/μm	Maximum pore size/μm
PP separator	30	134.5 ± 3.5	298 ± 10.6	42	65	0.065	0.1
CNF separator	30 ± 3	57 ± 5	929.5 ± 3.5	70 ± 3	380 ± 5	0.30 ± 0.1	0.83 ± 0.15
PET nonwoven	18 ± 3	–	–	59 ± 4	80 ± 7	64.7 ± 8	117.3 ± 15
PPTA/PET separator	30	–	–	36	370	0.18	1.23

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pressing certainly will decrease porosity. Finally, although PPTA fiber has many outstanding properties, it is very expensive. Physical properties of PPTA/PET separator are shown in Table 2.

The electrolyte wettability was examined by dropping liquid electrolyte onto the surface of separators and observing the spread area in 1 min. Fig. 4 exhibits that in comparison with PP separator, electrolyte was absorbed by the CNF separator immediately, moreover, the electrolyte easily spread over a wide area of the separator. However, due to hydrophobic property of polyolefin [34] and low porosity (40%) (Table 2), it is difficult for PP separator to absorb electrolyte. In order to further depict electrolyte wettability quantitatively, electrolyte immersion-height was measured in Fig. 4(b), after 1 min, the CNF separator present much higher immersion height than PP separator. For CNF separator, cellulose nanofibers and PET nonwoven paper could fast electrolyte absorption [35]. This excellent electrolyte wettability of CNF separators can also be ascribed to high porosity (70%) and its unique nanoporous network channels, which contribute to capillary intrusion [36] [37].

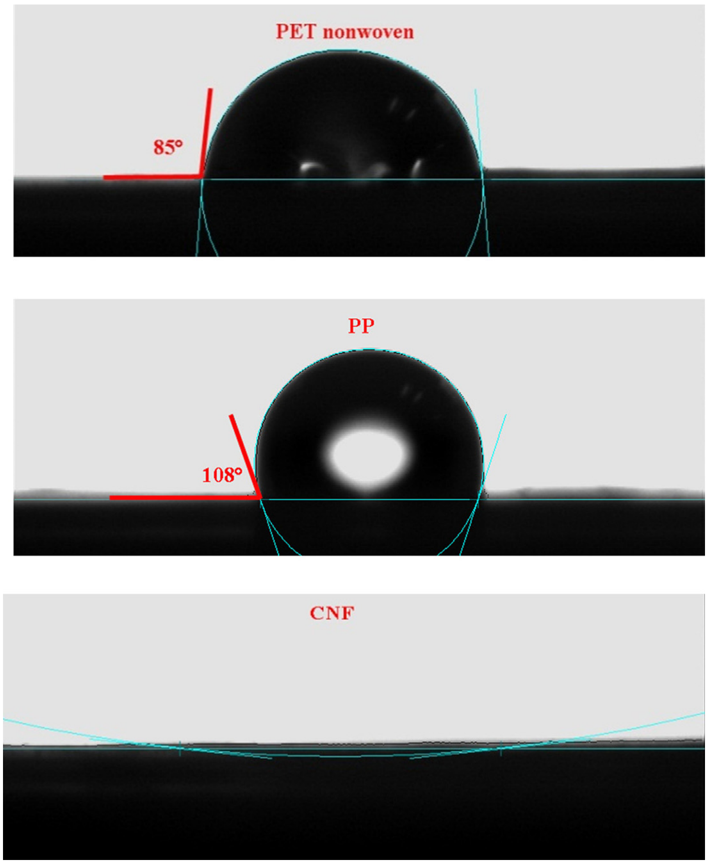

Fig. 3. Photographs of contact angles of PET nonwoven, PP and CNF separators.

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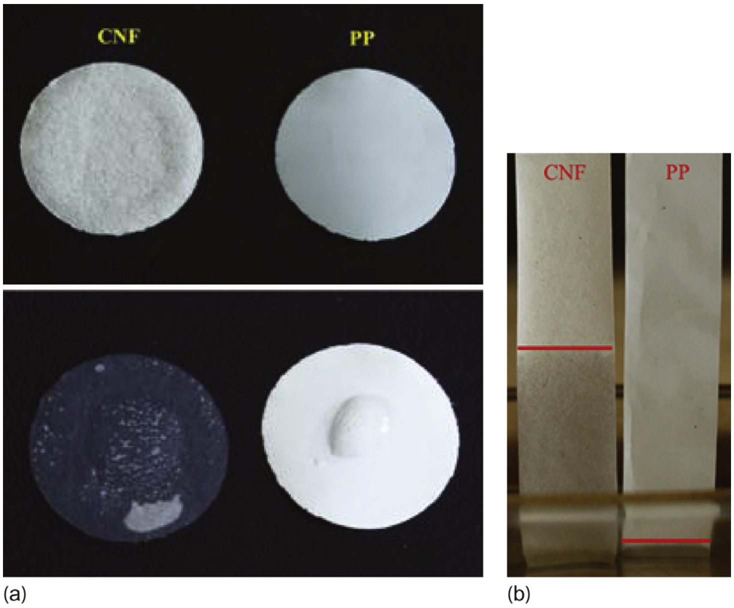

Fig. 4. (a): Photographs of electrolyte (EC/DEC) wettability of PP separator, CNF separator. (b): Photograph of electrolyte immersion height of PP separator, CNF separator.

Contact angles have been measured in order to evaluate wetting properties of separators. The angle of PP separator is 108° owing to its hydrophobic nature. But for CNF separator and PET nonwoven, both of them are exhibit good wetting ability. We can see from Fig. 3, the angle of PET nonwoven is 85°, CNF separator has totally wetted by electrolyte. So with excellent electrolyte wettability, the CNF separator is expected to fulfill better rate capability and cycle performance.

The thermal shrinkage of PP separator and CNF separator were compared based on area dimensional change with increasing temperature from 160 °C

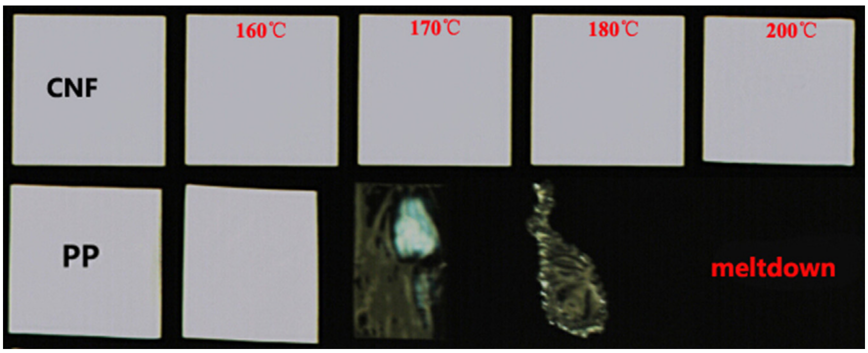

Fig. 5. Thermal shrinkage comparison of the PP separator and CNF separator.

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to 200 °C for 1 h in Fig. 5. The CNF separator exhibits much better thermal stability than PP separator. When temperature is above 150 °C, PP separator occurred shrinkage and deformation [38] due to its low melting point (160 °C), this could induce physical contact between cathode and anode and lead to internal short circuit [39]. On the other hand, CNF separator does not show significant shrinkage up to 180 °C, the aforementioned properties of cellulose nanofiber's excellent thermal resistance and PET nonwoven substrate's high melting point (above 250 °C) play a significant part in keep thermal stability.

In order to further confirm thermal characteristic of CNF separator, DSC was employed to examine melting point of CNF separator and PP separator, as shown in Fig. 6, CNF separator showed the endothermic peaks around 257 °C, which is the melting point of PET. However, PP separator showed endothermic peaks around 150 °C. On the contrary, CNF separator will never melt even at 250 °C. Therefore, it would be a fabulous material for separator to ensure the safety of batteries.

The electrochemical performance of cells with the commercial PP separator and CNF separator were shown in Fig. 7, and the cells were charged under a voltage range of 2.5–4.2 V at a constant charge current density of 0.2 C and discharged at various current densities ranging from 0.1 to 1.0 C.

The comparison of cycling performance (the current density of 0.2 °C as an example) between CNF separator and PP separator is presented in Fig. 7, it is observed that the cells with CNF separator exhibited better capacity than PP separator until the 100th cycle. Furthermore, the discharge capacity retention after the 100th cycle is found to be 91.7% for the CNF separator and 87.8%

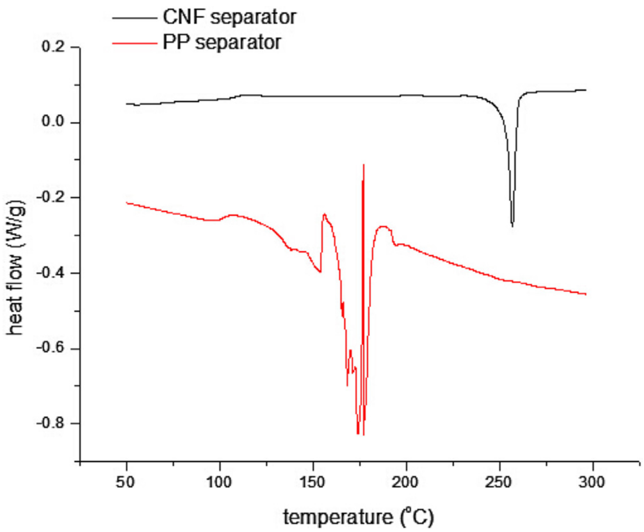

Fig. 6. DSC diagram of CNF separator and PP separator.

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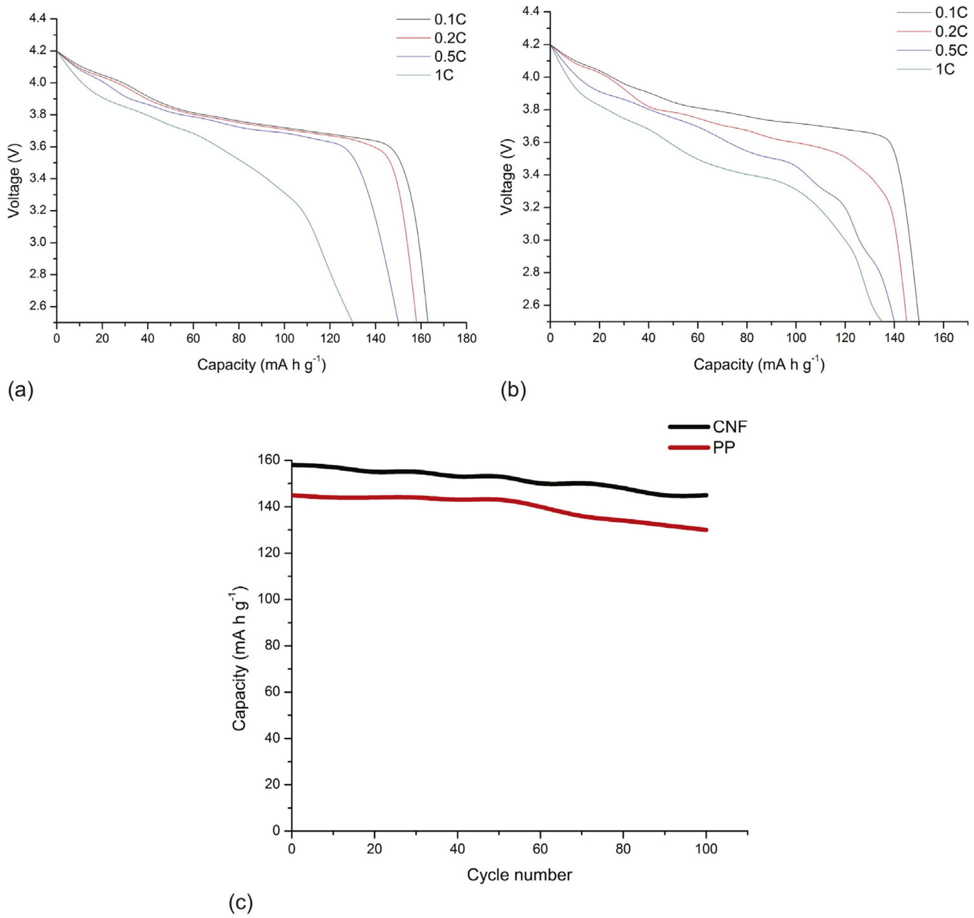

Fig. 7. Discharge profiles of cells with (a) CNF separator, (b) PP separator. (c) Comparison of cycling performance between CNF separator and PP separator.

for the PP separator, respectively. This better cycle performance can be attributed to nanostructure constructed by cellulose nanofibers and good electrolyte, all these factors offered Li^+ an flexible channels.

4. Conclusion

In this study, the preparation method and properties of separator from cellulose nanofibers for Lithium-ion battery were discussed, CNF separator comprised of PET layer and cellulose nanofibers layer exhibited a distinctive structure. The porosity of CNF separator is 70%, which is higher than that of PP separator (40%). CNF separator also has excellent hydrophilic property: it could be wetted in a few seconds and has higher immersion height in the same time period when compared to PP separator. The CNF separator keeps thermal dimensional stability even at 180 °C. But the PP separator has greatly shrunk at this temperature, intriguing capacity retention (91.7%) was observed in the test. Moreover, the papermaking process is low cost, so we believe that, based on

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these excellent physical properties and good electrochemical performance, the separator from cellulose nanofibers will be the promising alternative for the next-generation Lithium battery.

Declarations

Author contribution statement

Xiwen Wang: Conceived and designed the experiments; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Hongfeng Zhang: Performed the experiments; Analyzed and interpreted the data.

Yun Liang: Performed the experiments.

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Competing interest statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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