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Enhancement on the thermostability and wettability of lithium-ion batteries separator via surface chemical modification

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ABSTRACT

The separator is one of the most important components of the battery, which is related to safety performance of the battery. The traditional polyolefin separators have disadvantages such as the poor thermal stability and weak surface hydrophilicity. Herein, we introduced a SiO_2/PAM-grafted polypropylene (PP) separator by surface chemical modification. Compared with unmodified separator, the results showed that the modified separators had better thermal stability and superior wettability. The shrinkage of the bare PP and SiO_2/PAM-grafted PP separators were 70% and 12% at 150 °C for 30 min, respectively. In addition, the surface contact angle of PP separator is reduced from 105° to 37° after grafting modification. Therefore, modified separator not only improves the thermal stability and the wettability but also thickness of separator increase slightly. The dis/charge capacity of different separators were investigated. It was showed that the cell with SiO_2/PAM-grafted PP separator had better electrochemical performance than that with bare PP.

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1. Introduction

Due to their long cycle lifetime, high energy density and environmental friendliness, lithium-ion batteries (LIBs) have achieved great applications in many fields such as portable electronic devices, electric vehicle and renewable power stations during the past decades [1,2]. However, the safety of LIBs has raised much concerns, once the battery has encountered extreme harsh conditions including overcharging, external nobbing, elevated temperature thermal impacting, etc. The internal short circuit has emerged excessive heat within a short time, which causes the separator shrinkage, leading to thermal feedback, fire and even explosion [3]. In LIBs, the micro-porous separator plays a role in isolating the positive and negative electrodes of the battery and allowing Li-ion to pass through from micro-pores [4,5]. To some extent, the separator plays the most important role in the battery, especially for battery safety. Due to their non-polar and inferior surface energy, polyolefin separators have week wettability towards electrolytes [6,7]. Moreover, the polyolefin separator shrink significantly at elevated temperature, leading to direct contact between the electrodes thus internal short circuit. When the temperature of LIBs reaches the melting point of the micro-porous separator (135°Cfor PE, 165°Cfor PP) [8], the separator begins to melt. Therefore, we need to improve the thermal stability of the separator to ensure the safety of LIBs.

In this study, we report a novel SiO_2/PAM-grafted PP separator prepared by surface chemical modification which the polymer monomer and inorganic particles were grafted on pristine polypropylene (PP) separators. Compared with bare PP, SiO_2/PAM-grafted PP separators showed the better thermally stability and the higher electrolyte uptake. In addition, we also assembled into the battery to measured the electrochemical performance of the modified separator.

2. Experimental

2.1. Materials and chemicals

A commercial polypropylene (PP) separator (SKLiBS, SK Energy, 25 μm thickness) was washed with acetone and dried at room temperature for 12 h under vacuum before used. Tetraethylorthosilicate (TEOS, Sinopharm Chemical Reagent Co., Ltd.), acrylamide (AM, Sinopharm Chemical Reagent Co., Ltd.), ammonia, 2′-azobis(2-methylpropionitrile)(AIBN, Sinopharm Chemical Reagent Co., Ltd.), potassium persulfate and other reagents were of reagent-grade without further purification. The separators were assembled into coin cell (CR2330) in argon-filled glove box. The dis/charge cycle performance of cells with different separators were measured using a Battery Testing System (IM6ex) (ZAHNER, Germany).

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2.2. Preparation of PAM-grafted PP

The PP separators were firstly immersed into the solution of potassium persulfate (10wt%) for 1 h at 80 °C and protected by nitrogen. Then, the separators were taken out and washed with deionized water for three times. The processed separators were immersed into 8% concentration of acrylamide (AM) and stirred for 10 min. 0.1% 2′-azobis(2-methylpropionitrile, AIBN) was added slowly into the solution of acrylamide (AM) under nitrogen atmosphere. Subsequently, the system was continuous stirred for 3 h at 60 °C. The separators were washed by deionized water in an ultrasonic bath for three times. Finally, the products (PAM-grafted PP) were dried under vacuum over night.

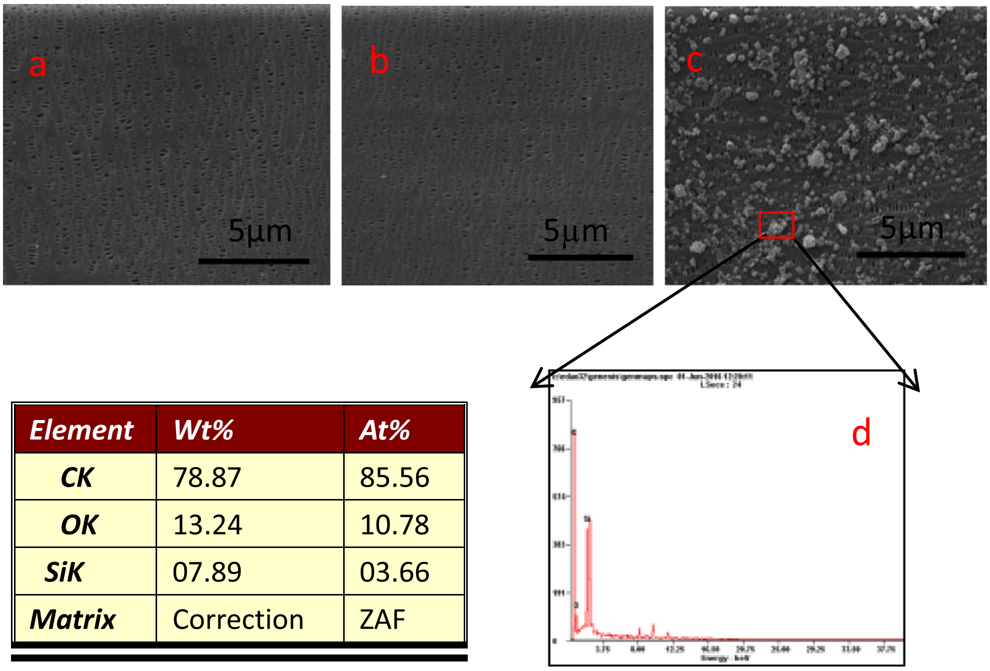
2.3. Preparation of SiO_2/PAM-grafted PP separator

The PAM-grafted PP separators were firstly immersed in the solution of 0.5 mol/L tetraethylorthosilicate (TEOS) stirred for 10 min. 50 ml Ethanol and 60 ml deionized water were added into the above solution and stirred for another 10 min. Afterwards, 0.1 mol ammonia and 40 ml ethanol were added into the above solution and stirred for 12 h [9]. The obtained SiO_2/PAM-grafted PP was washed by deionized water in an ultrasonic bath for 3 times to remove the residual SiO_2 particles. Then, the products (SiO_2/PAM-grafted PP) was dried under vacuum over night.

3. Results and discussion

The morphology of the bare PP and the modified PP separators were shown in Fig. 1. The bare PP separator indicates a uniform network structure with many sub-micron pores (Fig. 1a). After the AM monomer grafted, the surface morphology was significantly unchanged but the porous diameters were more uniform, wherein, the large-sized pore structure of the PAM-grafted PP separators was alleviated obviously (Fig. 1b). It was noteworthy that many particles are distributed uniformly on the surface of the PAM-grafted PP separators, which verified that the SiO_2 particles had been adhere on the surface of the separators via noncovalent bond (Fig. 1c). Fig. 1d depicted an energy dispersive X-ray (EDX) of the SiO_2/PAM-grafted PP separator, indicating that SiO_2 particles exists on the surface of the PP separator. Compared with the bare PP separators, the some properties of the PAM-grafted PP separators and SiO_2/PAM-grafted PP separators were summarized in Table 1. The porosity of the SiO_2/PAM-grafted PP separator had slightly decrease compared with bare PP separator. Therefore, it was likely that AM monomer mainly grafts in the internal pores of the separators instead of the external surface [10]. The increase of ionic conductivity showed that the grafted polymer chain had a good remain on the electrolyte, indicating the better wettability.

Fourier transform infrared spectroscopy (FTIR, WQF-510A) analysis could identify whether AM and SiO_2 were successful grafted onto the separators, as shown in Fig. 2(a). For all of the



Element	Wt%	At%
CK	78.87	85.56
OK	13.24	10.78
SiK	07.89	03.66
Matrix	Correction	ZAF

Fig. 1. SEM-EDX images of (a) bare PP separator (b) PAM-grafted PP separator (c) SiO_2/PAM-grafted PP separator (d) EDX analysis of SiO_2/PAM-grafted PP separator.

Table 1
Some properties of the bare PP and grafted PP separators.

Sample	Thickness (μm)	Porosity (%)	Electrolyte uptake (%)	Thermal shrinkage (%) (under 150 °C)	Ionic conductivity (mS cm^{-1})
Bare PP	25	50	267	70	1.0[13]
PAM-grafted pp	26	47	450	35	1.57
SiO_2/PAM-grafted PP	28	45	436	12	1.43

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