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High-performance and safety of lithium-ion batteries enabled by NiO@PP separator

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Abstract: Separator is a critical internal component, which can isolate the positive and negative electrodes, prevent short circuits, and allow lithium ions to pass freely, ensure safety and performance stability of battery. With the continuous improvement in the energy density of lithium-ion batteries, traditional polypropylene (PP) composite separators can no longer meet the growing demand for energy storage. To address this problem, this study proposes a simple method of modifying the conventional PP separators using an NiO coating to enhance the overall performance of PP separators. The results show that the separator with a 15 nm nickel oxide coating (NiO@PP-15) exhibits the best comprehensive performance. In 1C and 5C cycling tests, NiO@PP-15 demonstrates optimal stability, the slowest capacity decay, and more stable electrochemical performance. After a 5C cycle test, the NiO@PP-15 still has a battery capacity of 121.53 mAh g⁻¹. Compared to the specific capacity of the PP separator (35.56 mAh g⁻¹), both the NiO@PP-5 (61.16 mAh g⁻¹) and NiO@PP-10 (110.33 mAh g⁻¹) also exhibited markedly superior electrochemical performance. These findings indicate that NiO coating not only improves the high discharge capacity but also enables cycling stability under high discharge rate conditions, which provided reliable technical support for high-energy-density and high-rate energy storage systems.

Key words: Lithium-ion batteries; PP separator; NiO coating; NiO@PP separator

1. Introduction

Lithium-ion batteries (LIBs) have emerged as the most promising energy storage device owing to their high energy density, long cycle life and lack of memory effect. They have been extensively deployed in portable electronics and electric vehicles [1, 2]. Driven by the global climate crisis and the dual carbon strategic goals, the rapid development of new energy vehicles and smart grids has put forward more stringent requirements for the energy density, safety and fast charging performance of batteries, promoting the continuous innovation of energy storage technologies [3]. However, as the energy density of batteries keeps increasing, their safety issues have become increasingly prominent-fire accidents caused by battery thermal runaway have cast a shadow over the wide application of lithium-ion batteries.

As one of the key components of lithium-ion batteries, the separator, although not directly involved in the electrochemical reaction, plays an irreplaceable role in ensuring the safe operation of the battery [4, 5]. It needs to provide electrical insulation between the positive and negative electrodes to prevent short circuits, and at the same time, it must offer a smooth channel for lithium-ion transport. Currently, commercialized polyolefin separators (such as polyethylene and polypropylene) are widely used due to their excellent mechanical properties and electrochemical stability. However, their inherent drawback, such as low melting points (about 135°C for PE and about 165°C for PP), poor thermal stability and insufficient wettability with the electrolyte, make them prone to thermal shrinkage under high-temperature or high-rate conditions, leading to contact and short circuits between the positive and negative electrodes and triggering thermal runaway. Additionally, the non-polar surface of polyolefins limits the absorption and retention of the electrolyte, hindering the migration of lithium ions and causing uneven current distribution, which in turn promotes the growth of lithium dendrites, piercing the separator and causing battery failure.

In response to these challenges, functionalization modification of the separator membrane has become the key path to break through the performance bottleneck. Among them, the modification of metal oxide coatings has attracted much attention due to its simple process and significant effect. Nickel oxide (NiO), as a typical transition metal oxide, has the advantages of high thermal stability, rich polar surfaces, and low preparation cost. It shows good application prospects in the field of lithium-ion batteries. Studies have shown that coating nickel oxide on the surface of PP separator can, on one hand, construct a thermal stable layer by leveraging its high melting point to inhibit the thermal contraction of

the separator at high temperatures. On the other hand, the polar surface of NiO can significantly enhance the affinity between the separator and the electrolyte, promoting the infiltration of the electrolyte and the uniform transmission of lithium ions.

Hence, in this study, a NiO@PP separator was fabricated by coating NiO particles to enhance the electrochemical properties. By coating a conventional PP separator with NiO particles, we designed an advanced separator that improves electrolyte wettability and increases capacity by engaging in the electrochemical reactions throughout the charge-discharge cycle. This work provides a viable strategy to enhance the resource utilization of nickel monoxide ore concentrates and proposes new ideas for the development of lithium-ion battery separator materials.

2. Experimental Section

2.1 Preparation of NiO@PP separator

NiO microparticles ($D_{50}=10\ \mu\text{m}$, Aladdin Biochemical Technology) are blended with Super P (5 wt %) and polyvinylidene fluoride binder (5 wt %) in N-methyl-2-pyrrolidone. After 30 min of milling, the slurry was knife-coated onto one face of a 25 μm -thick Celgard 2400 polypropylene membrane and vacuum-dried at 60°C for 12 h. The resulting NiO-functionalized separators, carrying 5, 10 or 15 μm oxide layers, are labeled PP, NiO@PP-5, NiO@PP-10 and NiO@PP-15, respectively.

2.2 Characterization and measurements of NiO@PP separator

Microstructural observation of the NiO@PP separator was carried out with a scanning electron microscope (SEM, Rigaku SmartLab SE, Japan) under a 10 kV beam voltage. The tensile properties of the separator were evaluated on a CMT8102 universal testing machine by performing tests at room temperature, using a displacement rate of 10 mm min⁻¹. To examine electrolyte affinity, the wettability of the separators was determined via contact angle measurements between the electrolyte and the separator surface. The thermal stability of the separators was gauged by assessing their dimensional change after a heat treatment: samples of 19 mm in diameter were placed in an oven at 60-150°C for 30 minutes, and the subsequent area shrinkage was quantified. To measure the porosity of the separators, the samples were soaked in nbutanol for 2 h and estimated using an equation (1):

$$\text{porosity}(\%) = \frac{M_1 - M_0}{\rho \times V} \times 100\% \quad (1)$$

where, M_0 denotes the weight of the separator prior to immersion in n-butanol, M_1 denotes the weight of the separator following immersion in n-butanol, ρ stands for the density of n-butanol, V refers to the

volume of the separator.

The electrolyte uptake of the separator is calculated using the specified equation (2) [12]:

$$\text{Electrolyte uptake(\%)} = \frac{M_1 - M_0}{M_0} \times 100\% \quad (2)$$

2.3 Electrochemical Measurements

The positive electrode was prepared via slurry coating. Lithium iron phosphate (LiFePO_4), polyvinylidene fluoride (PVDF) and conductive carbon black (Super P) were dispersed in N-methyl-2-pyrrolidone (NMP) at an 8:1:1 weight ratio to form a homogeneous suspension. Subsequently, the suspension was coated onto aluminum foil and dried at 80°C for 12 hours under vacuum. For the electrolyte, a mixed solvent system was used, where 1 M lithium hexafluorophosphate (LiPF_6) was dissolved in a mixture of ethylene carbonate (EC), dimethyl carbonate (DMC) and ethyl methyl carbonate (EMC) at a 1:1:1 volume ratio. The battery was assembled in an argon-filled glove box, and the separators used were a polypropylene (PP) separator and a copper-modified polypropylene separator (NiO@PP), respectively. DC charge-discharge tests were conducted at 25°C using a battery testing system (Xinwei BTS3000, China). The test voltage range was 2.0-4.2 V (vs. Li^+/Li), and the current density was based on $1C = 170 \text{ mAh g}^{-1}$. Cyclic voltammetry (CV) tests were performed using an electrochemical workstation (Chenhua Chi760E, China) at a scan rate of 0.1-0.5 mV s^{-1} within a potential window of 2.5-4.2 V (vs. Li^+/Li). The test frequency range for electrochemical impedance spectroscopy (EIS) was 10 MHz to 100 kHz.

3. Results and Discussion

Fig. 1a reveals that the pristine PP separator possesses a well-defined porous framework with submicron-level apertures. After coating modification, as displayed in Figs. 1b, c and d, NiO particles are evenly dispersed on the PP substrate with the assistance of PVDF binder, presenting a regular spherical morphology without noticeable agglomeration. Such uniform particle distribution generates abundant interconnected voids among NiO grains, which is favorable for electrolyte wetting and lithium-ion diffusion [15, 16]. Cross-sectional SEM observations (Figs. 1e, f, g and h) further verify that the PP matrix maintains intact structural stability. The interface between the NiO coating layer and the PP separator is distinct and compact, suggesting robust interfacial adhesion. Impressively, the thickness of NiO coatings can be precisely regulated at about 5, 10 and 15 μm , respectively. This accurate thickness control plays a vital role in sustaining structural uniformity and guaranteeing long-term cycling durability

of lithium-ion batteries [17].

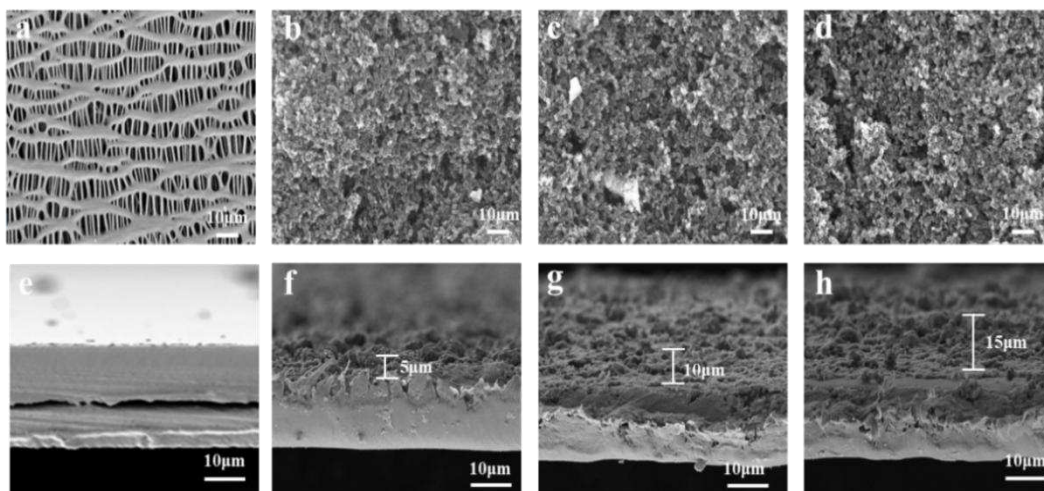


Fig. 1 Surface and cross-section SEM image of the PP (a, e), NiO@PP-5 (b, f), NiO@PP-10 (c, g) and NiO@PP-15 separator (d, h)

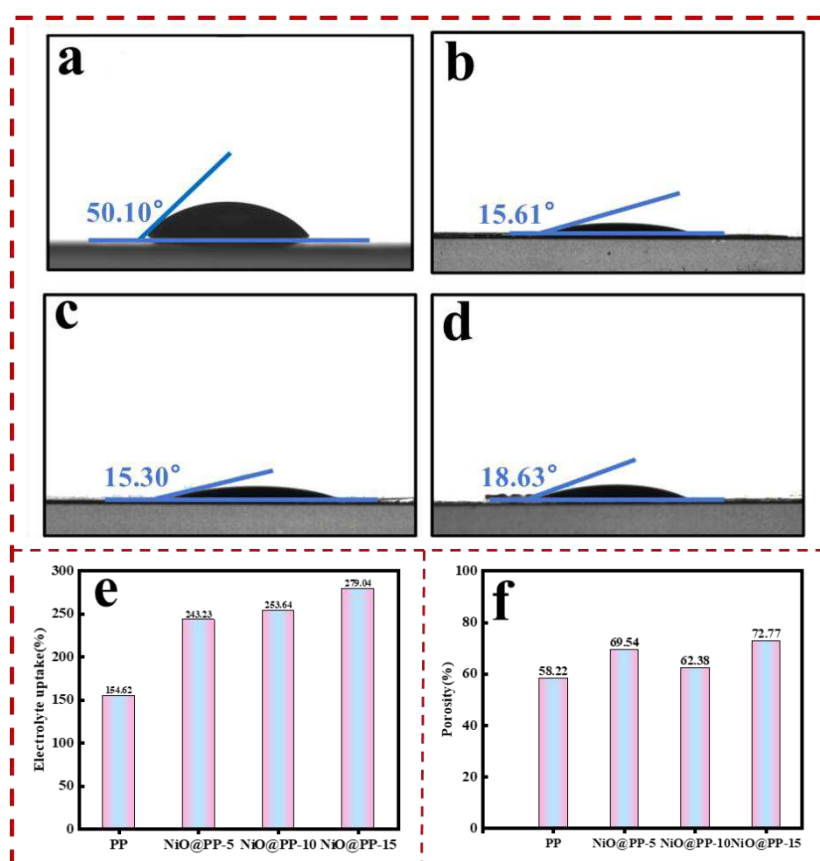


Fig. 2. Contact angle images of (a) the PP, (b) NiO@PP-5, (c) NiO@PP-10, (d) NiO@PP-15 separator after dripping the electrolyte for 10 s, (e) electrolyte uptake of the PP and NiO@PP separators, (f) porosity of the PP and NiO@PP separators.

The wettability of the separator surface is a crucial property that determines its suitability and performance. The electrolyte was applied to four separators, and the results are presented in Figs. 2a, b, c, and d. The NiO@PP separator exhibits a contact angle of 15.61°, 15.30° and 18.63°, significantly lower than the 50.10° of the PP separator, indicating excellent wetting properties. According to Fig. 2f, the porosity of the NiO@PP separator is 69.54%, 62.38% and 72.77%, all higher than that of the PP separator, which is 58.22%. Fig. 2e reveals that NiO@PP separator exhibits higher electrolyte absorption rates compared to PP separator. The electrolyte absorption rate of PP separator is 154.62%, whereas that of nickel oxide@PP separator reaches 243.23%, 253.64%, and 279.04%, respectively.

High thermal stability is critical for maintaining the structural integrity of the diaphragm under elevated temperatures, ensuring it retains its original pore structure, thickness, and flatness without thermal collapse. The coating preserves the porous channels, preventing blockage due to thermal contraction and enhancing thermal stability. Consequently, the diaphragm maintains structural integrity over a broader temperature range and prevents internal short circuits caused by degradation or melting. Fig. 3b compares the morphology of the two separators before and after a 30-minute heat treatment. It shows the separators following exposure to temperatures of 60, 90, 120, and 150°C for 30 minutes in a vacuum oven. The area shrinkage rate, which quantifies the dimensional change, is calculated using the formula (3) [18]:

$$\text{Shrinkage (\%)} = \frac{S_0 - S}{S_0} \times 100\% \quad (3)$$

Where, S_0 and S represent the initial and post-heating areas, respectively.

The PP separator was found to be susceptible to shrinkage as temperature increased. As shown in Fig. 3a, the relationship between material shrinkage rate and temperature demonstrates that as temperature increases, the shrinkage rates of materials such as PP, NiO@PP-5, NiO@PP-10 and NiO@PP-15 all exhibit an increasing trend. The PP material shows the highest shrinkage rate, indicating its inferior thermal stability. In contrast, the NiO@PP-15 material exhibits the lowest shrinkage rate, demonstrating superior thermal stability and suitability for high-temperature environments. When selecting materials, if dimensional stability under high temperatures is a critical consideration, NiO@PP-15 represents the optimal choice. The NiO coating plays a critical role in greatly enhancing the thermal stability of the separator, as shown by these findings.

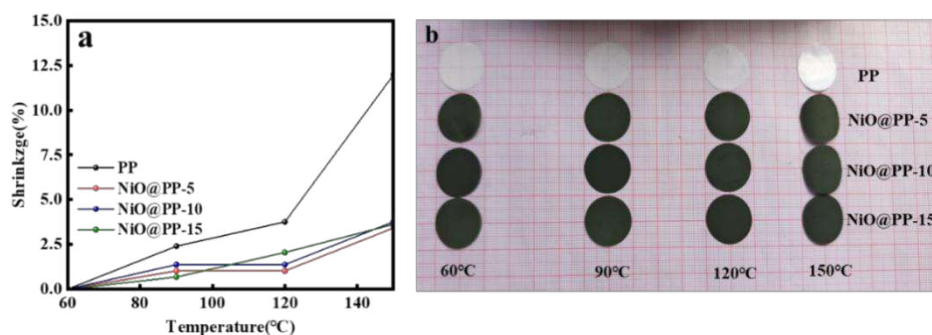


Fig. 3. (a) Thermal shrinkage ratio of the PP and NiO@PP separators after being kept at 60, 90, 120 and 150°C for 30 min; (b) Photographs of the PP and NiO@PP separators after heat treatment.

We use the NiO@PP-5, NiO@PP-10 and NiO@PP-15 separators as the control group for comparison with the battery assembled using the PP separator. Fig. 4a illustrates the discharge capacity with a PP separator and other three types of NiO@PP separators after 100 cycles at a rate of 1C. The initial discharge capacity is 139.37, 149.24, and 140.61 mAh g⁻¹, according to the thickness of the NiO coating, from 5 to 15 μm. After 100 cycles, it increases to 140.83, 145.2 and 142.69 mAh g⁻¹, demonstrating an excellent capacity retention rate of 101.0%, 97.3%, and 101.5%. In contrast, the initial cycle discharge capacity of the battery prepared with PP separator was 139.66 mAh g⁻¹, which decreased to 124 mAh g⁻¹ at the 100th cycle. As shown in Fig. 4c, the rate performance of batteries with different coating thicknesses was evaluated across various current densities ranging from 0.1C to 5C. Compared to the cell prepared using a PP separator, the cell prepared using a NiO@PP separator demonstrates excellent electrochemical performance at extremely high discharge rates. The battery with the NiO@PP separators at least maintains a relatively high discharge capacity of 61.168 mAh g⁻¹ and NiO @ PP-15 even maintaining a discharge capacity of 121.534 mAh g⁻¹ even at an extremely high discharge rate of 5C. Under the same conditions, the cell using the PP separator achieves only 35.566 mAh g⁻¹. This demonstrates that the NiO@PP separator enhances performance by facilitating rapid lithium-ion transport[21, 22]. Fig. 4b illustrates the first charge-discharge curves at 1 C, revealing that the initial discharge capacities for cells using the PP separator and the NiO@PP separators are 138.95, 147.38, 132 and 130.44 mAh g⁻¹, respectively. Remarkably, the voltage difference between the charge and discharge platforms of batteries using the PP and NiO@PP separators represents the magnitude of the internal resistance. A smaller voltage difference indicates lower internal resistance. Therefore, the battery using the NiO@PP separator has smaller internal resistance and lower polarization than the battery using the

PP separator. Fig. 4d illustrates the first charge-discharge curves at 5 C, which is similar to Fig. 4b. The results confirm that the NiO coating significantly enhances the fast charging and discharging capabilities of the battery [19, 20].

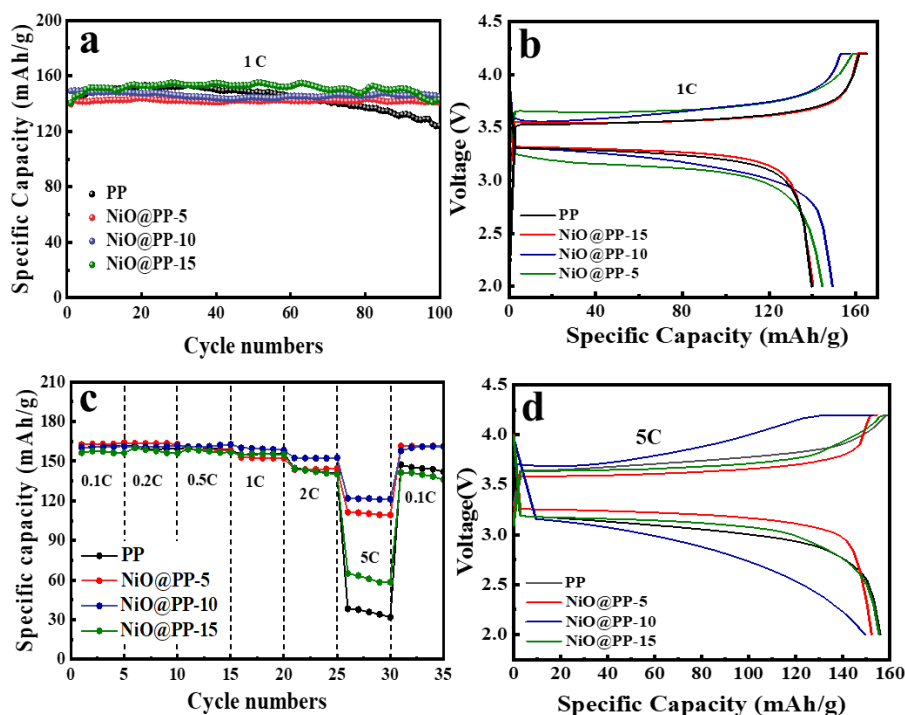


Fig. 4 (a) 1 C cycling capability, (b) The first charge-discharge curves at 1 C, (c) Rate performances at various discharge rates between 0.1 and 10 C, (d) The first charge-discharge curves at 5 C.

The EIS curves of the batteries prepared using the PP separator and the NiO@PP separator are shown in Fig. 5a. From the perspective of electrochemical processes, the high-frequency region (left semicircle) reflects the electron transfer process of the electrode material, where the diameter of the semicircle corresponds to the charge transfer resistance (R_{ct}) [23]. A smaller diameter indicates better conductivity. As depicted in the corresponding figure, the R_{ct} value for the cell using PP, NiO@PP-5, NiO@PP-10 and NiO@PP-15 separators are 320.7, 258.6, 203.3 and 242.3 Ω , respectively [8]. Furthermore, in the Fig. 5c, the low-frequency region (the inclined line on the right) corresponds to the ion diffusion process, showing an impedance characteristic with a slope close to 45° [24]. The conductivity of different materials was compared and analyzed, and the results show that the PP separator

presents the largest semicircle diameter, demonstrating the highest charge transfer resistance (R_{ct}) and the worst electrical conductivity [6,7]. Fig. 5b shows a plot of Z' (the real part of impedance) versus $\omega^{-1/2}$ (the reciprocal of the square root of frequency) derived from an EIS measurement, used to analyze the ion diffusion and charge transfer characteristics of the separators. The figure demonstrates that the Z' values of all samples increase linearly with rising $\omega^{-1/2}$, indicative of typical diffusion behavior and confirming that ion diffusion constitutes one of the primary contributions to impedance. At the same $\omega^{-1/2}$ value, lithium-ion batteries using the NiO@PP-10 separator exhibited the lowest Z' values, reflecting its minimum R_{ct} and ohmic impedance, as well as superior conductivity. Batteries employing PP separators or NiO@PP-5 separators followed, while those with a 15-micrometer NiO coating on the PP separator achieved the highest Z' values and poorest conductivity. Additionally, the slope of the line is inversely correlated with the ion diffusion coefficient-the steeper the slope, the higher the diffusion coefficient and the better the diffusion performance. This further confirms that lithium-ion batteries using a 10-micrometer NiO-coated separator exhibit the fastest ion diffusion. NiO@PP-10 simultaneously achieves the lowest charge transfer impedance and optimal ion diffusion performance. Fig. 5d shows a significant increase in current at 3.5 V on the CV curve, indicating oxidation and the formation of an oxidation peak [25, 26]. The slope corresponding to the current change between 2.75 and 3.25 V on the charge-discharge curve represents the charging plateau of LiFePO_4 . Analysis of the discharge curve clearly demonstrates that as the discharge process approaches completion, the curve shifts to the right, resulting in an increased discharge capacity. This suggests that the copper oxide coating on the separator participates in the electrochemical reaction within the voltage range of 2.75-3.25 V. The essentially constant width of the discharge platform also reflects the similar contribution of cathode LiFePO_4 to the discharge capacity in batteries fabricated with NiO @PP and PP separators. The observed increase in

discharge capacity in NiO@PP equipped batteries can be attributed to the contribution of the copper oxide coating participating in the reaction. The lithium ions may have undergone an electrochemical reaction with the copper oxide coating, as shown by the following chemical equation [9-11]:



Overall, the EIS and CV testing systems investigated the effects of PP separators and NiO-modified PP separators with varying thicknesses on the electrochemical performance of lithium-ion batteries. EIS results demonstrated that NiO modification effectively reduced the charge transfer resistance and enhanced conductivity. Analysis of CV and charge-discharge curves revealed a significant improvement in the discharge capacity of NiO-modified separator batteries.

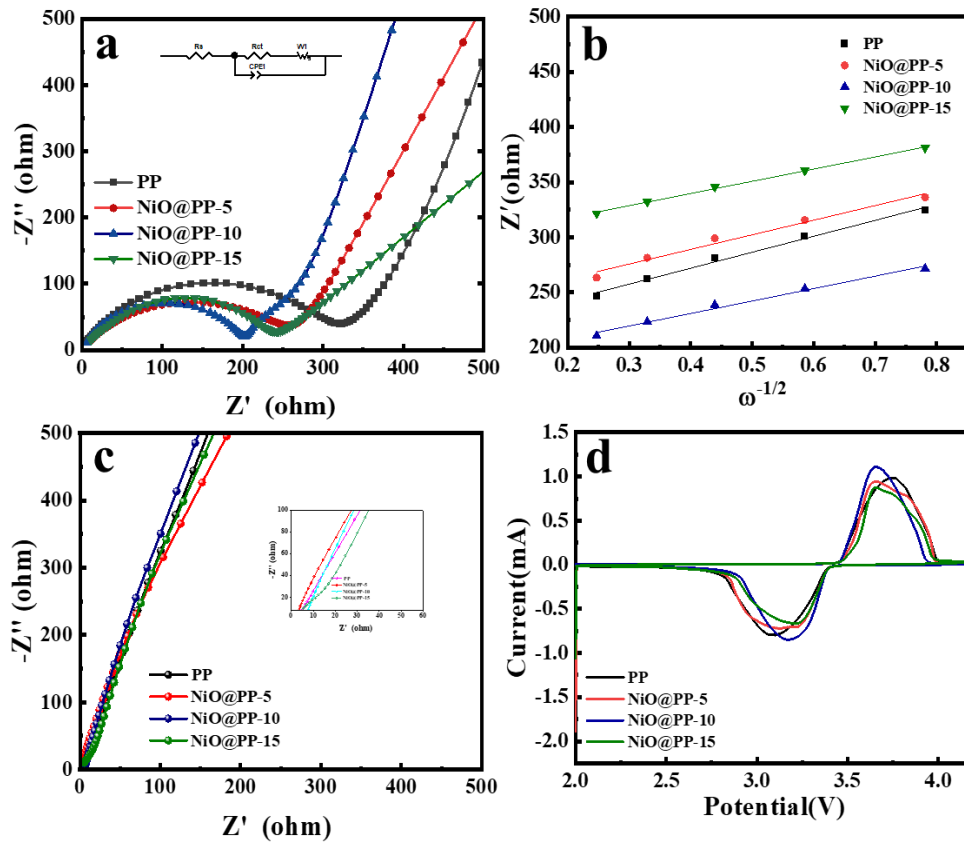


Fig.5. (a, c) EIS plots, (b) Charge transfer resistance-frequency image, (d) CV profiles at scan rates of 0.1, 0.2, 0.3, 0.4 and 0.5 mV/s of PP, and NiO@PP separators.

Furthermore, the lithium-ion diffusion coefficient (D_{Li^+}) can be determined using the Randles-Sevcik equation [13, 14]:

$$I_p = 2.687 \times 10^5 n^3 D^{1/2} A C \gamma^{1/2} \quad (5)$$

Where, n represents the number of electrons transferred in the Fe^{2+}/Fe^{3+} redox couples, D represents the Li^+ diffusion coefficient, A represents the electrode surface area, C represents the lithium-ion concentration and γ represents the scan rate.

The analysis of Fig. 6 reveals significant variations in D_{Li^+} among different samples. PP separators exhibit the highest Li^+ diffusion coefficient at $3.93 \times 10^{-14} \text{ cm}^2/\text{s}$. With varying degrees of NiO modification, the D_{Li^+} of NiO@PP-5, NiO@PP-10, and NiO@PP-15 are measured at 2.73×10^{-14} , 3.77×10^{-14} , and $2.30 \times 10^{-14} \text{ cm}^2 \text{ s}^{-1}$, respectively. This indicates that the incorporation of NiO regulates the lithium diffusion behavior, and variations in NiO thickness lead to systematic changes in the diffusion coefficient. Among the samples, NiO@PP-15 exhibits the weakest lithium diffusion capability, whereas NiO@PP-10 demonstrates the optimal lithium transport performance under the modified conditions.

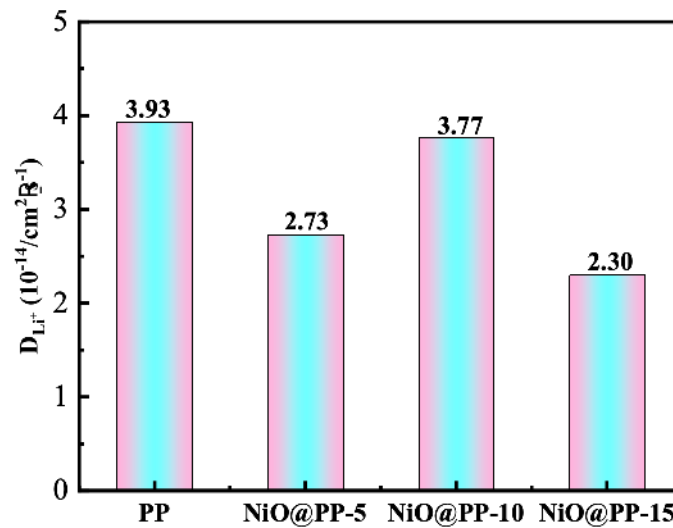


Fig.6 Diffusion coefficients of four samples

4. Conclusion

This study prepared NiO-coated PP separators with varying NiO coating thicknesses (NiO@PP-5, NiO@PP-10, NiO@PP-15) by modifying PP separators with NiO coatings. The results demonstrate that the NiO@PP separators outperform PP separators in terms of wettability, porosity, and electrolyte uptake. Among them, NiO@PP-15 exhibits the best thermal stability with the lowest shrinkage rate at high temperatures. In electrochemical performance tests, NiO@PP-15 shows optimal stability during 1C and 5C cycling, with the slowest capacity decay and the most stable voltage curves. At high rates, its discharge capacity is significantly higher than that of PP separators. EIS and CV tests reveal that NiO@PP-10 has the best conductivity, while NiO@PP-15, despite having a lower Li diffusion coefficient compared to PP and NiO@PP-10, demonstrates superior overall electrochemical performance. Of note, it demonstrated outstanding performance at high rates, retaining a capacity of around 145 mAh g⁻¹ through 100 cycles, while the cells maintained a high capacity retention of 83.5% at a rate of 5C. In conclusion, NiO coating modification effectively enhances the physicochemical and electrochemical properties of PP separators, with NiO@PP-15 providing reliable technical support for high-energy-density, high-rate lithium-ion battery energy storage systems.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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