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A gel polymer electrolyte based on composite of nonwoven fabric and methyl cellulose with good performance for lithium ion batteries

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A new composite gel polymer electrolyte of nonwoven fabric (NWF) and methyl cellulose (MC) with good mechanical property and outstanding thermal and electrochemical stability is prepared by a simple and green casting process followed by absorbing liquid electrolyte. Its characteristics are investigated by scanning electron microscopy, FT-IR, thermogravimetric analysis (TGA). Due to the synergistic action between MC matrix and the NWF framework, the composite gel polymer electrolyte achieves higher ionic conductivity (0.29 mS cm^{-1}) at ambient temperature and larger lithium ion transference number (0.34) than those for the conventional Celgard 2730 separator (0.21 mS cm^{-1} and 0.27, respectively) in 1mol L^{-1} LiPF$_6$ electrolyte, and their activation energies are similar. In addition, the composite membrane shows better mechanical strength than the pure MC membrane. The evaporation rate of the liquid electrolyte at elevated temperature is much decreased. The assembled Li//LiFePO$_4$ cell using this composite gel membrane exhibits better cycling retention and higher discharge capacity than those based on Celgard 2730 separator and pure MC gel membrane. These fascinating characteristics suggest that this unique composite gel polymer electrolyte can be used for lithium ion batteries with good performance and low cost.

1 Introduction

Lithium ion batteries have attracted considerable attention in the past few decades. Their high specific energy, high efficiency and light weight make them promising candidates as the power sources for portable electronics, hybrid electric vehicles, and pure electric vehicles.[1, 2] Moreover, lithium ion batteries could be used as energy storage of wind and solar energy. However, safety issues of lithium ion batteries are of primary concern in their practical applications due to the potential leakage and combustion of organic liquids.[3, 4]

Solid polymer electrolytes (SPEs) consisting of a polar polymer matrix and lithium salts were proposed to solve the above safety problems,[5] and present a great deal of attraction in terms of both fundamental and applied research. However, their lower ionic conductivities ranging from 10^{-8} to 10^{-5} S cm^{-1} at room temperature, poor mechanical strength, and sometimes high sensitivity to humidity of SPEs have limited their uses in large capacity battery modules.[6-11]

Gel polymer electrolytes (GPEs), which are prepared by absorbing liquid electrolyte into the polymer matrix and own the characteristics of both solid and liquid electrolytes, can achieve high ionic conductivity of liquid electrolyte, wide electrochemical window, good compatibility with the electrodes and good thermal stability.[12-16] In the past decades, polymer electrolytes based on polyacrylonitrile (PAN), poly (ethylene oxide) (PEO), poly esters and poly(vinylidene fluoride) (PVDF) have been widely studied.[17-23] Although these polymer electrolytes have appealing advantages, their further development is limited by the poor mechanical strength and high cost.[24] Besides, environment effects should be taken into consideration，the above polymers are not eco-friendly for end use and renewable.[25]

Formerly we reported a new kind of methyl cellulose (MC)-based GPE, which not only can sharply decrease the cost, but also can achieve good thermal stability, high lithium ion transference number and stable cycling performance.[26] More importantly, cellulose, which is abundant and biodegradable, is being widely used because of the increasing demand for environmentally friendly and biocompatible products.[27, 28] However, the MC membrane has poor mechanical strength and low ionic conductivity. As a result, these two problems should be solved prior to their possible application.

Inspired by our former work that composite membrane based on nonwoven fabric (NWF) could sharply decrease the cost together with excellent electrochemical performance and good mechanical strength,[29] here we reported a GPE based on a composite GPE of NWF and MC (MC-NWF). Primary results show that it owns better mechanical strength and higher ionic conductivity than pure MC membrane. It also presents better electrochemical performance compared to commercial separator in some ways. Moreover, the composite GPE exhibits high safety and good electrochemical performance, which is very attractive for application to large-capacity lithium ion batteries.

2 Experimental

Preparation of the composite membrane of MC–NWF is very simple,

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which is schematically shown in Scheme 1, and its process is as follows: methyl cellulose (MC) (Aladdin, CAS 9004-67-5, –OCH_3:25–30%, 1 g) was dissolved in distilled water (40 mL) at room temperature with constant stirring, and then the MC solution was casted on a nonwoven fabric (NWF) (National Bridge Industrial (Shenzhen) Co., Ltd, thickness: 60 µm), which was fixed on a glass plate. After dying at 80 ^{o}C, the other side of NWF was coated by the above process to get the MC-NWF composite membrane (thickness: 60µm). After drying under vacuum at 80 ^{o}C for 24 h, the pieces were soaked in the organic electrolyte 1 mol L^{-1} $LiPF_6$ solution in EC/DMC/EMC (ethylene carbonate/dimethyl carbonate/ethyl methyl carbonate, 1/1/1, w/w/w, Zhangjiagang Guotai-Huarong New Chemical Materials Co., Ltd) over 12 h in a glove box to obtain the composite GPE for further measurement.

A field emission scanning electron microscope (FESEM, Hitachi FEG-S4800) was used to observe the morphology of the prepared polymer membranes. FT-IR measurement was carried out on a BRUKER VECTOR-22 spectrometer. Thermal stabilities of the polymer membranes were measured by thermogravimetric analysis using a Perkin-Elmer TGA7. A stress–strain test was conducted using a Sansi YG832 tensile testing machine with a crosshead speed of 1 mm min^{-1}.

The amount of liquid electrolyte uptake (η) is calculated using Eq. (1).

$$\eta = \frac{W_t - W_0}{W_0} \times 100\% \qquad (1)$$

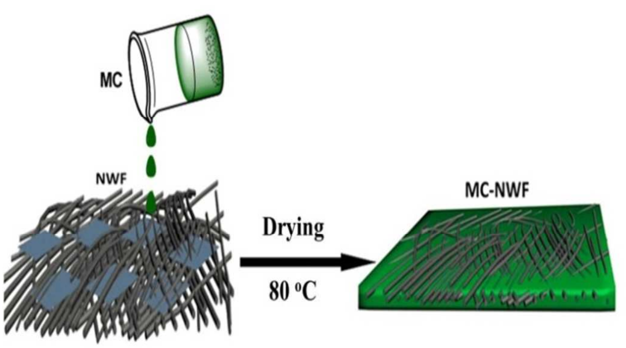
where η is the uptake of liquid electrolyte, W_0 and W_t are the weight of the membranes before and after absorption of the liquid electrolyte, respectively.

The ionic conductivities (σ) of the GPEs were measured in the temperature range of 298–348 K by electrochemical impedance spectroscopy (EIS) on an electrochemical working station CHI660C (Chenhua). The samples were measured in blocking-type cells in the frequency range 10 Hz–100 kHz which were fabricated by sandwiching the membranes between two stainless steel electrodes.

The ionic conductivity was calculated from Eq. (2).

$$\sigma = \frac{l}{R_b A} \ (\mathrm{Scm^{-1}}) \qquad (2)$$

where σ is the ionic conductivity, l is the thickness of the polymer membrane, R_b is the bulk resistance and A is the area of the stainless steel electrode.



Scheme 1 The preparation process of MC-NWF composite.

Linear sweep voltammetry measurements were conducted to determine the electrochemical stability of the GPEs. The GPE membrane was sandwiched between a lithium anode and stainless steel electrode. The measurement was done between 2 and 6 V (*vs*. Li^+/Li) at a scan rate of 2 mV s^{-1} on an electrochemical work station (CHI660C, Chenhua). In order to calculate the lithium ion transference number of the membranes, the chronoamperometry profile was obtained by the electrochemical working station measuring in blocking-type cells, in which the gel membrane was sandwiched between two lithium metal electrodes.

Electrochemical performance of the membranes was evaluated by CR2025 coin-type cells with lithium metal as the counter and reference electrode. The mixture of $LiFePO_4$ (E60, China), acetylene black and PVDF in the weight ratio of 8: 1: 1 as the working electrode. The composite GPE membrane was used as the separator and electrolyte. All cells were assembled in an argon-filled glove box with water and oxygen content lower than 1 ppm. The cyclic voltammogram was obtained between 2.5 and 4.2 V at a scan rate of 0.1 mV s^{-1}. The cycling performance of the cells was carried out by a Land battery tester (CT2001A) at the current density of 0.2 C between 2.5 and 4.2 V.

3 Results and discussion

Fig. 1 shows the FESEM micrographs of the nonwoven fabric (NWF) and the composite membrane (MC-NWF). Apparently, the pore size of the fibrous matrix of NWF is too large to be used as separator for lithium ion batteries, and the uniformity of the pores is also lower than that of the current commercial separators. After forming the composite membrane with MC, a thin solid layer of MC was observed on the surface (Fig. 1d and 1e). The pores of the fibrous matrix of NWF and the inner space of the NWF membrane are also filled with MC matrix (Fig. 1e and 1f). This is evidently different from the well-reported and used separators, which is porous. This unique characteristic indicates that this solid structure could prevent the micro short-circuits of lithium ion batteries such as from the peeling-off of electrochemical active particles from the electrode pellets. From the process shown in Scheme 1, it is clear that this way is very simple and does not contain any unfriendly organic solvent. From the viewpoint of manufacturing, it is of great attraction.

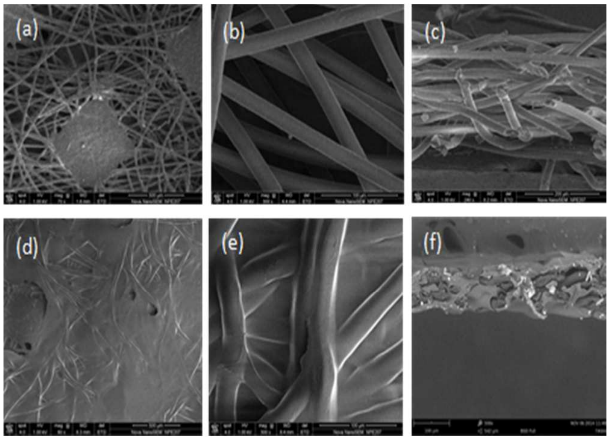


Fig. 1 SEM micrographs of (a and b) surface and (c) cross-section of NWF, (d and e) surface, (f) cross-section of MC-NWF.

The FT-IR spectra of MC, NWF and the MC–NWF composite are shown in Fig. 2a. The typical absorption peaks of MC are observed at 3000–2800 cm^{-1} (C–H stretching in CH_2 and CH_3), 1646 cm^{-1} (C–C ring stretching), 1460 cm^{-1} (C–H bending in -CH_2), 1300–1000 cm^{-1} (C–O stretching).[30] Since the methylation is not 100%, there are still some remained–OH groups, which are at 3446 cm^{-1} as a large absorption peak. The NWF shows a wide peak at 3000–2800 cm^{-1} due to C–H of –CH_3, –CH_2– and –CH– stretching vibrations from its polypropylene (PP) matrix, and the peaks at 1460 and 1380 cm^{-1} can be ascribed to the asymmetric and symmetric bending vibrations of methyl and methylene groups, respectively .[31] In the case of MC-NWF, all the typical peaks of NWF and MC are present clearly. This indicates that the composite MC-NWF is successfully obtained. Fig. 2b shows the tensile strength of the Celgard 2730 is nearly 40 MPa, while that of MC membrane was only 20 MPa. In the case of MC-NWF membrane, the maximum of stress is 28 MPa, which are smaller than that of Celgard 2730. In comparison with pure MC, it is increased, indicating that the MC-NWF composite membrane could be used for lithium ion batteries.

The thermal stability is a key point for polymer electrolytes during application in lithium ion batteries. As shown in Fig. 2c, pure MC does not appear obvious weight loss until 300 ^{o}C, the membrane of MC-NWF is thermally stable up to nearly 350 ^{o}C. In the case of the NWF, which mainly consists of polypropylene (PP), it appears obvious weight loss at 400 ^{o}C. Fig. 2d depicts the thermal gravimetric curves of the gel MC-NWF membrane, pure MC membrane and Celgard 2730 separator soaked with the same amount of the electrolyte (0.6 mL g^{-1}). The organic electrolyte in the MC begins to evaporate at about 70 ^{o}C. In terms of the MC-NWF, the absorbed electrolyte starts to evaporate at 65 ^{o}C, which evaporated at the same temperature as that for Celgard 2730. However, its evaporation rate is much slower than that of the commercial separator. As shown in Fig. 2d, even when the temperature increases to 140 ^{o}C, only about 30% weight loss was observed, indicating that most of the electrolyte is still remained in the composite membrane. Obviously, the organic electrolyte can be absorbed and retained by the MC matrix in the composite membrane.[26] It is known that PE (polyethyelen) and PP will be melt down at about 120 and 150 ^{o}C, respectively. The former component in the separator can lead to the so-called shut-down behavior. However, if all electrolyte is evaporated before 120 ^{o}C, the effects of the shut-down really do not contribute much to the safety of lithium ion battery since there is no liquid electrolyte left in the separator. This is the main reason why there were so many accidents even the separator with shut-down behavior was used. In our case, when the temperature increases, the liquid electrolyte is evaporated much slower. This will reduce the possibility of physical explosion caused by high pressure from the evaporated organic solvents. This is the main reason why GPEs are of great attraction since it can lead to improved safety.

The uptake amount of a membrane is the main factor for ionic conductivity, and that of the MC-NWF can be up to 80.7 wt.%, which is a little lower than 90.9 wt.% of the commercial separator Celgard 2730.[32] The former is due to the interaction between carbonate-based organic electrolytes and the remained hydroxyl groups in the MC matrix and the latter is due to absorption in the pores.[33] Fig. 3a shows the ionic conductivity dependence on temperature of the MC-NWF composite GPE at the temperature range from 25 to 75 ^{o}C. As expected, the ionic conductivity increases with temperature, exactly obeying the typical Arrhenius behavior of lithium ion polymer electrolytes.[34] The activation energy (E_a) of MC-NWF is 0.024 eV, just a little higher than that of Celgard 2730 (0.023eV) as we reported formerly.[33] The inset of Fig. 3a depicts the impedance plots of the composite MC-NWF, from which we could know that the ionic conductivity of the gel MC-NWF composite electrolyte at 25 ^{o}C is 0.29 mS cm^{-1}, higher than that of Celgard 2730 (0.21 mS cm^{-1}) and MC membrane (0.20 mS

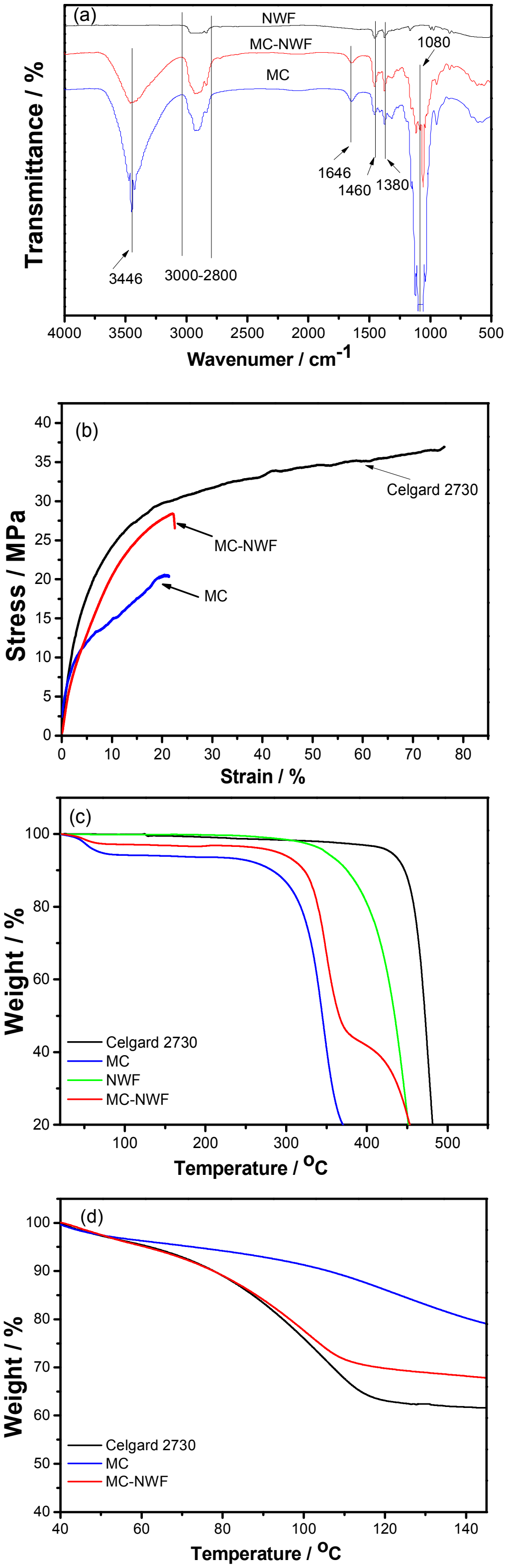
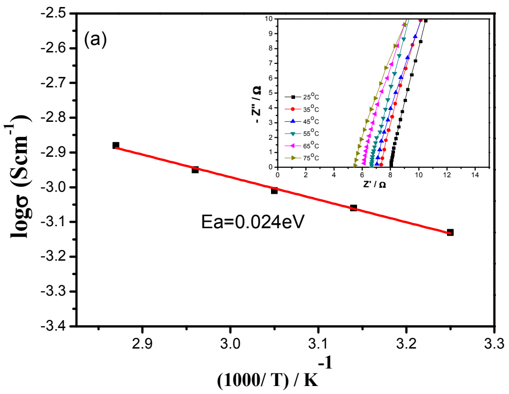


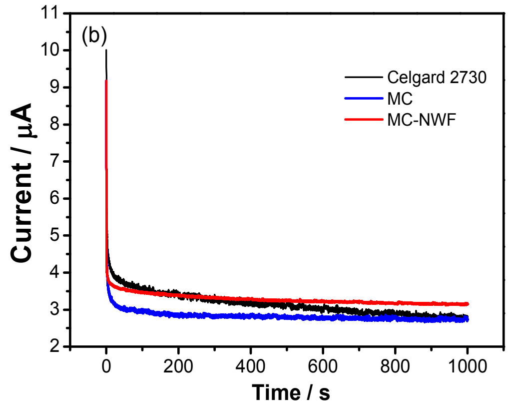
Fig. 2 (a) IR spectra of MC, NWF and MC-NWF; (b) Stress-strain curves of Celgard 2730, MC, and MC-NWF; (c) Thermogravimetric (TG) curves of Celgard 2730, MC, NWF and MC-NWF under air and the temperature rising rate is 10 ^{o}C min^{-1}; (d) TG curves of

Celgard 2730, MC, and MC-NWF membrane after absorbed the same amount of 1 mol L^{-1} $LiPF_6$ electrolyte (0.6 mL g^{-1}) at the temperature rising rate of 2 °C min^{-1} under nitrogen.

cm^{-1}) saturated with the organic electrolyte.[26] The main reason is that the synergistic action between MC matrix and the NWF framework plays a key role in improving the performance of the MC-NWF composite GPE, which is similar to the composite of PVDF with NWF. [29] On the one hand, MC matrix, including a part of hydroxyl groups, could interact with carbonate-based organic electrolytes to achieve higher uptake amount of liquid electrolyte and lithium ion transference number. On the other hand, NWF consisting of a single polyolefin, or a combination of polyolefins, owns some advantages of commercial separator, such as good stability with Li metal and good mechanical property.

The lithium ion transference number (t_+) is obtained by comparing the final and initial current. Fig. 3b shows the transference number (t_+) of lithium ion for Celgard 2730, pure MC and the MC-NWF gel composite electrolyte, which is an important parameter for lithium ion batteries. It is clear that t_+ for Celgard 2730 is 0.27, while t_+ of composite MC-NWF is 0.34, higher than that of pure MC (t_+=0.30). As to the reason, it is perhaps due to the synergic action between MC and NWF, which is similar to that between PVDF and NWF.[29] However, further work and evidence are needed. A linear sweep voltammetry experiment performed in the potential range of 0-6.0 V (vs. Li^+/Li) at the scan rate of 1 mV s^{-1} is shown in Fig. 3c. It depicts that the current flow is very small when the voltage is below 5 V (vs. Li^+/Li), indicating the suitability for the application in lithium ion batteries.





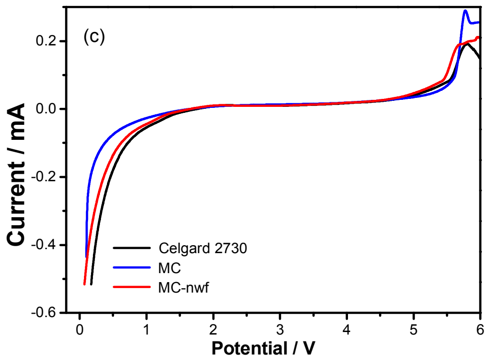


Fig. 3 (a) Impedance plots of the conductivity data at different temperatures and Arrhenius plots of MC-NWF; (b) chronoamperometry profiles for the Celgard 2730, MC, and MC-NWF saturated with the liquid electrolyte at 25 °C in block cells using Li metal as both electrodes with a step potential of 10 mV; (c) linear sweep voltammograms of Celgard 2730, MC, and MC-NWF composite membrane after saturated with 1 mol L^{-1} $LiPF_6$ electrolyte.

The electrochemical performances of all electrolyte membranes were tested in coin cells, where a lithium metal was used as the reference and counter electrode while $LiFePO_4$ was used as positive electrode. Fig. 4a shows that the oxidation and reduction peaks of $LiFePO_4$ using the MC-NWF gel composite electrolyte appear at around 3.7 and 3.1 V (vs. Li^+/Li) in the CVs, respectively. The potential separation between two peaks is 0.6 V, a little larger than that with commercial separators (0.5 V) , which may be caused by the left hydroxyl groups in the MC.[26] While there is some influence on the charge and discharge performance (shown in Fig. 4b). When the MC-NWF gel composite electrolyte was tested as the separator and electrolyte in Li//$LiFePO_4$ cell, the reversible capacity of the positive electrode is about 140 mAh g^{-1} at 0.2 C, higher than those for pure MC and Celgard 2730, about 130 mAh g^{-1} and 120 mAh g^{-1}, respectively. The reason is that both ionic conductivity and transference number of Li^+ ion are improved for the MC-NWF composite GPE. Fig. 4c shows the cycling performance and coulombic efficience of $LiFePO_4$ between 2.5 and 4.2 V (vs. Li^+/Li) using all the electrolyte membranes at 0.2 C (34 mA g^{-1}). After 55 cycles, there is still no evident fading for the MC-NWF composite GPE, similar to commercial separator, and the Coulombic efficiency could also keep stable (nearly 98%), which is higher than that for MC and lower than that for Cegard 2730, indicating that the MC-NWF GPE displays good cycling stability. This is consistent with the above results. Since there is no –OH group for Celgard 2730, its contact with Li metal is very stable and the Coulombic efficiency is nearly 100%. In the case of MC, some –OH groups will react with Li and the Coulombic efficiency is lower. In the case of the composite, due to the introduction of PVDF, the contact of –OH groups with Li metal is markedly reduced. As a result, its Coulombic efficiency is higher than that for MC.

4 Conclusions

An environmentally friendly polymer material, methyl cellulose (MC), was successfully used to form composite with nonwoven fabric (NWF). The MC-NWF composite displays good thermal stability and good mechanical properties. The ionic conductivity of the prepared composite gel polymer electrolyte at ambient temperature is higher than that of the commercial separator (Celgard

2730) and pure MC membrane, and it also presents higher transference number of Li^+ ion. The evaporation rate of the liquid electrolyte at elevated temperature is much slower than that for the commercial separator, suggesting good safety performance for lithium ion batteries. Furthermore, when the MC-NWF composite gel polymer electrolyte was tested as separator and electrolyte, a $LiFePO_4$ positive electrode displays superior electrochemical properties including high discharge capacity and stable cycling. These results show that this composite gel polymer electrolyte is attractive for lithium ion batteries requiring good performance with low cost.

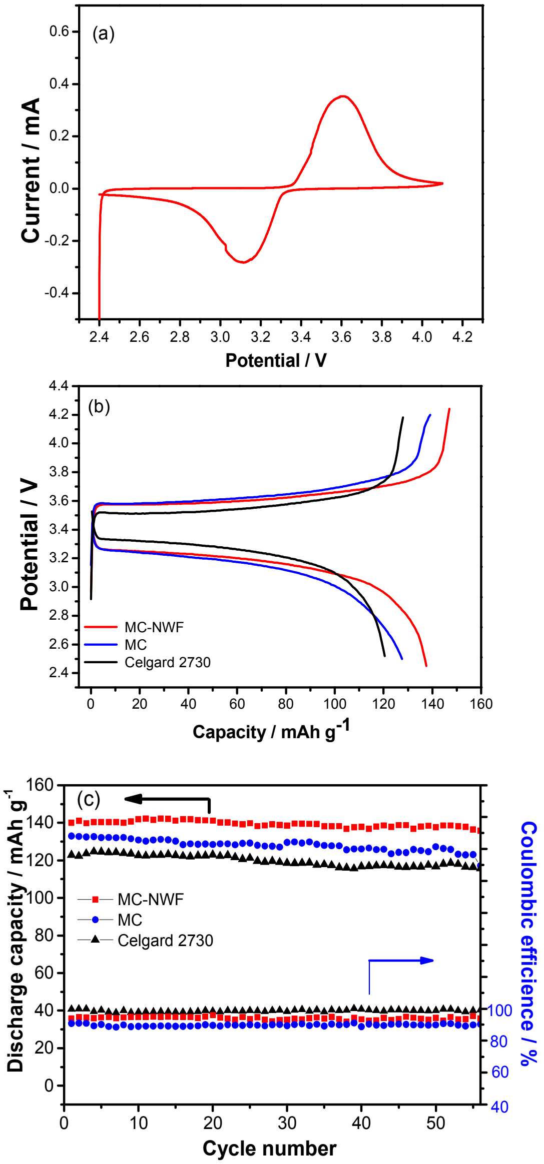


Fig. 4 Electrochemical performance of the $LiFePO_4$ positive electrode tested by using Celgard 2730, MC, and the MC-NWF composite membrane saturated with 1 mol L^{-1} $LiPF_6$ electrolyte and Li metal as the counter electrode: (a) cyclic voltammetric (CV) curves of the MC-NWF composite GPE at the scan rate of 0.1 mV s^{-1}; (b) charge-discharge curves of Celgard 2730, MC, and the MC-NWF composite GPE at 0.2 C; (c) cycling behavior of Celgard 2730, MC, and the MC-NWF composite GPE at 0.2 C.

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A new composite gel polymer membrane of nonwoven fabric (NWF) and methyl cellulose (MC) exhibiting good mechanical property and outstanding thermal and electrochemical stability is prepared by simple casting green process.