Stable Cellulose Separator Based on Sodium Chloride for Lithium-Ion Battery

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Research Article

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Abstract

In this study, we fabricated porous membrane based on cellulose acetate by adding NaCl for a low cost and pro-environmental process. We succeeded in manufacturing the cellulose acetate (CA)/NaCl composites with small micro-sized pores for stable separator. NaCl existed as ionic aggregates in CA polymer and disrupts the interaction between CA chains. Thus, the CA/NaCl composites were plasticized. Plasticized regions of the CA/NaCl composites became small micro-sized pore, applying hydrostatic force at 8 bar. The pore morphology of surface and cross section of the CA/NaCl composites were confirmed by SEM. The small micro-sized pores were measured by water flux. In addition, the interaction between NaCl and functional group of CA was measured by using FT-IR spectroscopy. TGA was used to identify for thermostability of the CA/NaCl composites matrix.

Introduction

Porous materials have been important in various fields.(Cheng et al. 2020, Costa et al. 2019, Kumar et al. 2019, Slater and Cooper 2015, Sun et al. 2016, Gunathilake et al. 2017, Zhai and Jana 2017) Among porous materials, porous membranes are used in chemical products and various fields. The porous membrane is used to purify wastewater or to remove contaminants floating in the air as a filter in biology, environmental engineering, etc.(Cheng et al. 2020, Gunathilake et al. 2017, Zhai and Jana 2017) It is also applied as a separator to separate the positive and negative electrodes of batteries in the electrochemistry.(Arora and Zhang 2004, Cannarella et al. 2014, Costa et al. 2019, Lagadec et al. 2016, Zhang 2007) In the battery, the separator separates the cathode and the anode to prevent an internal short circuit and serves as a passage for lithium ions to move between the cathode and the anode.(Arora and Zhang 2004, Costa et al. 2019, Zhang 2007) Separator must withstand the harsh environment thermally and electrochemically inside the battery. Thus, it must be thermally, chemically and electrochemically stable to both the electrode and electrolyte, and it must have good mechanical strength. (Cannarella et al. 2014, Lagadec et al. 2016) The materials mainly used for battery separator are polyolefin materials such as polypropylene and polyethylene. It is economically advantageous because it is inexpensive and has good mechanical properties.(Choi et al. 2001, Heidari and Mahdavi 2020, Lagadec et al. 2018) Thus, it is widely used as a battery separator. However, polypropylene separator has a low thermal decomposition temperature. Therefore, there is a problem of stability such as battery explosion when the battery become overheated. To increase the stability of the polypropylene separator, a method of coating ceramic particles on the polypropylene separator was used.(Choi et al. 2010, Liu et al. 2014, Shi et al. 2014) *Liu et al.*, coated the $\text{Al}_2\text{O}_3/\text{SiO}_2$ composite ceramic layer on both sides of the polypropylene separator to not only strengthen the thermostability and mechanical strength of the polypropylene film, but also improve the electrolyte absorption and ionic conductivity of the separator surface.(Liu et al. 2014) However, the ceramic coating method is expensive and there are still problems for stability, such as a battery fire, since polypropylene is decomposed at a low temperature. Therefore, a battery separator was created based on cellulose acetate (CA), which has a higher thermal decomposition temperature than polypropylene (Asghar et al. 2020, Bhute and Kondawar 2019, Chen et al. 2018) and

high stability was achieved by using electrospinning technique to manufacture a thermally and mechanically stable battery separator.(Boriboon et al. 2018, Chen et al. 2020, Yvonne et al. 2014, Zhang et al. 2013) In addition, there was a study to develop the process as an environmentally and low-cost process by using an additive in a cellulose acetate-based separator.(Hong et al. 2020, Kim and Kang 2021, Lee et al. 2021, Lee and Kang 2019, 2020) *Lee et al.*, prepared a film by adding CaCl_2 to a CA solution made using acetone and H_2O at a mol ratio of 1:0.4, and applied water pressure to 8 bar to prepare a straight porous separator.(Lee et al. 2021) *Kim et al.*, added lactic acid to the CA matrix with hydrostatic force to develop a separator with better thermal stability than separator consisting of polypropylene and polyethylene.(Kim and Kang 2021) Since high water pressure at 8 bar is applied to the film, a straight, porous separator can be easily fabricated. H_2O and Acetone, which are used as solvents for dissolving CA, are cheap and widely used industrially. They can be easily separated and recycled. Therefore, it was possible to develop an eco-friendly and low-cost battery separator process.

In previous studies, *Lee et al.*, demonstrated that an advanced porous separator can be fabricated by adding an inorganic compound such as a metal salt to a CA polymer solution.(Lee and Kang 2019, 2020) $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ was added to cellulose acetate to control the pore size on the surface of the CA matrix. (Lee and Kang 2019) In addition, using $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, the $\text{Zn}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}/\text{CA}$ separator was thermally stable and showed a water flux of $182 \text{ L/m}^2 \text{ h}$ at a water pressure at 8 bar.(Lee and Kang 2020) *Hong et al.*, fabricated a thermally stable CA film with nano-pores by adding glycerin, an organic molecule, to cellulose acetate polymer without using a metal salts.(Hong et al. 2020) However, the method has the disadvantage that the additives used to make the film are expensive, even if the results are meaningful. In this study, NaCl, the cheapest among salts, was applied to a low cost and pro-environmental process based on cellulose acetate. The suggested method has the advantage of being environmentally and low cost by using CA and H_2O . NaCl is cheaper than organic materials and metal salts used as additives. In addition, NaCl is the most common salts and the cheapest among inorganic salts. Therefore, NaCl was added to the process to fabricate a CA-based separator for practical applications.

Experimental

Sodium chloride and cellulose acetate (CA, $\text{Mn} = \sim 50,000$) were purchased from Sigma Aldrich Chemical CO. Acetone (Daejung Chemical & Metals) was used as the solvent. All materials were used without pretreatment.

A 10 wt% neat CA solution was prepared by dissolving cellulose acetate polymer in a mixed solvent of acetone/water (wt/wt 8:2 ratio). NaCl was added to the 10 wt% neat CA solution as the mole ratio of 1:0.04. The solution was then stirred for 24 hr. After that, the film was fabricated by a free-standing method using a doctor blade on the glass plate. It was dried in a thermo-hygrostat at 25°C and 50% humidity for 30 min. The dried film was placed in water pressure treatment equipment. And then, the mol ratio 1:0.04 CA/NaCl matrix was applied by water-pressure from 2 to 8 bar. At this time, each time the

pressure was increased by 1 bar, it was stabilized for 30 minute and the flux was measured. The unit of Flux is $\text{L}/\text{m}^2\text{h}$ (LMH). After that, the membranes were dried for 24 hr.

The surface and cross section of the CA/NaCl membrane was observed using a SEM (JSM-5600LV, JEOL). The interaction force between the functional group of cellulose acetate polymer and NaCl was analyzed by FT-IR spectroscopy (VERTEX 70/70V FT-IR spectrometers, Bruker Optics). The thermal stability of the CA polymer membrane was measured using TGA (Universal V4. 5A, TA instruments)

Result & Discussion

SEM

SEM was used to determine the size and shape of pores on the surface of the CA membrane and the morphology of cross-section. Figure 1. is the surface of neat CA film. As shown in Figure 1. a) and b), the surface of neat CA film has almost no pores and is pristine. Figure 2. a), b) and c) show the surface of 1:0.04 CA/NaCl membrane before applying water pressure. As shown in Figure 2. a), a few of pores were formed on the surface of 1:0.04 CA/NaCl at 0 bar. In constant, Figure 2. c) has more pores on the surface than Figure 2. a). Figure 2. b) shows that the pores were formed with a larger size than Figure 2. a) and c). The reason that the size and number of pores are different on the surface of 1:0.04 CA/NaCl at 0 bar is that H_2O solvates NaCl as the form of ionic aggregates. NaCl in the state of ionic aggregates is relatively larger. Therefore, As shown in Scheme 1. B, NaCl permeates between the cellulose acetate chains, increasing the distance between the chains. When the CA/NaCl membrane is dried, H_2O is evaporated. As water evaporates, the regions occupied by H_2O forms the pores. At this time, depending on the dispersion of NaCl, the size and the number of ionic aggregates of NaCl change and the number and size of pores are varied. Figure 3. a) and b) show the surface of 1:0.04 CA/NaCl membrane treated with water pressure at 8 bar. As shown in Figure 3. a), many pores were formed and there were dents on the surface of 1:0.04 CA/NaCl at 8 bar. Figure 3. b) is an enlarged view of the surface of the membrane. In Figure 3. b), pores existed as smaller than $10\text{ }\mu\text{m}$ were formed. This indicates that NaCl was dispersed in the CA matrix and small sized pores were formed due to the bottleneck phenomena when high water-pressure was applied. Figure 4. a) is a cross-section of a 1:0.04 CA/NaCl membrane at 0 bar. In the cross-section, the region with pores is the part where H_2O is solvating NaCl. H_2O was evaporated as the membrane was dried, forming the pores on the cross section. Figure 4. b) shows a cross section of a 1:0.04 CA/NaCl at 8bar. As shown in Figure 4. b), the cross-section has a sponge form. As the distance between the cellulose polymer chains increased as NaCl was added, the CA/NaCl composite was plasticized. When high hydraulic pressure is applied to this polymer, the plasticized part of the CA matrix is pierced by the hydraulic pressure, creating the pores and creating a nearly linear channel.

TGA

TGA was used to compare the thermal stability of neat CA, 1:0.04 CA/NaCl at 0 bar, and 1:0.04 CA/NaCl at 8 bar. Figure 5. a) shows the thermal decomposition temperature of neat CA, 1:0.04 CA/NaCl at 0 bar,

and 1:0.04 CA/NaCl at 8 bar. Figure 5. b) is an enlarged graph of Figure 5. a). In Figure 5. b), 1:0.04 CA/NaCl composite at 0 bar had the lowest thermal decomposition temperature among the three composites. Neat CA film was decomposed at about 300 °C. however, the pyrolysis temperature of 1:0.04 CA/NaCl at 0 bar is 249 °C. This indicates that the thermal stability of 1:0.04 CA/NaCl at 0 bar decreases as the flexibility of CA membrane increases. NaCl added to CA was present as ionic aggregates, preventing the interaction between CA chains and increasing the flexibility. As a result, the CA matrix was plasticized and the pyrolysis temperature was lowered. On the other hand, the 1:0.04 CA/NaCl composite at 8 bar was decomposed at about 264 °C, which is higher than the thermal decomposition temperature of 1:0.04 CA/NaCl at 0 bar. This means that the thermostability of 1:0.04 CA/NaCl at 8 bar increased since NaCl added to the CA/NaCl composite was removed partially by water treatment, as shown in scheme 1 C. When NaCl was partially removed by water pressure, the interaction between CA polymer chains is recovered. Thus, pyrolysis temperature of 1:0.04 CA/NaCl at 8 bar was recovered.

Infrared spectroscopy

FT-IR spectroscopy was used to identify the interaction force between the functional group of the cellulose polymers and NaCl. Figure 6. is an analysis of the functional group of CA. Figure 6. a) is ether peaks of neat CA, CA/NaCl at 0 bar and CA/NaCl at 8 bar. The peak of three membranes was 1033 cm^{-1} for ether of CA polymer. According to Table 1. and Figure 7, there was no remarkable peak change in the deconvolution of ether peaks. Therefore, it was thought that ether of CA was not interacting with NaCl.

Figure 6. b) is carbonyl group in IR spectrum of neat CA, CA/NaCl at 0 bar, CA/NaCl at 8 bar. The peak of all membranes was 1737 cm^{-1} by the carbonyl group. However, the slight shift was observed for FT-IR spectra. Therefore, the shift was analyzed by deconvolution. (Figure 8) According to Table 2, in neat CA, the area in the range of 1728 cm^{-1} occupied 14.27 %. For the area in the range of 1729 cm^{-1} , 44.67 % was observed and the area at 1749 cm^{-1} occupied 41.06 %. When NaCl was added to neat CA polymer, the area decreased to 6.39 % in the range of 1728 cm^{-1} , and the ratio of the area in the range of 1729 cm^{-1} increased to 50.05 %. In other words, when NaCl was added to neat CA polymer, the proportion of areas with relatively high wavenumbers increased. This indicates the vibration of the C=O bond is strengthened by NaCl. Looking at Scheme 1 A, neat CA polymer chains exists close to each other. Therefore, the intermolecular interaction between the carbonyl groups of the chain became strengthened. However, when NaCl is added to CA polymer, NaCl is solvated as ionic aggregates and penetrated into CA chains. Thus, as in Scheme 1 B, the intermolecular interaction between carbonyl groups is weakened by the increasing distance between cellulose polymer chains. As a result, the C=O bond becomes stronger and vibrated at a high wavenumber.

Figure 6. c) is the hydroxyl group for IR data of neat CA, CA/NaCl at 0 bar, CA/NaCl at 8 bar. The peak at 3490 cm^{-1} was observed for neat CA, and at 3483 cm^{-1} for the NaCl-added CA composites. This is the peak shifted by adding NaCl to the CA polymer. Therefore, the shift was analyzed by deconvolution. (Figure 9) According to Table 3, when NaCl is added to neat CA, the ratio of the area in the wavenumber of 3493 cm^{-1} increased by 4.5 %, and the ratio of the area in the wavenumber of 3616 cm^{-1} decreased by

4.5 %. However, when 1:0.04 CA/NaCl membrane was applied by water pressure at 8 bar, the ratio of the area in the two ranges was not significantly changed. Even though the change in ratio of area was not exactly observed, this change means the hydroxyl group of CA polymer was interacting with NaCl. NaCl is interacting with the hydroxyl group of CA polymer while the distance between the cellulose acetate chains became lengthened like Scheme 1 B. The interaction of hydroxyl group with NaCl would have been observed at a relatively low wavenumber. However, when CA/NaCl membrane was applied by water pressure at 8 bar, NaCl is partially removed. Thus, the intermolecular interaction between the hydroxyl group and NaCl become recovered to a higher wavenumber. However, since the amount of NaCl to be removed was imperceptible, no significant change was observed in the IR data.

Table 1. deconvolution of ether group IR data

Wave number	Neat CA	CA / NaCl 0bar	CA / NaCl 8bar
985.6 cm ⁻¹	6.000 %	6.370 %	6.260 %
1034 cm ⁻¹	80.01 %	81.15 %	79.94 %
1069 cm ⁻¹	9.250 %	8.970 %	9.540 %
1087 cm ⁻¹	4.740 %	5.510 %	4.26 %

Table 2. deconvolution of carbonyl group IR data

Wave number	Neat CA	CA / NaCl 0bar	CA / NaCl 8bar
1728 cm ⁻¹	14.27 %	6.390 %	7.540 %
1729 cm ⁻¹	44.67 %	50.05 %	48.74 %
1749 cm ⁻¹	41.06 %	43.56 %	43.72 %

Table 3. deconvolution of hydroxyl group IR data

Wavenumber	Neat CA	CA / NaCl at 0 bar	CA / NaCl at 8 bar
3493 cm ⁻¹	89.12 %	93.62 %	95.74 %
3616 cm ⁻¹	10.88 %	6.380 %	4.260 %

Water Flux

By adjusting the ratio of NaCl, the pore size of the CA/NaCl membrane was controlled. When the water flux was measured by applying a water pressure at 8 bar to the CA/NaCl membrane, the flux didn't show a

proportional relationship as the mol ratio of NaCl increased. This means that there is an optimal ratio in which NaCl can be well dispersed. According to Table 4, the composition of NaCl that produced the best flux is 0.02 ratio, and the average flux is 4.84 L/m²h. However, 4.711 L/m²h, a value similar to the average flux of 0.02 ratio of NaCl, is measured at 0.04 ratio, and the reproducibility is also enhanced. This indicates that micro-sized plasticized parts were formed in the CA matrix by NaCl and as water pressure was applied to the CA/NaCl membrane, small-sized pores were formed, resulting in relatively low flux.

Table 4. Flux data based on NaCl mol ratio

ratio	Flux (L/m ² h)	Flux (L/m ² h)	Flux (L/m ² h)	Average Flux (L/m ² h)
0.040	22.909	7.149	0.000	4.711
	3.781	4.578	5.230	
	4.310	2.998	4.930	
0.020	4.204	3.854	4.512	4.840
	304.694	27.549	54.801	
	6.789	54.410	13.938	

Conclusion

In this study, we used NaCl as an additive, which is the cheapest of the additives used for the low cost and pro-environmental process. When NaCl is added to cellulose acetate, it exists as ionic aggregates in CA polymer and increases the distance between CA polymer chains. Due to these phenomena, the CA matrix becomes flexible and plasticized. The pores formed on the surface and the sponge-shaped pores in the cross-section of the mol ratio 1:0.04 CA/NaCl membrane were observed by using SEM. Furthermore, it was confirmed by using FT-IR that NaCl interacts with the functional group of CA. Through FT-IR spectroscopy, it was confirmed that NaCl interferes with the intermolecular interaction between the carbonyl groups and interacts with the hydroxyl group of CA. Based on the TGA data, the thermal decomposition temperature of the mol ratio 1:0.04 CA/NaCl membrane applied water pressure at 8 bar was higher than that of the CA/NaCl membrane at 0 bar. Through these properties, it was found that when high water pressure was applied to the CA/NaCl film, NaCl was removed partially, and the thermal decomposition temperature was recovered. On the other hand, the water flux of the mol ratio 1:0.04 CA/NaCl composite was measured as 4.711 L/m²h, and reproducible data were obtained. It means that NaCl at 0.04 mol ratio was relatively well dispersed compared to other compositions and formed small micro-sized pores. Finally, we demonstrated that porous polymers can be fabricated by applying NaCl for low cost and pro-environmental process.

Declarations

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Schemes

Scheme 1 is available in the Supplemental Files section

Figures

Figure 1

a) and b) surface of neat CA membrane surface without water pressure and NaCl

Figure 2

a), b) and c) surface of a mol ratio 1:0.04 CA/NaCl membrane without water pressure

Figure 3

a) surface of a mol ratio 1:0.04 CA/NaCl membrane with water pressure at 8 bar, b) enlargement of surface of a mol ratio 1:0.04 CA/NaCl membrane with water pressure at 8 bar

Figure 4

a) cross section of a mol ratio 1:0.04 CA/NaCl membrane without water pressure, b) cross section of a mol ratio 1:0.04 CA/NaCl membrane with water pressure at 8 bar

Figure 5

a) TGA data of Neat CA, 1:0.04 CA/NaCl membrane at 0 bar, 1:0.04 CA/NaCl membrane at 8bar, b) enlargement of a)

Figure 6

IR data of Neat CA, 1:0.04 CA/NaCl membrane at 0 bar, 1:0.04 CA/NaCl membrane at 8 bar

Figure 7

a) Ether group IR data deconvolution of Neat CA film, b) Ether group IR data deconvolution of 1:0.04 CA/NaCl film at 0 bar, c) Ether group IR data deconvolution of 1:0.04 CA/NaCl film at 8 bar

Figure 8

a) carbonyl group IR data deconvolution of Neat CA film, b) carbonyl group IR data deconvolution of 1:0.04 CA/NaCl film at 0 bar, c) carbonyl group IR data deconvolution of 1:0.04 CA/NaCl film at 8 bar

Figure 9

a) hydroxyl group IR data deconvolution of Neat CA film, b) hydroxyl group IR data deconvolution of 1:0.04 CA/NaCl film at 0 bar, c) hydroxyl group IR data deconvolution of 1:0.04 CA/NaCl film at 8 bar

Supplementary Files

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- [Scheme1.png](#)