Analysis of the Degradation of PE Separators for Lithium-ion Batteries under Extreme Conditions

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Introduction

Reason for choosing this issue

- Separators allows lithuim ions to pass through submicron-sized pores while preventing a short circuit due to contact between cathode and anode.
- Damage of the separator may cause an internal short circuit, which directly cause fire and explosion accidents.
- Currently, the demand for lithium-ion batteries for EVs and ESS, which require long-term use under various conditions, is increasing rapidly.
- At this point, it is necessary to study the degradation pattern of the separator under various conditions. For this study, purpose is to understand the degradation mechanism of the separator exposed under extreme conditions.

Experimental

Progress

Disassemble of waste LIBs for celluar phone → Obatined separator reference → Reassemble into pouch cell → Storage under extreme condition → Disassemble of pouch cell & Obatained separator → Separator Washing & Analysis

Storage conditions

Storage for 5 days

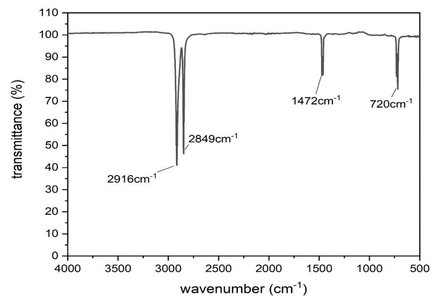
- High temperature (110℃)
- Distilled Water
- Salt solution (20% NaCl)
- Acid solution (pH 2)

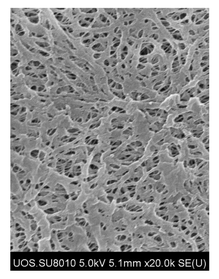
Analysis instrument

- UTM (Tensile Test)
- FT-IR
- FE-SEM
- EDS

Separator from the waste LIBs for cellular phone

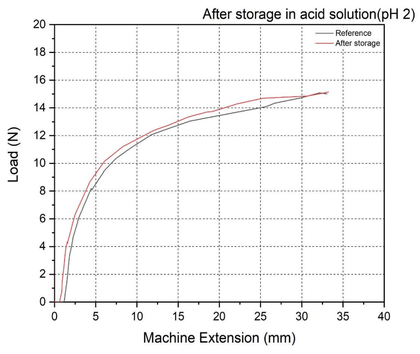
Materials: PE separator prepared by wet process

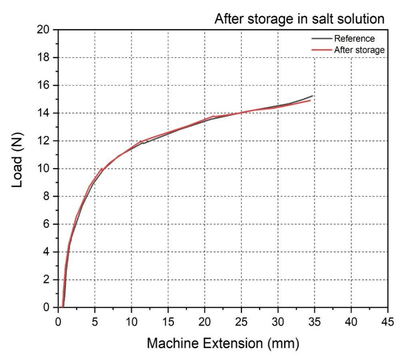


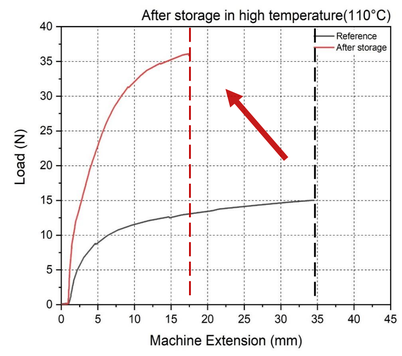


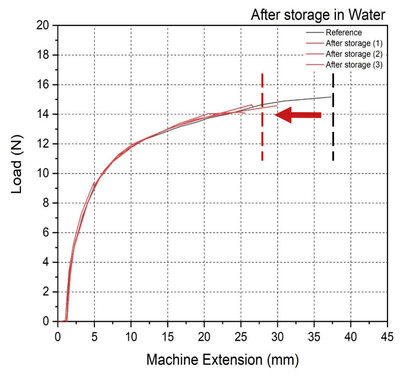
Results & Discussion

Universal testing machine (UTM)







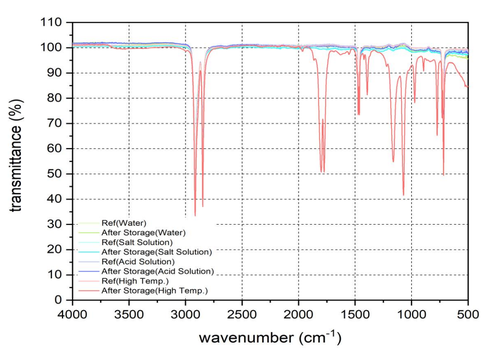


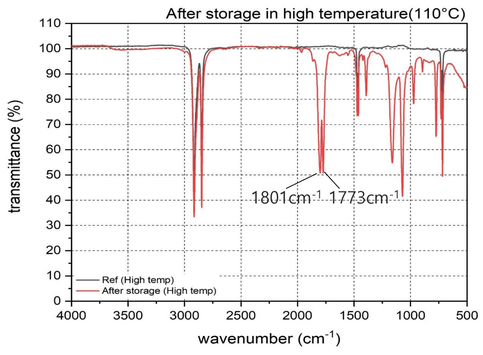
High Temperature	Tensile strength (MPa)	Elongation at break (%)
Reference	0.0751	33.532
After storage	0.1804	16.407

Water	Tensile strength (MPa)	Elongation at break (%)
Reference	0.0758	36.294
After storage	0.0719	28.968

- After storage at high temperature, tensile strength increased and elongation decreased.
- Elongation decreased after storage at distilled water.
- After storage at acid and salt condition, there were not changed.

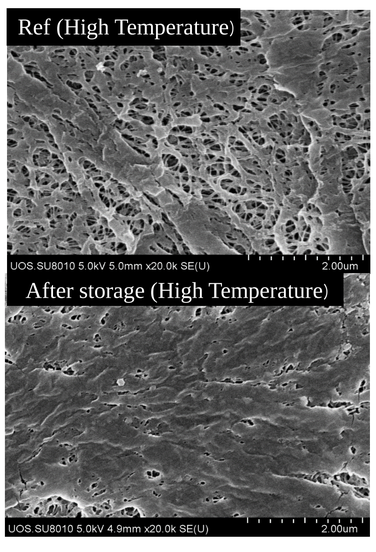
FT-IR

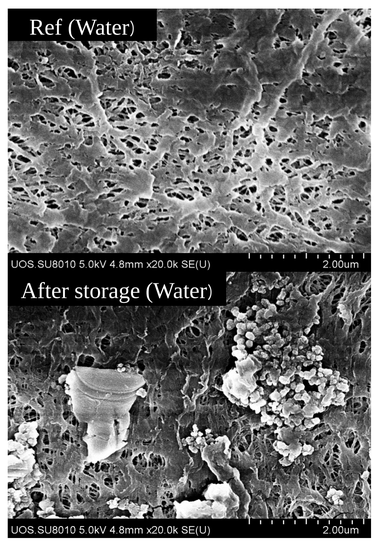




- At high temperature, a new peak was identified at 1801, 1773 cm^{-1}
  : Cyclic carbonate (C=O stretching) in EC electrolyte
- All other conditions were the same as reference peak.

FE-SEM & EDS



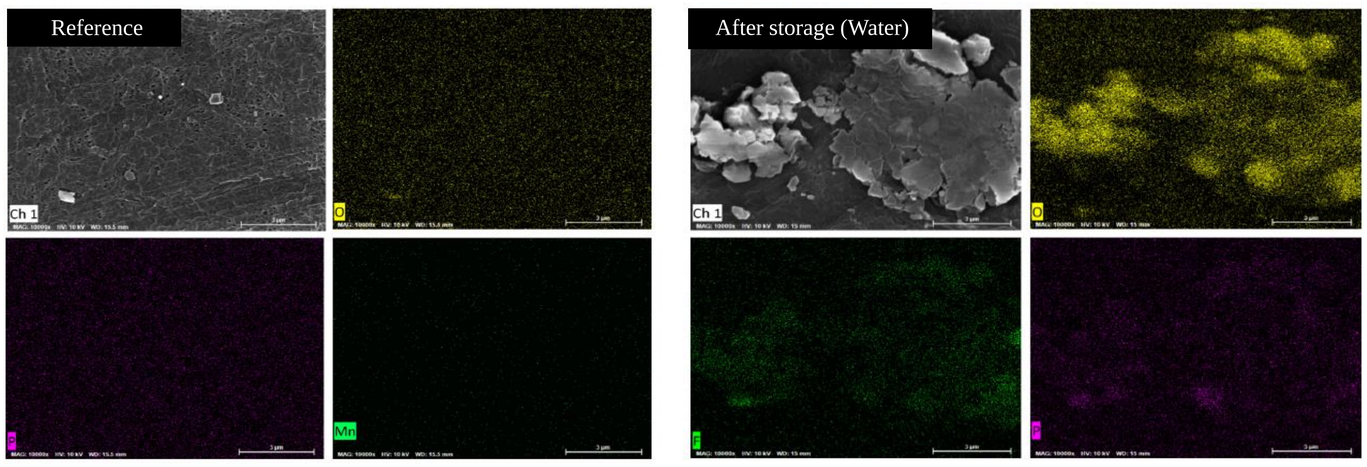


FE-SEM

- Pore was blocked after high temperature.
- Particles were absorbed in distilled water condition.
- No change at acid and salt condition.

EDS

- Components of particles which were discovered in distilled water condition were consisted of O, P, F.

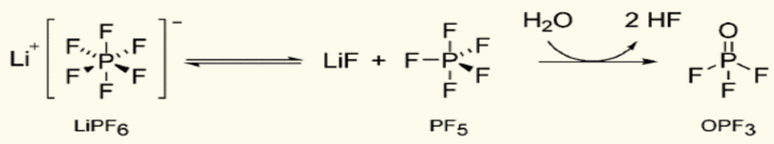


[After storage in high temperature]

- 110℃ is sufficient to melt separator. It was confirmed that the pore was blocked in FE-SEM, the tensile strength changed in UTM and EC peak was identified in FT-IR.
- In conclusion, the EC of the electrolyte and PE were reacted and it has a significant impact on the separator.

[After storage in distilled water]

- It was confirmed that particles were absorbed on the surface in FE-SEM.
- Based on the result of EDS analysis, $LiPF_6$ degradation had occurred through the following mechanism.



- The difference in concentration between the electrolytes in separator and the distilled water resulted in the escape of these particles from the separator. It made an empty space of the separator. Thus the elongation in UTM decreased.

[After storage in acid and salt solutions]

- They had not changed in FT-IR and UTM. Because the difference in concentration between the electrolytes in separator and the solution was not as large as the distilled water condition.

Conclusions

- In high temperature condition, PE structure was changed as PE melted at 110℃ and cooled again. So dramatically increased load was observed in UTM. Considering a peak occurred at 1773, 1801 cm^{-1} in FT-IR, melted PE reacted with EC (cyclic carbonate peak).
- In distilled water condition, there was a change of tensile strength because of a large amount of particle production.
- In acid and salt extreme conditions, these conditions did not significantly affect the degradation of the separator

References

[1] Sem, Vilma et al. “Identification of historical polymers using Near-Infrared Spectroscopy.”. Polymer Degradation and Stability. (2014) 107

[2] Bernardine L. D. Rinkel, et al. “Electrolyte Oxidation Pathways in Lithium-Ion Batteries” Journal of the American Chemical Society (2020) (142) 35

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