

# *CHAPTER 9*

SUMMARY AND CONCLUSION

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In this study, chitosan with 85% deacetylation was used. Chitosan, lithium acetate dihydrate and dispersoids (aluminium oxide and cerium oxide) were mixed in certain weight percentages and dissolved in 100ml of 0.5% acetic acid solution. These solutions were then poured into petry dishes and left at room temperature for film formation. It was found that precipitation of dispersoids started to occur for samples with 6wt% of  $\text{Al}_2\text{O}_3$  and  $\text{CeO}_2$  and above, which the precipitation can be observed with naked eyes.

FTIR has shown that there is interaction between chitosan, LiOAc and the dispersoid, where changes in vibrational modes are observed. The interaction between chitosan and lithium acetate occur with and without introduction of dispersoid ( $\text{Al}_2\text{O}_3$  and  $\text{CeO}_2$ ). The relative intensity of C-O vibrations around  $1100\text{cm}^{-1}$  changed. The intensity increased as the concentration of  $\text{Al}_2\text{O}_3$  increased. The amine group spectrum at around  $1600\text{cm}^{-1}$  with double peaks splits with increasing  $\text{Al}_2\text{O}_3$  concentration. At the same time, the C-H vibration at around  $3000\text{ cm}^{-1}$  shifted to lower wavenumbers. This changes have showed the occurrence of complexation. Only C-H vibration peak broaden when  $\text{CeO}_2$  concentration increased, showing that there might be minimal interaction between  $\text{CeO}_2$ , chitosan and LiOAc salt.

The electrical conductivity of all samples was calculated using bulk resistance value obtained from the complex admittance plot. The highest electrical conductivity at room temperature obtained for the film containing 2 wt%  $\text{Al}_2\text{O}_3$  which is  $8.92 \times 10^{-9} \text{ Scm}^{-1}$ , 28 times higher than the original film with no added  $\text{Al}_2\text{O}_3$ . While  $\text{CeO}_2$  samples stay at the same magnitude with varying  $\text{CeO}_2$  concentration. This observation is explained by its crystallinity nature using X-ray diffraction (XRD) technique. The dielectric behavior and modulus formalism shows that the samples of  $\text{Al}_2\text{O}_3$  and  $\text{CeO}_2$  are ionic conductors.  $\text{Al}_2\text{O}_3$  dispersed samples are better ionic conductors.

The crystallinity of all samples was examined using XRD. Chitosan is an amorphous material. When lithium acetate was added, the film exhibited lithium acetate peaks with XRD at  $25.9^\circ$  and  $30.7^\circ$ . The peaks were depressed when 1 wt%, 2 wt% and 3 wt% of  $\text{Al}_2\text{O}_3$  was added. Subsequent addition of  $\text{Al}_2\text{O}_3$  caused the crystallinity to increase where LiOAc peaks and  $\text{Al}_2\text{O}_3$  are obvious. Sample with 2 wt% of  $\text{Al}_2\text{O}_3$  showed the least crystallinity. This was proven by the calculated Scherrer length.

For  $\text{CeO}_2$  dispersed samples the  $\text{CeO}_2$  peaks are observed even in sample C1. This is because the overall crystallinity of the samples did not decrease as the way it happened to  $\text{Al}_2\text{O}_3$  dispersed samples. The increase in concentration of  $\text{CeO}_2$  caused the sample to become more crystalline which was clear when Scherrer length was calculated.

Thermal analysis showed that all films showed good stability up to 350°C. Three melting temperatures were observed using TGA. They are at 58°C, 285°C and 460°C, which correspond to the melting temperature for lithium acetate dihydrate, pure lithium acetate and lithium hydroxide respectively. The same melting points were observed in DSC scan, with melting temperatures 8°C lower compared to TGA values.  $T_g$  value for chitosan is at 220.35°C. Other electrolytes prepared do not show any  $T_g$  value. No prominent shift of melting point is observed. This inferred that the electrolytes become more crystalline compared to the polymer host.

Surface morphology study with SEM showed small grains in the sample with highest conductivity (2 wt%  $\text{Al}_2\text{O}_3$ ) compared to the sample without  $\text{Al}_2\text{O}_3$ . This is different from the flaky layer observed in samples where precipitation is seen with the naked eye. Golodnitsky and coworkers (1996) have suggested that the addition of small particle size (about 150Å) oxides increases the degree of the local crystal disorder of the PEO-LiI complexes by preventing agglomeration of the chains. The small  $\text{Al}_2\text{O}_3$  particles may enhance interfacial conduction at the grain boundaries of the Li-P(EO)<sub>n</sub> (n<6)- $\text{Al}_2\text{O}_3$  small disorder crystals. It has been widely accepted that the mechanism of enhancement is through generation of excess defects in the matrix phase in the vicinity of the dispersoid (Kumar and Shahi, 1994).

We have successfully shown that the chitosan film can be used as an electrolyte. The conductivity values need to be improved by decreasing the

degree of crystallinity. This can be achieved by adding suitable plasticizers. Further study need to be carried out to study on the feasibility of the formation of this new electrolyte for the application in solid state polymer cell and other electrochromatic devices.

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