

CHAPTER V

CONCLUSIONS

The esterification of cotton in DMAc/LiCl system was investigated by using stearic acid as an esterified agent, toluenesulfonyl chloride as catalyst and pyridine as co-catalyst /medium under microwave irradiation. After esterified procedure, the resulting products, cotton stearates were characterized. Cotton stearate films were prepared by casting method. Finally, the effects of % esterification of cotton stearate on the physical, mechanical and biodegradable properties of cotton stearate films were investigated. The results are summarized as follows.

1. The optimum conditions for carrying out esterification of cotton in DMAc/LiCl system using stearic acid, toluenesulfonyl chloride, and pyridine under microwave heating are 2eq./OH of stearic acid, 2eq./OH of TsCl, 4 eq. pyridine/TsCl and power output 270 watts for duration time 3.30 min. The resulting product is cotton stearate with 155.88% yield and 71.38% esterification without degradation of cotton and side reaction of tosylation and chlorination.

2. Cotton stearate exhibited evidence of esterification as evidenced by the presence of three important ester bands at 2850 cm^{-1} (methyl absorption), 1752 cm^{-1} (carbonyl of ester) and decrease in the intensity at $3000\text{-}3650\text{ cm}^{-1}$ (hydroxyl). As %esterification of cotton stearate increased, the cotton stearate exhibited strong carbonyl of ester and methyl absorption with considerable decrease in the intensity of hydroxyl group of cellulose.

3. Cotton stearate displayed three significant weight losses at about $220\text{ }^{\circ}\text{C}$, $250\text{-}370\text{ }^{\circ}\text{C}$, and $430\text{-}460\text{ }^{\circ}\text{C}$ which are corresponding to the decomposition of aliphatic hydrocarbon chain of stearic acid, cotton and lignin respectively.

4. Cotton stearate revealed side-chain crystallization and amorphous region by the presence of melting peak at a temperature ($T_{m,L}$) of $24.82\text{ }^{\circ}\text{C}$ and barely visible second-order (T_g) at $128.69\text{ }^{\circ}\text{C}$, respectively.

5. Based on water absorption and wettability of the surface, cotton stearate is more hydrophobic than unmodified-cotton and its solubility is proportional to the %esterification.

6. The gloss values of cotton stearate films increased significantly with increasing %esterification of cotton because of increasing of surface regularity and the highest gloss value was about 50 units (standard black calibration = 95.1 units).

7. The results on mechanical properties showed that the tensile properties of cotton stearate film were dependent on acyl substitution content of stearic acid. The increasing of tensile strength and tensile modulus as increasing of acyl substituent due to the parts of the side chain that overlap and crystallize and becoming larger with increasing of acyl substituent. The slightly increasing of % elongation as increasing of acyl substitute may probably due to the internal plasticization effect from non-alignment part or crystallized part of long chain aliphatic (stearic) leading to an increase in the mobility between chains.

8. The cellulose stearate films showed potential of biodegradation as evidence by increasing in the amount of cavities and %weight loss. The potential also increased with % esterification of cellulose due to their nature abundance of both cellulose and increasing of stearic acid.

9. SEM micrographs of cotton stearate revealed large granule with rough surface while short fiber with smooth surface was observed in unesterified-cotton. The different morphology was resulted from aggregation of acyl group on cotton-esterified surface in which its dimension was increased with an increasing of % esterification.