CHAPTER –1 SYNTHESIS, CHARACTERIZATION AND APPLICATION OF BIODEGRADABLE POLYESTERS

1.1. Introduction

1.2. Source of monomer

1.2.1 Source of L-lactic acid

1.3. Production of L-lactic acid

1.3.1 Fermentation route to lactic acid

1.3.2 Isolation of lactic acid

1.4. Purity and impurities in lactic acid

1.4.1 Chemical purity

1.4.1a Purification of fermentation-produced lactic acid

1.4.1b Analytical methods for purity determination

1.4.2 Optical purity

1.5. Production and purification of lactide

1.6. Synthesis and characterization of poly (lactic Acid)s

1.6.1. Dehydropolycondensation of lactic acid

1.6.2. Dehydropolycondensation of L-lactic acid using zeolites

1.6.3. Isolation, purification, homopolymerization and copolymerization of aleuritic acid with L-lactic acid:

1.6.3a. Chemical Structure of Shellac

1.6.3b. Isolation of different constituent of Shellac
1.6.3c. Isolation of aleuritic acid
1.6.3d. Physical properties
1.6.3e. Isomers of aleuritic acid
1.6.3f. Homopolymer and copolymer of aleuritic acid
1.6.3g. Uses of aleuritic acid
1.6.4. Dehydropolycondensation of L-lactic acid with 12-hydroxy stearic acid

1.7. Ring Opening polymerization of lactide
1.7.1. Coordination-insertion polymerization
1.7.2. Cationic polymerization
1.7.3. Anionic polymerization
1.7.4. Nucleophilic polymerization

1.8. Structure and properties of poly (lactic acid)s
1.8.1. General structure-property relationship
1.8.2. Stereocomplex

1.9. Properties of copolymers

1.10. Stability of polymer
1.11. Depolymerization

1.12. Degradation Mechanisms and Degradability
1.12.1. Hydrolytic / Enzymatic
1.12.2. Thermo-oxidative

1.13. Conclusion and Future Directions

1.14. References

CHAPTER – 2
SCOPE AND OBJECTIVE OF THE PRESENT INVESTIGATION

2.1. Objectives of the present thesis

2.2. Approaches
CHAPTER 3
ANALYSIS OF L-LACTIC ACID AND POLYLACTIC ACID

3.1. Introduction

3.2. Materials and Methods

3.2.1. Materials

3.2.2. Preparation of ethyl lactate from L-lactic acid

3.2.3. General procedure for ROP of L-lactide

3.3. Analysis

3.4. Results and Discussion

3.4.1. Impurity detection and analysis in the L-lactic acid and ethyl L-lactate

3.4.1a L-lactic acid

3.4.1b Ethyl lactate

3.4.2. Synthesis and characterization of linear PLA oligomers of controlled number average molecular weight and with both carboxylic and hydroxyl end group

3.4.3. Determination of molecular weights of oligomers

3.4.4. Thermal characterization (DSC) and powder XRD of oligomers

3.5. Conclusion

References

CHAPTER 4
SYNTHESIS OF POLY (L-LACTIC ACID) BY DEHYDROPOLYCONDENSATION USING VARIOUS PORE SIZES OF ZEOLITES AND DETERMINATION OF SEQUENCE BY $^{13}$C NMR METHOD

4.1. Introduction

4.2. Experimental part

4.2.1. Synthesis of distannoxane catalysts
CHAPTER 5: HOMO AND COPOLYMERIZATION OF ALEURITIC ACID WITH L-LACTIC ACID AND STUDY THE AGGREGATION BEHAVIOR IN DIFFERENT SOLVENTS

5.1. Introduction

5.2. Experimental

5.2.1. Materials and Method

5.2.2. Synthesis of methyl ester of aleuritic acid

5.2.3. Synthesis of protected aleuritic acid

5.2.4. Homo polymerization of ProAL

5.2.5. Deprotection of homopolymer

5.3. Characterizations

5.4. Result and discussion

5.4.1. Molecular weight determination
5.4.2. Thermal analysis 144
5.4.3. $^{13}$C Nuclear Magnetic Resonance Spectroscopy 144
5.4.4. Effect of catalyst concentration on polymerization reaction 146
5.4.5. Effect of reaction temperature on polymerization reaction 147
5.4.6. TEM analysis 149

5.5. Copolymerization of L-lactic acid with aleuritic acid 151
5.5.1. SEC Analysis 154
5.5.2. Nuclear Magnetic Resonance 157
5.5.3. Thermal properties 157
5.5.4. Transmission Electron Microscopy (TEM) 158

5.6. Conclusion 162

References 163

CHAPTER 6: GRAFTING OF POLY (L-LACTIC ACID) AND COPOLYMER OF L-LACTIC ACID WITH 12-HYDROXY STEARIC ACID ON THE SURFACE OF MWCNTS 167

6.1. Introduction 167
6.2. Experimental 169
6.2.1. Materials 169
6.2.2. Purification of MWCNTs 169
6.3. Characterization 169
6.4. Result and Discussion 171
6.4.1. Molecular weight determination 172
6.4.2. FT-IR 172
6.4.3. Thermogravimetric analysis 173
6.4.4. Scanning electron microscopy 175
6.4.5. Transmission electron microscopy 178
6.4.6. Atomic force microscopy

6.4.7. $^{13}$C Cross Polarization/Magic Angle Spinning ($^{13}$C CP/MAS)

6.5. Grafting of copolymer on the surface of MWCNTs

6.5.1. Molecular weight determination

6.5.2. $^1$H NMR of copolymer

6.5.3. FT-IR

6.5.4. Thermo gravimetric analysis

6.5.5. Scanning electron microscopy

6.5.6. Transmission electron microscopy

6.5.7. Atomic Force Microscopy

6.5.8. $^{13}$C Cross Polarization Magic Angle Spinning ($^{13}$C CP/MAS)

6.6. Conclusion

References

CHAPTER 7: HOMOPOLYMERIZATION AND COPOLYMERIZATION OF L, L-LACTIDE WITH e-CAPROLACTONE IN PRESENCE NOVEL OF ZINC PROLINATE DERIVATIVES

7.1. Introduction

7.2. Experimental

7.2.1. Materials and methods

7.2.2. Synthesis of zinc prolinate catalysts

7.3. Characterization

7.4. Results and Discussion

7.4.1. General procedure for synthesis of PLA by ring opening polymerization

7.4.2. Molecular weight determination

7.4.3. Thermal Analysis

7.4.4. Nuclear Magnetic Resonance Spectroscopy (NMR)
7.4.5.  $^{13}$C Cross Polarization/Magic Angle Spinning ($^{13}$C CP/MAS) NMR spectra

7.4.6.  MALDI-ToF-MS analysis

7.5.  Synthesis of copolymer of L, L-lactide with ε-caprolactone

7.5.1.  FT-IR

7.5.2.  Molecular weight determination

7.5.3.  Thermal characterization

7.5.4.  Determination of polymer structure by NMR

6.5.4a.  Determination of copolymer structure by NMR

7.5.5.  MALDI-ToF-MS analysis

7.5.5a.  Determination of comonomer incorporation as well as end groups by MALDI-TOF MS

7.5.6.  Mechanistic discussion

7.6.  Conclusion

References

8.0  SUMMARY AND CONCLUSIONS

LIST OF PUBLICATIONS