

SYNTHESIS OF PLA USING FROM LACTIDE MONOMER AND STUDY ABOUT MECHANICAL PROPERTIES USING ORGANOCCLAYS

Soumya Ranjan Kar*¹

*¹associate Professor, Mechanical, S.V.S.E & T, Bhubaneswar, Odisha, India

DOI : <https://www.doi.org/10.56726/IRJMETS33161>

ABSTRACT

For past many years a number of aliphatic polyesters have aroused considerable interest due to their biodegradability nature and biocompatibility with other polymers. Poly Lactic Acid is growing interest because of its biomedical and many uses in pharmacological applications. Lactic acid is the building block of Poly Lactic Acid (PLA), which would ultimately be the input for producing biodegradable plastic. The USDA survey indicates that Lactic acid can be produced by utilizing agricultural products like corn starch and fibrous crops residues like the residue from sugarcane industry. In this study we will be discussing about the physical and mechanical properties of Poly Lactic Acid (PLA) and future prospects of PLA by mixing PLA with some organocclays as Closite C15A, Closite C25A and Closite C93A.

Keywords : PLA, aliphatic polyesters, organocclays, biodegradability, biocompatibility

I. INTRODUCTION

Worldwide production of high volume consumer plastics continues to be dominated by petroleum based polymers. Two factors have made biodegradable polymers economically attractive. These are environmental and economic concerns associated with waste disposal and rising costs of petroleum production resulting from the depletion of the most easily accessible reserves. Ease of degradability, versatility and anticipated price or performance of the new generation of polymers which is PLA that will enable it to displace a significant volume of fossil fuel based polymer.

Unlike other biodegradable polymers the degradation of PLA is not biodegradation as it is caused by hydrolytic cleavage of the polymer backbone with little or no evidence of participation of enzymes. Lactide monomers are synthesized by conventional process based on the cyclic compound synthesis through depolymerization of the corresponding oligomers which are synthesized by polycondensation. This lactide is then used for production of high molecular weight PLA through ring – opening polymerization. Poly Glycolic Acid (PGA) was synthesized by the synthesis of high molecular weight Poly Lactic acid (PLA) and copolymers of PLA and PGA. These α -polyesters have been investigated for use as structures and as implant materials for repairing a variety of issues. With increasing applications of PLA, considerable attention has been paid on its mechanical properties.

II. METHODOLOGY

2.1. SYNTHESIS OF PLA

Synthesis of a polymer begins with its monomer. Monomer of high molecular weight PLA, the lactide, is made from the lactic acid. An important feature of lactic acid is that it exists in two optically active forms L and D isomer. Fermentation derived lactic acid consists of 99.5 % of the L isomer. Production of the cyclic lactide intermediate results in three potential forms. The D and L forms are optically active, the D-L (a mixture of the D and L forms) and Meso form is optically active. The ratio of these three forms is readily controlled in the polymerization conditions by judicious selection of the initiator and reaction temperature. Ring opening polymerization of the lactide results in a family of polymers containing different isomer ratios and in the range of molecular weights. Polymers with high L levels can be used to produce crystalline products whereas the higher D levels greater than 15 % result in an amorphous product.

The thermal, mechanical and biodegradation characteristics of lactic acid polymers are known to depend on the choice and distribution of stereoisomers within the polymer chains. High purity L and D lactides form stereo regular isotactic Poly L-Lactic Acid (PLLA) and poly (D-Lactide) PDLA respectively. These are semi crystalline polymers with a high melting point 180 degree centigrade and a glass transition temperature in the 55 to 60

degree centigrade range. The degree of crystallinity depends on many factors such as molecular weight, thermal and processing industry and the temperature and the time of annealing treatments. The Meso and D-L lactide on the other hand form Poly (DL-Lactide) (PLLA) which is amorphous. The mechanical properties and degradation kinetics of the semi crystalline PLLA are quite different from those of completely amorphous PDLA. In particular, PLLA is always preferred whenever higher mechanical strength and longer degradation time is required. After the formation of PLA oligomers from the lactic acid by removal of water, de-polymerization of oligomers takes place to give a thermodynamically favored lactide. This monomer is readily polymerized under vacuum.

III. PROCEDURE

3.1. SYNTHESIS OF PLA FROM LACTIC ACID

There are two major routes to produce PLA from lactic acid monomer. The first route is removal of water of condensation by the use of solvent under high vacuum and high temperature. This process is known as condensation polymerization. In the other process water is removed under mild conditions to give an intermediate dimer, the lactide which on ring polymerization gives high molecular weight PLA.

3.2. SYNTHESIS OF LOW MOLECULAR WEIGHT PLA BY CONDENSATION POLYMERIZATION

Polycondensation of lactic acid is usually performed in bulk by distillation of condensation water, with or without catalyst while vacuum and temperature are progressively increased. Although high molecular weight polyesters with good mechanical properties are not easy to obtain the properties of lactic acid oligomers which can further be used as intermediates in the synthesis of polyurethanes that can be controlled by the use of different catalysts and functionalization agents. Addition of acidic catalysts such as boric or sulfuric acid accelerates the esterification and transesterification processes but also catalyzes side reactions at temperatures above 120 degree centigrade.

3.3. SYNTHESIS OF HIGH MOLECULAR WEIGHT PLA BY RING OPENING POLYMERIZATION OF LACTIDE

Water is removed under mild conditions without solvent to produce a cyclic intermediate dimer, referred to as lactide. This monomer is readily purified by vacuum distillation accomplished by heating again without the need for solvent. By controlling the purity of the dimer, it is possible to produce a wide range of molecular weights.

IV. RESULTS AND DISCUSSIONS

DSC measurement was performed with a LARPM /BBS /EQUIP/01 DSC(DSC Q 20, TA Instruments, USA). About 10 mg of the dried, ground samples were placed in aluminium sample pan. The samples were heated from 25 to 200 degree centigrade at a heating rate of 10 degree centigrade per minute in a nitrogen atmosphere. The sample was kept at 200 degree centigrade for 1 minute for the elimination of the previous heat history and was subsequently cooled to 25 degree centigrade at 10 degree centigrade per minute. The sample was then heated again to 200 degree centigrade at 10 degree centigrade per minute. The glass transition temperature T_g of the nanocomposite was taken as the inflection point of the increment of specific heat capacity. The values of melting temperature was determined as the temperature at the maximum value of the endothermic peak of the heating scans. The transition enthalpy was determined from the areas under the melting peaks.

DIFFRACTION PEAKS, DOO-SPACINGS AND DOO1 SPACINGS OF 15A, 25A AND 93A AND THEIR NANOCOMPOSITE FOAMS WITH PLA

SAMPLES	DIFFRACTION PEAK (2θ, DEGREE)	DOO –SPACING 1	DOO SPCINGS 2	DEGREE OF SWELL
25A	4.45	19.46		
PLA/25A	3.02	29.25	9.79	33.46
93A	3.78	23.37		
PLA/93A	2.76	32.01	8.64	26.99
15A PLA/15A	2.78	31.78	3.46	10.89

THERMAL PROPERTIES OF PLA AND PLA/CLAY NANOCOMPOSITE

SAMPLE	THERMAL PROPERTY FIRST HEATING Tg (Degree Centigrade)	SECOND HEATING Tm (Degree Centigrade)	Tm (Degree Centigrade)
PLA	63.4	171.5	165.0
PLA/25A	60.8	171.6	161.4
PLA/93A	63.8	157.4	163.9
PLA/15A	62.4	169.1	163.9

PHYSICAL AND MECHANICAL PROPERTIES OF PLA AND PLA/ CLAY NANOCOMPOSITE

SAMPLES	PHYSICAL PROPERTY			MECHANICAL PROPERTY	
	Radial expansion ratio	Unit density (Kg/m3)	Bulk Spring Index	Bulk Compressibility (Mpa)	Youngs Modulus (Kpa)
PLA	26.8 + 0.870 b	43.4 + 4.60 b	0.955 + 0.009 ab	8.78 + 298 c	135.2 + 18.2 b
PLA/25A	15.2 + 0.651 e	53.4 + 2.60 a	0.960 + 0.004 a	12.8 + 1.03 b	154.2 + 13.7 b
PLA/93A	46.4 + 1.80 a	23.0 + 0.80 c	0.944 + 0.015 b	0.989 + 0.025 d	49.97 + 5.32 c
PLA/15A	17.4 + 0.584 d	57.6 + 5.30 a	0.955 + 0.005 ab	8.05 + 0.426 c	143.2 + 6.73 b

V. CONCLUSIONS

A discussion on synthesis of PLA by ring opening polymerization is done. A brief description of the methodology used is also given. Investigation on the influence of types of nanoclay on the intercalation and properties of PLA nanocomposite is done. The nanoclay addition showed great improvement in structural, thermal, morphological and mechanical properties. The degree of enlargement of the doo1 with organoclay 25 a (9.79 AA⁰), Organoclay 93 A (8.64 A⁰) and oragnoclay 15A (3.46 A⁰), suggesting a decrease in interaction between PLA and these organoclays.

VI. REFERENCES

- [1] Thermal and bio- disintegration properties of PLA or natural rubber or organoclay nanocomposites, Natacha Bitinis, volume 93-94, May 2014
- [2] Tensile , watervapor barrier and antimicrobial properties of PLA/ nanoclay composite films J.W.Rhim et al. Volume 42, Issue 2, March 2009
- [3] Effect of an organo- modified montmorillonite on PLA crystallization and gas barrier properties, Emilie Picard, Eliane Espuche , Volume 53, Issue 1, July 2011
- [4] General crystallization behavior of PLA, B.Kalb, A.J. Pennings Volume 21, Issue 6, June 1980.
- [5] Gas permeation properties of PLA, Hans J. Lehermeier, J. Douglas Way Volume 190, Issue 2 , 15 September 2001
- [6] Thermal and mechanical properties of PLA based multiscale cellulosic biocomposites, Volume 18, May – June 2022
- [7] Natural fibres as reinforcement in PLA composites , K . Oksman , M. Skrifvars, J. F Selin, compus sci Technol, 63 (2003),pp. 1317-1324
- [8] Thermal and bio- disintegration properties of PLA or natural rubber or organoclay nanocomposites, Natacha Bitinis, volume 93-94, May 2014

- [9] Tensile , watervapor barrier and antimicrobial properties of PLA/ nanoclay composite films J.W.Rhim et al. Volume 42, Issue 2, March 2009
- [10] Effect of an organo- modified montmorillonite on PLA crystallization and gas barrier properties, Emilie Picard, Eliane Espuche , Volume 53, Issue 1, July 2011
- [11] General crystallization behavior of PLA, B.Kalb, A.J. PenningsVolume 21, Issue 6, June 1980.
- [12] Gas permeation properties of PLA, Hans J. Lehermeier, J. Douglas Way Volume 190, Issue 2 , 15 September 2001
- [13] Thermal and mechanical properties of PLA based multiscale cellulosic biocomposites, Volume 18, May – June 2022
- [14] Natural fibres as reinforcement in PLA composites , K . Oksman , M. Skrifvars, J. F Selin, compus sci Technol, 63 (2003),pp. 1317-1324