

POLYMERIZATION ON THE LACTIDE TO PLA THE EFFECT OF LACTIDE CONCENTRATION

Mohd Aizat Bin Zamri, DR Norliza Ibrahim

Faculty of Chemical Engineering, Universiti Teknologi Mara

Abstract—Polylactide was synthesized via ring –opening polymerization of lactide. The ring opening polymerization of lactide usually used the homogenous catalyst for the process polymerization of the lactide. But, for the polymerization process used the homogenous catalyst need the complex separation process in liquid-liquid component. Then, the heterogeneous catalyst need to use so the process of separation of PLA more easy. The tin octoate was used as the catalyst for this experiment. The aim of this experiment to study the polymerization of lactide with the heterogeneous catalyst. The properties of PLA were studied as a function of the concentration of lactide (0.5M, 0.3 and 0.1M) relative to the synthesis of PLA. The PLA were characterize by uv-visible spectroscopy (UV-vis) and Fourier transform infrared spectroscopy (FT-IR). The UV-vis showed the wavelength 233 nm of the PLA in the solution with different absorbance. FT-IR spectra showed the different between PLA and the lactide.

Keywords—lactide, polylactide, tin octoate, UV-visible spectroscopy, Fourier transform infrared spectroscopy.

I. INTRODUCTION

Plastic materials are widely used for various kinds of industries based on their high specific strength and light weight. The plastic also have two type, biodegradable and non-biodegradability. However, general plastic materials have been process from petrochemical resources, which can give the effect to environmental because non biodegradability. Recently, the biodegradable plastic widely used to

reduce the environment problem. Polylactides are nontoxic, biodegradable and also renewable polymer use in biomedical, pharmaceutical and packaging application(Xiao, Wang, Yang, & Gauthier, 2012). Polylactide. Polylactide (PLA) is a commercial biopolymer that from lactide acid as monomer. The monomer is obtained from vegetable raw material. It also nontoxic material and environmentally safe. There are many application of PLA such as in the field of drug delivery system, sutures, orthopaedic implants, tissue engineering and packaging material(H. Tsuji and Y. Ikada, 1998).

Then, for full manufacturing process of PLA a few unit of processes are used which, fermentation, separation, lactide conversion and lastly polymerization. Fermentation is a biological process by converted the sugar such as glucose, fructose and sucrose into lactic acid. From the lactic acid the process condensation occur to remove water and the product is low molecular weight prepolymer. The next process depolymerization to converted low molecular weight prepolymer to lactide and the last process is ring opening polymerization for manufacturing of PLA. The figure below show the process for production of PLA.

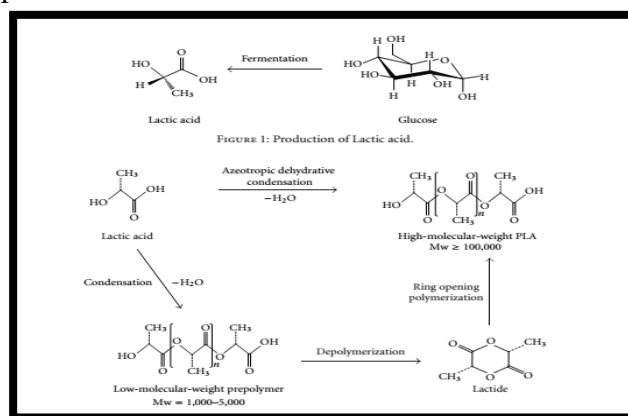


Figure 1. Production routes of lactide and PLA

Methods to produce PLA: Direct polycondensation and ring opening polymerization (ROP). For direct polycondensation there are two type of method which is solution polycondensation and melt polycondensation. Meanwhile the Lactide monomer has group of $-OH$ and $-COOH$, required for polymerization, the reaction can occur by self-codensation. Polylactide(PLA) is usually produced from reaction in the ring-opening polymerization (ROP), this process significant and effective method to manufactured high molecular weight PLA. This process involves strict purity of the LA monomer, the lactide achieved by the dimerization process of the lactic acid monomer. The polylactide polymerization used the catalyst with the lactide monomer under inert atmosphere. The monitoring at optimum temperature and residence time with the type and optimum concentration of catalyst, this parameter can control the ratio and sequence of lactide unit in polymerization(Lunt, 1998). The mechanism involved in the polymerization can be ionic, coordination,, or free-radical, depending on the type of catalyst used(Penczek, 2000).

In the ring-opening polymerization there are a few parameter that effect the molecular weight of the PLA. The parameter are the effect of temperature, effect of solvent used, effect of catalyst usage, and the effect of concentration of lactide in polymerization of PLA. In this research focus on the effect of the concentration of lactide for producing high molecular weight of PLA. In the polymerization of the lactide to PLA, the number average molecular weight of PLA rise proportional to the monomer conversion(Hu, Zhang, Ma, Han, & Song, 2018). When increasing the concenctration of lactide, the concentration of the PLA also increase.

II. METHODOLOGY

A. Materials polylactide(PLA) was suppliedby Nature works 4060D,(USA), Tin Octoate ($Sn(Oct)_2$) and lauryl alcohol $C_{12}H_{26}O$, all aqueous solutions were preparedusing double distilled water.

B. *Preparation of thin layer tin octoate* ,The solution of ethylene glycol was mix with the tin octoate catalyst with the ratio 10:1. The solution of ethylene glycol and tin octoate was add with nitric

acid and stir and mixed for 30 minute. *After* 30 minute, the solution add with 10 ml of polyethylene glycol and stirred for 6 hours. Then after 6 hour of stirred, leave the solution ageing for overnight.

C. *Synthesis of PLA*, The powder of lactide was weighed and placed in a conical flask, the magnetic stirrer and thermometer also provide for the which was equipped with a magnetic stirrer and thermometer. Lactide was heated until it melted. Lactide was heated at temperature $90^{\circ}C$ until lactide melted in the distilled water.. The melting temperature (T_m) of lactide is $93^{\circ}C$. Powder lactide was then added at concentrations of 0.5M, 0.3M and 0.1M. The solution of lactide was added in the the bio- reactor. After a several minute the solution of lauryl alcohol added in the solution as initiator. In the bio-reactor ceramic coating with tin octoate layer as catalyst was setting at impeller in tne reactor. After the temperature the solution at $120^{\circ}C$ the sample was taked for 15 minute for four time, 30 minute 2 tme and 1 hour.

III. RESULT AND DISCUSSION

A. *The effects of concentration of lactide on the synthesis PLA.*

In this study, the analysis was done on the PLA polymer solution which prepared throuhgth the ring opening polymerization of lactide.The concentration of lactide also can effect the synthesis of the PLA in the polymerization. In polymerization of the lactide the optimum temperature that was obtaion is $120^{\circ}C$. The In the polymerization of the lactide to PLA, the number average molecular weight of PLA rise proportional to the monomer conversion(Hu et al., 2018).The FT-IR spectra analysis were done for obtain that the PLA exist in the solution after the polymerization process. IR spectra exhibited specific absorption peaks of both PLA and lactide at $3000-2939\text{ cm}^{-1}$ for $-CH$ stretching, $1759-1735\text{ cm}^{-1}$ for $-C=O$ stretching, 1458 cm^{-1} for $-CH_3$ bending, and $1198-1185\text{ cm}^{-1}$ for $-C-O-C-$ vibration. However, as assume, absorption peak at 936 cm^{-1} for $-CO-O-$ ring of lactide could not appear in IR spectrum of PLA. This peak is characteristic for lactide monomer and has been used to differentiate between PLA and lactide(Chaiyut & Ksapabutr, 2010). For all sample 0.5M, 0.3M and 0.1M the specific absorption peak

range for the PLA solution for $-CH$ stretching is between $2978-2940\text{ cm}^{-1}$. $1745-1740\text{ cm}^{-1}$ for $-C=O$ stretching. 1460 for $-CH_3$ bending and for the $-C-O-C-$ vibration 1182 cm^{-1} .

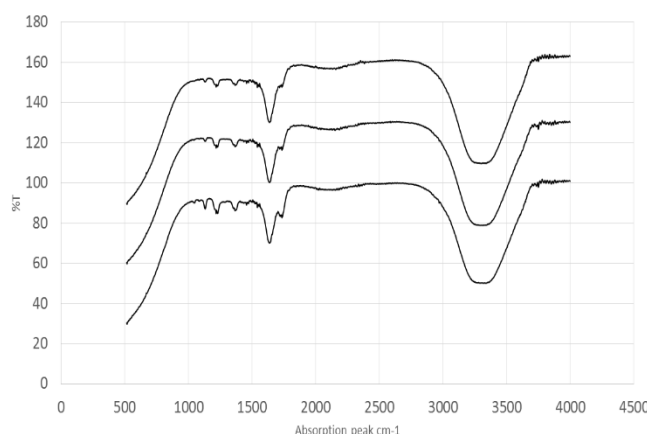


Figure 2 FT-IR spectra analysis of PLA solution

Concentration of PLA from polymerization of lactide was determined using the dilution PLA solid calibration curve that was be diluted. The calibration curve was obtain at 233 nm . The range of the wavelength for the PLA between ($315-280\text{ nm}$). The figure 3 show the graft calibration curve. In this analysis, the sample was choose from the 2 hour sample that produce the high of value of absorbance that come from the uv-vis analysis. The sample have different concentration which 0.5M , 0.3M and 0.1M . From the figure 4, the wavelength that obtain high of value of absorbance was 233 nm . For the sample of 0.5M that show in the figure 3 the absorbance value 3.290 that value is higher value compare to the sample 0.3M and 0.1M . Then, for the sample 0.3 M the absorbance value was obtain 2.856 , that value is lower than sample 0.5 M . For the sample 0.1 M the value of absorbance cannot obtain at the wavelength 233 nm . From this result, that can conclude the higher concentration of the sample from the polymerization process can produce the higher value of absorbance. The concentration of lactide also can effect the molecular weight of the PLA in the polymerization. In the polymerization of the lactide to PLA, the number average molecular weight of PLA rise proportional to the monomer conversion(Hu et al., 2018)

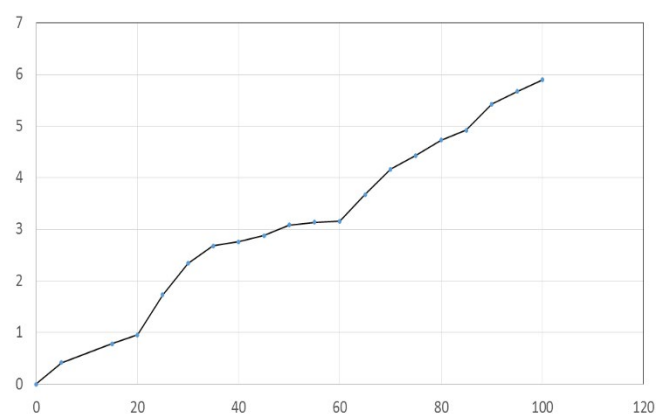


Figure 3 Dilution PLA solid calibration curve

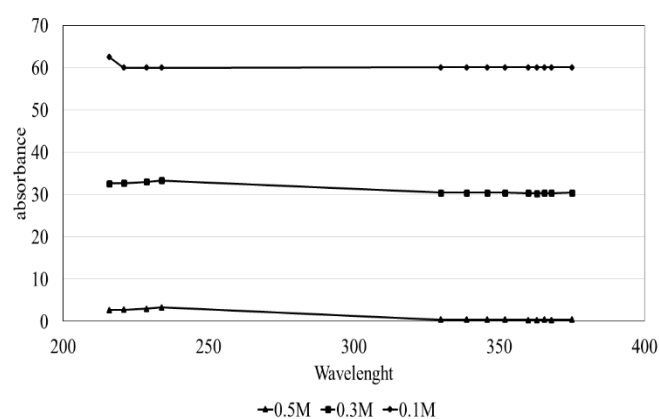


Figure 4 UV-visible spectroscopy of PLA solution

IV. CONCLUSION

The experiment was don successfully, from the experiment that can conclude the polymerization of lactide by using the heterogeneous catalyst that can also produce PLA.

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