

# Synthesis, Characterization and Determination of Weight Poly Molecule (Lactic acid)

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**Abstract:** Experiments have been successfully carried out to react with lactic acid and 1,4-butanediol, tin (II) chloride dihydrate ( $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ ), chloroform, methanol, liquid nitrogen, nitrogen gas, and silicone oil at various concentrations. The objective exists to synthesize polylactic acid by forming polylactic acid diol (PLA-OH) via a direct polycondensation reaction of lactic acid and 1,4-butanediol in a glass reactor following a specific reaction scheme. Synthesis was carried out using different amounts of reagents according to the predetermined mole ratio of lactic acid (AL) and 1,4-butanediol (BD). The FTIR,  $^1\text{H}$  NMR, and GPC analyses that characterized the PLA-OH revealed its physicochemical characteristics. The FTIR and  $^1\text{H}$  NMR characterization results show new absorption peaks and a shift in PLA-OH absorption peaks. It indicates that a bond has been formed from the reaction between lactic acid molecules and 1,4-butanediol to produce PLA-OH. From the spectrum analysis, it can be concluded that the structure of the synthesized PLA-OH has four different proton environments (there are four different peaks in the spectrum). The peaks originate from the protons in methylene ( $-\text{OCH}_2\text{CH}_2\text{CH}_2\text{CH}_2\text{O}$ ), internal methine ( $-\text{O}-\text{CH}-$ ), and methine at the end of the PLA-OH chain, as well as proton peaks in methyl ( $\text{H}_3\text{C}-$ ). The characterization results with GPC showed that the tendency to increase  $M_n$  PLA was directly proportional to the increasing amount of lactic acid in the PLA chain.

**Keywords:** Biodegradable polymer; Molecular weight; POLY (lactic acid); Polycondensation

## Introduction

Finding alternatives to lessen plastic pollution has emerged as one of the biggest problems in contemporary society. A few alternatives have the potential to drive plastic use in a more circular and sustainable direction. Among these alternatives is the use of biodegradable polymers (Bher et al., 2022)

Efforts to develop polymers made from aliphatic polyesters are continuing. This is because aliphatic polymers are biodegradable, so they have the potential to be developed as biomedical materials or in other fields that require environmentally friendly materials. (Ábrahám et al., 2022; Kojima et al., 2021; Lee et al., 2021; Šerá et al., 2022). These aliphatic polyesters have a number of methylene groups between their ester bonds which can be degraded chemically at a certain time by hydrolytic cleavage of the ester bonds or through certain

enzymatic reactions (Campana et al., 2022; Djouonkep et al., 2022; Gkountela & Vouyiouka, 2022)

One type of aliphatic polyester is poly (lactic acid) (PLA) (T. Zhang et al., 2022). Its good solubility in organic solvents causes PLA to continue to be developed for its use, especially in the medical field (Damadzadeh et al., 2010). In addition, increasing environmental pollution and the high price of oil-based polymer materials have caused PLA to be considered to replace oil-based polymer materials (Chaisit et al., 2022)

PLA can be synthesized via lactic acid polycondensation or cyclic lactate ring-opening polymerization reactions (Rahaman et al., 2022; T. Zhang et al., 2022). The second method is not preferred because of its low efficiency, because it takes a long route through lactate ring-opening polymerization and requires pure lactic acid. This method is expensive and makes the PLA less competitive (Mekpothi et al., 2021; Stefaniak & Masek, 2021)

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Researchers have conducted several studies to synthesize PLA through lactic acid polycondensation reactions. For example, Tsai et al. (2008) reported the research results with a review of the effect of the differences in the two-mole reagents (ethylene glycol and 1,3-propanediol) used to react with lactic acid on the mechanical properties of the resulting PLA.

The research synthesized PLA from 1,4-butanediol and lactic acid reagents for optimal use. The choice of 1,4-butanediol is based on the theory that the composition of the diol in a PLA will significantly affect the structure and properties of the poly (urethane ester) (Hiltunen et al., 1998; Pretsch et al., 2009; Saad et al., 2002). Huskić & Žigon (2004) researched the effect of the length of the constituent chains of polyester on the thermal properties of polyester using various chain lengths (-CH<sub>2</sub>-) that bind themselves at both ends. FTIR, <sup>1</sup>H NMR, and GPC then characterized the successfully synthesized PLA. Through this research, it is hoped that PLA can be produced and its physicochemical properties can be controlled by controlling the amount of lactic acid and 1,4-butanediol used.

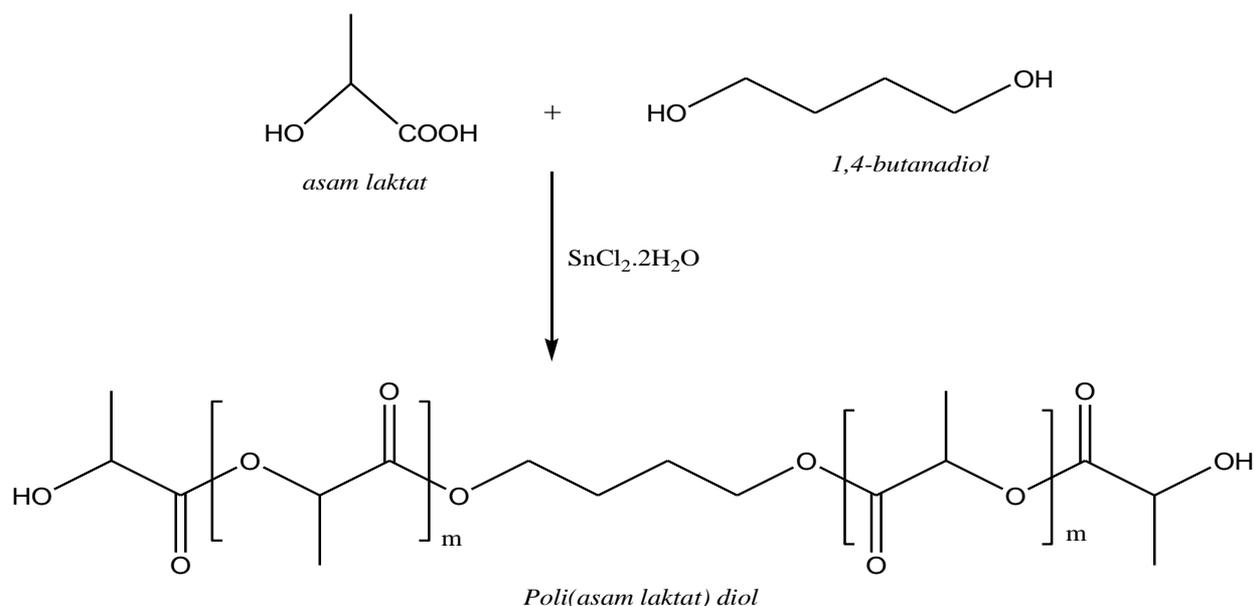
## Method

### *The Materials and Instruments Used*

The materials used are lactic acid, 1,4-butanediol, tin (II) chloride dihydrate (SnCl<sub>2</sub>·2H<sub>2</sub>O), chloroform, methanol, liquid nitrogen, nitrogen gas, and silicone oil. The tools used are vacuum pumps, vacuum ovens, mechanical stirrers, hot plates, evaporators, viscometers, electric shakers, and glassware commonly used in chemical laboratories.

### *Synthesis of PLA*

PLA was synthesized by forming dihydroxyl-terminated PLA (PLA-OH, poly(lactic acid) diol) from the direct polycondensation reaction of lactic acid and 1,4-butanediol using SnCl<sub>2</sub>·2H<sub>2</sub>O as a catalyst in a glass reactor immersed in heated silicone oil (Little et al., 2021; T. Zhang et al., 2022) according to the reaction scheme shown in Figure 1 (Zeng et al., 2009). Synthesis was carried out using different amounts of reagents according to the predetermined mole ratios of lactic acid (AL) and 1,4-butanediol (BD) (Table 1).



**Figure 1.** Schematic of the synthesis reaction of poly (lactic acid) diol

**Table 1.** PLA-OH synthesis data

PLA-OH	AL/BD (mole/mole)	Lactic acid		1,4-butanadiol		SnCl <sub>2</sub> ·2H <sub>2</sub> O (grams)
		mole	mL	mole	mL	
PLA-OH (1)	25 : 1	1	83	0.04	3.55	0.54
PLA-OH (2)	50 : 1	1	83	0.02	1.77	0.54
PLA-OH (3)	75 : 1	1	83	0.0133	1.18	0.53
PLA-OH (4)	100 : 1	1	83	0.01	0.88	0.53

After 12 hours of reaction at a temperature of 160°C to 180°C and a pressure of 0.004 atm, a product is obtained, which is then purified by dissolving it in chloroform and precipitating it in excess methanol. The

remaining chloroform and methanol are removed by evaporation. The white precipitate was dried in a vacuum oven to form a powder.

*Characterization*

The characterization of the successfully synthesized PLA-OH compounds was carried out at the Indonesian University of Education (UPI) in Bandung (FTIR, or Fourier Transform Infrared), the Indonesian Institute of Sciences (LIPI) in Tangerang, the Polymer Technology Center (STP) in Serpong (<sup>1</sup>H NMR, or proton nuclear magnetic resonance), and at Newcastle University in New South Wales, Australia (GPC, or gel permeation chromatography).

**Result and Discussion**

*Results of Characterization with FTIR*

The FTIR spectrum of lactic acid, 1,4-butanediol, and PLA-OH is shown in Figure 2. In the spectrum of lactic acid (Figure 2), it is observed that the absorption of the stretched C=O group from the carbonyl group in the 1730 cm<sup>-1</sup> area, the absorption of the stretched O-H group, is widened. In the 3392 cm<sup>-1</sup> area, the peaks from C-O bond absorption are in 1220 cm<sup>-1</sup>, 1128 cm<sup>-1</sup>, and 1045 cm<sup>-1</sup> areas, and the peaks in 2989 cm<sup>-1</sup> and 2943 cm<sup>-1</sup> areas come from the absorption of stretched C-H bonds.

The peak in the 3298 cm<sup>-1</sup> regions in the 1,4-butanediol spectrum (Figure 2) is produced by the stretched O-H group, while the absorption from the C-O bond produces peaks in 1265 cm<sup>-1</sup>, 1222 cm<sup>-1</sup>, and 1174 cm<sup>-1</sup> regions. The stretched C-H group gave absorption peaks in 2939 cm<sup>-1</sup> and 2871 cm<sup>-1</sup> regions. In the PLA-OH spectrum (Figure 2), the strong and sharp band in the 1759 cm<sup>-1</sup> region indicates the presence of a stretched C=O group from the carbonyl, and the absorption of the bending C=O group produces a peak in the 1213 cm<sup>-1</sup> region.

**Table 2.** Comparison of PLA-OH absorption peaks

functional group	Wavenumber (cm <sup>-1</sup> )	
	PLA-OH	PLA-OH (Garlotta, 2001)
C=O stretch	1759	1759
C=O bend	1213	1268
O-H stretch	3423	3571
O-H bend	1045	1047
C-H stretched	2957; 2947	2995; 2944
C-H bend	1384; 1361	1382; 1362
CH <sub>3</sub>	1458	1453
C-O stretched	1190; 1134; 1095	1194; 1130; 1093
C-C stretch	921; 871	926; 868

When comparing the absorption in the spectrum in Table 2 with the absorption peak data in Figure 2, it can be seen that several new absorption peaks have formed and that some of the absorption peaks in PLA-OH have shifted. This indicates that a bond has been created from

the reaction between lactic acid molecules and 1,4-butanediol to produce PLA-OH.

The absorption of O-H stretching and O-H bending was observed at 3423 cm<sup>-1</sup> and 1045 cm<sup>-1</sup>, respectively. Absorption from stretched C-H bonds appears in the areas 2997 cm<sup>-1</sup> and 2947 cm<sup>-1</sup> while bending C-H bonds gives absorption at peaks in the areas 1384 cm<sup>-1</sup> and 1361 cm<sup>-1</sup>. The bent CH<sub>3</sub> group provides absorption in the 1458 cm<sup>-1</sup> region, and absorption peaks of the -C-O- stretched bond is observed in 1190 cm<sup>-1</sup>, 1134 cm<sup>-1</sup>, and 1095 cm<sup>-1</sup> regions. The -C-C- bond is stretched to produce peaks observed in the 921 cm<sup>-1</sup> and 871 cm<sup>-1</sup> regions. The absorptions observed in the PLA-OH spectrum have values similar to those of previously synthesized PLA-OH (Garlotta, 2001), as shown in Table 2.

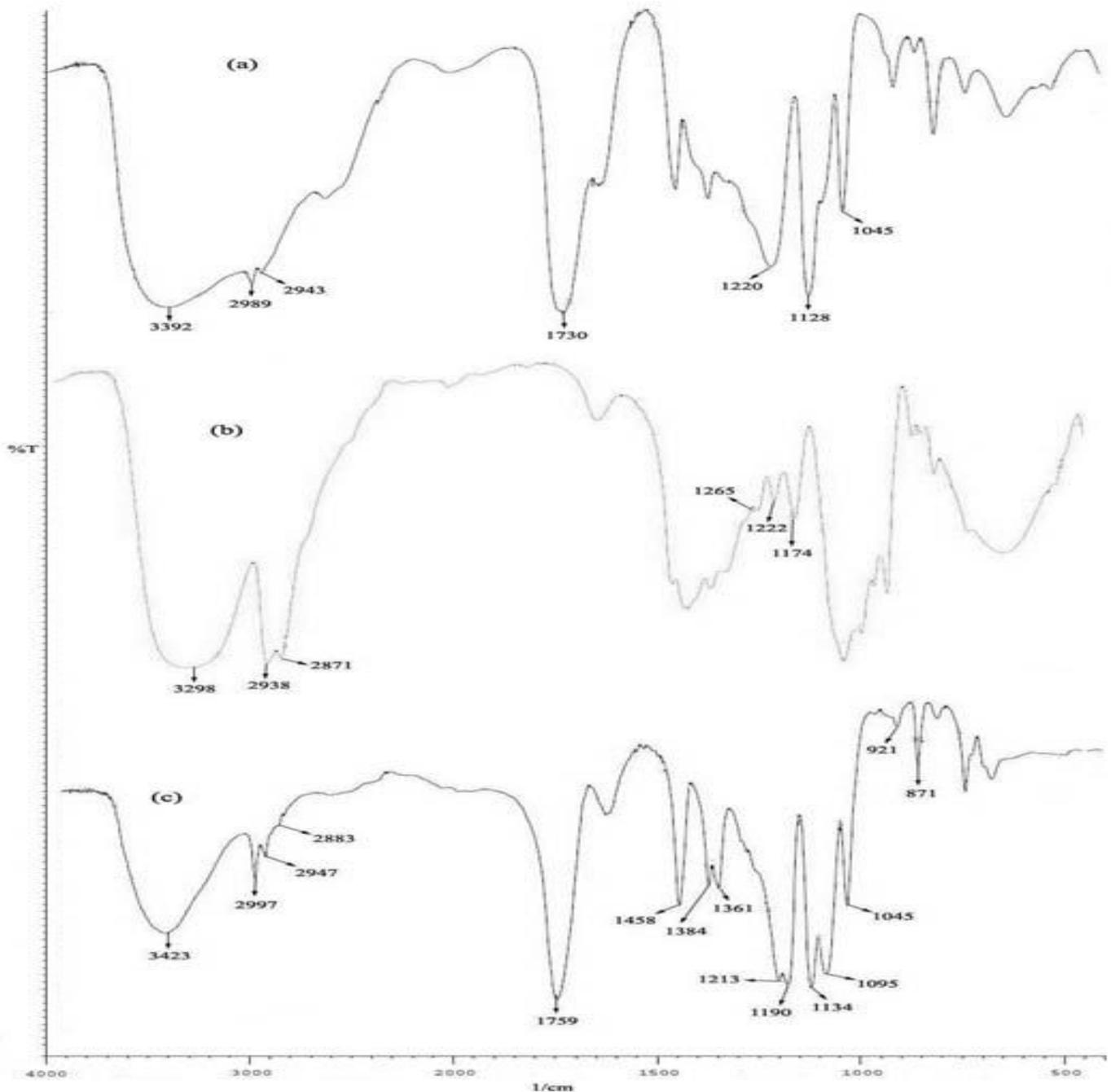
The new peaks formed are 1213 cm<sup>-1</sup> (C=O bend, ester) and 1045 cm<sup>-1</sup> (O-H bend, alcohol). These peaks were not previously found in lactic acid uptake or 1,4-butanediol. Meanwhile, a shift in the absorption peak occurred; for example, the typical lactic acid peak in the region of 1730 cm<sup>-1</sup> shifted to 1759 cm<sup>-1</sup> in the PLA-OH spectrum. This shift is mainly caused by differences in bond strength between the monomer and polymer for the same functional group, which causes differences in absorption frequencies.

For example, the C=O (ester) bond in the PLA-OH molecule is stronger than the C=O (carboxylic) bond in lactic acid due to the influence of the chemical environment, namely differences in other atoms/groups that bind to the C=O. This means that the bond energy is also greater, which causes the absorption to be at a higher wave number in accordance with the concept of the energy equation for light (Creswell, 1982; Sastrohamidjojo, 1985)  $E = hv = h\frac{c}{\lambda} = hc\bar{\nu}$ .

The difference in bond strength also has implications for the force constant (K) for the bond. The stronger the bond, the greater the value of K, which is directly proportional to the magnitude of the absorption frequency, according to Hooke's Law equation (Creswell, 1982).

$$\nu = \frac{1}{2\pi} \sqrt{\frac{K}{(m_1 m_2) / (m_1 + m_2)}} \tag{1}$$

The shift in the absorption region in the FTIR spectrum of the polymer and its constituent monomers was also observed by Ramesh et al. (2007) when synthesizing the PVC-PMMA polymer from PVC and PMMA monomers.



**Figure 2.** FTIR spectrum (lactic acid); (b) 1,4-butanediol; (c) PLA-OH

*Result of characterization with <sup>1</sup>H NMR*

The <sup>1</sup>H NMR spectrum of lactic acid, 1,4-butanediol, and PLA-OH are shown in Figures 3-5. The three figures show that the peaks in lactic acid and 1,4-butanediol as a constituent of PLA-OH are still visible in the PLA-OH spectrum and experience a slight shift. Such shifts are for example, at the peak of lactic acid at 5.1741 ppm, 4.3640 ppm, and 1.5837 ppm, which appears

in the PLA-OH spectrum at 5.1618 ppm, 4.3563 ppm, and 1.5822 ppm. Meanwhile, the peak of 1,4-butanediol is in the area of 3.6471 ppm; 3.6364 ppm; 3.6257 ppm; and 1.5685 ppm appears in the PLA-OH spectrum in the 3.7664 regions; 3.7404 ppm; 3.7220 ppm; and 1.5669 ppm. This shift indicates that the condensation polymerization reaction between lactic acid and 1,4-butanediol has occurred.

From the analysis of the FTIR and <sup>1</sup>H NMR spectrum above, it can be concluded that the PLA-OH structure synthesized is as shown in Figure 6. The PLA-OH structure has four proton environments (a, b, c, and d). These four proton neighborhoods give four distinct peaks in their NMR spectrum (Figure 5). The peaks that occur at 1.59 ppm (δH<sup>d</sup>) and 4.34 ppm (δH<sup>c</sup>) in the spectrum belong to the protons of methylene (-

OCH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>CH<sub>2</sub>O-). Proton peaks in methine (-O-CH-) were observed at the 5.15 ppm (δH<sup>a</sup>) region, which is internal methine, and at 4.36 ppm (δH<sup>a'</sup>) which indicates methine at the end of the PLA-OH chain. while the proton peaks in methyl (H<sub>3</sub>C-) appear in the region of 1.58 ppm (δH<sup>b</sup>) and 1.55 ppm (δH<sup>b'</sup>)

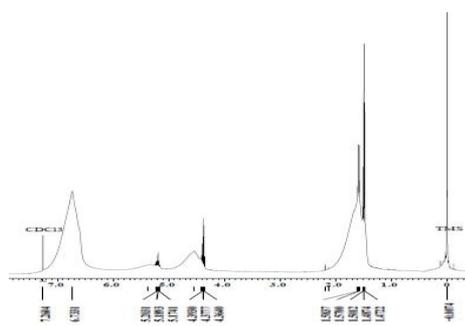


Figure 3. Lactic acid <sup>1</sup>H NMR spectrum

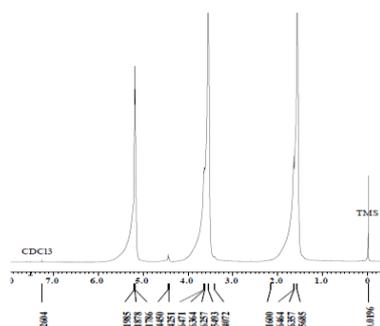


Figure 4. <sup>1</sup>H NMR spectrum of 1,4-butanediol

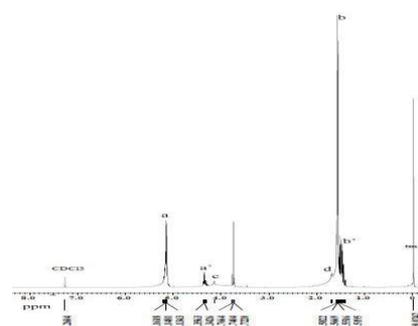


Figure 5. Proton environment in the <sup>1</sup>H NMR spectrum of PLA-OH

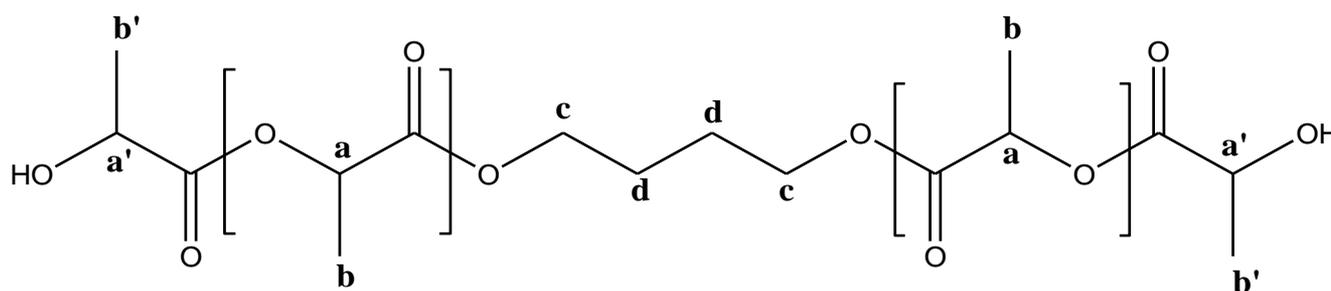


Figure 6. Structure of synthesized PLA-OH

The  $M_n$  determination of the synthesized PLA-OH was carried out based on the proton intensity in the NMR spectrum above. This intensity is used to calculate the degree of polymerization (DP) of PLA-OH using the equation  $DP_{PLA-OH} = 2 \times \frac{I_{m+I_n}}{I_n}$  (Zeng et al., 2009). Furthermore, the average molecular weight ( $M_n$ ) of

PLA-OH was calculated using the DP values obtained  $M_n(PLA-OH) = (DP_{PLA-OH} \times 72) + 88 + 2$ . The complete average molecular weight ( $M_n$ ) of PLA-OH is shown in Table 3 which is equipped with data for calculating molecular weight using GPC.

Table 3. Average molecular weight ( $M_n$ ) of PLA-OH

PLA-OH	$M_n, NMR$ (g/mole)	$M_n, GPC$ (g/mole)	$M_w, GPC$ (g/mole)	$M_w / M_n$	$M_n, Theory$ (g/mole)
PLA-OH (1)	1460	3537	4267	1,21	1890
PLA-OH (2)	2011	4737	6598	1,39	3690
PLA-OH (3)	3034	5362	7524	1,40	5490
PLA-OH (4)	3952	7061	7457	1,06	7290

The value of  $M_{n,NMR}$  is always smaller than the value of  $M_{n,theory}$  in all comparisons of lactic acid and 1,4-butanediol. This is caused by the loss of lactic acid which cannot be avoided during the polycondensation reaction process (Maryanty et al., 2021; L. Zhang et al., 2021).

### Conclusion

PLA has been successfully synthesized by forming poly (lactic acid) diol (PLA-OH) from the polycondensation reaction of lactic acid and 1,4-butanediol. The physicochemical properties of PLA have been characterized by FTIR, <sup>1</sup>H NMR, and GPC, which

include determining the structure and molecular weight. The characterization results with <sup>1</sup>H NMR and GPC showed that the tendency to increase M<sub>n</sub> PLA was directly proportional to the greater amount of lactic acid in the PLA chain.

#### Author Contribution

Saefuddin: Conceptualization, design experiment, perform experiment, collected data and analysis data. Peter Davey: Validation.

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#### Conflicts of Interest

The authors declare no conflict of interest.

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