

# Preparation and Characterization of Polylactic Acid by Ring Opening Polymerization Using Unconventional Heating System

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## ABSTRACT

Polylactic acid (PLA) has been synthesized via ring opening polymerization (ROP) for the Lactide cyclic dimmer under microwave irradiation with a constant power of 300W using ZnO catalyst. The structural formula of PLA was established using spectroscopic techniques FTIR, <sup>1</sup>HNMR, and <sup>13</sup>CNMR. The thermal properties were investigated using DSC and TGA techniques. The DSC thermogram exhibited two endothermic transitions at (115-132) C° which related to the melting of the polymer, while the TGA thermogram showed one main decomposition step at 265°C. The viscosity average molecular weight [M<sub>V</sub>] of the obtained polymer was (7.8×10<sup>3</sup> gm/mol). The SEM morphology study exhibited homogenous and dense surface with no separate entities

**KEYWORDS:** polylactic acid; microwave techniques; biodegradable polymers; green chemistry.

## الخلاصة

تم تحضير بوليمر البولي لالاكتيك بواسطة البلمرة بفتح الحلقة للمركب الحلقي ( اللالاكتايد) باستخدام اشعاع المايكرويف (300) واط وبوجود Zn كعامل مساعد. تم اثبات الصيغة التركيبية للمركب باستخدام تقنيات FTIR و<sup>13</sup>CNMR و<sup>1</sup>HNMR فحوصات الثباتية الحرارية تمت باستخدام تقنيتي الـ DSC و TGA تم حساب المعدل اللزجي للوزن الجزيئي للبوليمر الناتج (7.8×10<sup>3</sup> gm/mol) وقد اظهرت صورة لسطح البوليمر تم التقاطها بواسطة المجهر الالكتروني وجود سطح البوليمر المتكون سطح متجانس وتركيب كثيف.

## INTRODUCTION

In recent decades, a huge global problem appears and attracted a remarkable attention, this problem is a "plastic waste" since it represents about 20%–30% of plastic production[1]. Scientists in chemical and environmental engineering spent significant efforts to deal with this problem. One of the attractive solutions is using the biodegradable polymers generated from renewable resources [2-3]. These polymers undergo chemical and biological transformations end with water, carbon dioxide and other natural compounds [4]. Polymers from these resources can significantly reduce the greenhouse gas emissions and fossil energy use. PLA is one of the most promising biodegradable polymers [5,6] It is a linear aliphatic with a valuable properties make it a very good alternative to the petroleum-based polymers [7,8]. PLA with a low-molecular

weight was firstly synthesized by Carothers, in 1932[9] However, the properties of the produced polymer was unfavorable. After two decades, DuPont produce a higher-molecular weight PLA patented in 1954[10]. PLA generally synthesized by two major routes: condensation polymerization[11,12]and ring opening polymerization of Lactic acid[13]. Synthesis of PLA by condensation polymerization required rigorous conditions such as, long polymerization time, high energy consumption duo to high temperature degrees needed, and high pressure[14,15]. Ring-opening polymerization (ROP) is the most common used approach to obtain high molecular weight PLA[16,17]. This controlled process deals with the cyclic dimmers and could lead to produce a polymer with specific and favorable properties like high molecular weight and reflective index[18]. ROP

reaction involving polycondensation of lactic acid monomers to low-molecular weight PLA, depolymerization of the oligomeric PLA into the lactide, and catalytic ring-opening polymerization of the lactide intermediate resulting in a high molecular weight PLA[19,20]. Ring-opening polymerization of lactide can happen through either anionic, cationic, or coordination-insertion mechanisms[21]. The first two mechanisms have high reactivity, and usually suffer from transesterification and racemization during the reaction process leading to formation of a polymer with impurities[22,23]. ROP by Coordination-insertion mechanism has been extensively studied due to the high molecular weight polymer obtained[24]. A wide range of metals catalytic systems such as Zn[25], Sn[26], Al, Mg and Ca derivatives used in this mechanism to produce polymers with narrow polydispersity[27]. Microwave (MW) technique is a clean ecofriendly method for chemical synthesis due to its high efficiency and dramatic reduction in time compared with the classical routes which proceed for about 45-60 hours[28,29]. In addition, (MW) provide a homogeneous heating for the whole reaction mixture. This technique is a form of electromagnetic energy [30], considered to be a non-ionizing energy and have no effect on the molecular structure of the compounds but supply thermal activation only[31]. The first microwave-irradiated polymerization of D,L-Lactide (DLLA) using stannous octoate as a catalyst was reported in 2001 by Liu *et al* [32]. The reaction showed a remarkable advantage in reducing time, rising purity, molecular weight and yield. In 2006 Peng *et al*[33]. Perform polymerization of D, L-lactide under atmospheric pressure in the presence of Sn(Oct)<sub>2</sub>, the molecular weight of the obtained polymer was  $21.87 \times 10^4$  gm/mol. One year later, Gong *et al*[34]. synthesized a triblock copolymer of poly(ethylene glycol) and L-lactide using microwave-assisted ROP in the presence of tin (II) 2-ethylhexanoate as a catalyst. The using of microwave in the chemistry leads to a huge change in the methodology of chemical synthesis and perform the same reactions in only several minutes[35-36]. Microwave irradiation is considered to be a promising technique as its

cheap, safe, environmentally friendly and advanced step toward green chemistry[37,38].

In this study, PLA was obtained in reduced time using microwave oven in two stages, the first involves the formation of the cyclic dimer (lactide), and the second was a ROP of lactide in the presence of ZnO as a commercially available and highly reactive catalyst. No sealed tube was used in this approach, only traditional equipment for the synthesis of PLA in the conventional procedures.

## MATERIAL AND METHODOLOGY

### Chemicals

L-Lactic acid (85%) was purchased from Aldrich Chemicals Company; ZnO powder was purchased from Fluka while dichloro methane (CH<sub>2</sub>Cl<sub>2</sub>) and Methanol (CH<sub>3</sub>OH) were purchased from BDH. All chemicals used without any further purification.

### Instruments

A microwave oven from (MAS-II sineo) with a working frequency of 2.45 GHz supplied with a LCD color television with digital camera and power of 12V DC. FTIR characterization was collected using SHIMADZU FTIR3800S with KBr technique from 4000-400cm<sup>-1</sup>. Nuclear Magnetic Resonance (NMR) for both proton <sup>1</sup>H NMR and carbon, <sup>13</sup>C NMR were recorded on a Bruker Avance II spectrometer operating at 400 MHz for <sup>1</sup>H NMR and 100 MHz <sup>13</sup>C NMR. Chemical shifts were recorded in ppm relative to (TMS) (0.00 ppm) in CDCl<sub>3</sub> as a solvent. Differential scanning calorimetry (DSC) measured using Q20 V24.4 instrument with heating rate of 10°C/min from room temperature to 300°C under air. Thermal gravimetric analysis (TGA) was carried out using TGA 50SHIMADZU with heating rate of 10°C/min up to 600°C. Scanning Electron Microscope SEM was applied in the study using Tescan VEGA3 SB with accelerating Voltage 200V to 30kv.

### Synthesis of Polylactic Acid Using Microwave Irradiation

In a 250 ml round bottom flask, 125ml of (85%) lactic acid was added, the flask was placed in a microwave oven at (2.45) GHz connected to a vacuum line, the power was set at (300) W,

temperature at 80°C with stirring the reaction proceed for a period of time until a pale yellow viscose material was obtained. The round bottom flask left to cool down to room temperature. ZnO (1%) wt. was added to the flask and the reaction proceeds under constant power, the temperature quickly raised to 170°C, the viscosity of the material was increased and the color changed to light brown. The reaction was stopped and the round bottom flask left to cool under vacuum for an hour, the produced material dissolved in dichloro methane (CH<sub>2</sub>Cl<sub>2</sub>) and filtered to remove the ZnO, precipitated in 80ml of cold methanol then collected and dried at 60°C in a vacuum oven.

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### Determination of the viscosity average molecular weight ( $\overline{M}_v$ ) for the synthesized PLA

The determination of ( $\overline{M}_v$ ) for the produced PLA involving preparation of solutions with different concentrations of the polymer (0.4%, 0.6%, 0.8%, 1.0%) using pure chloroform 99% as a solvent, both the polymer solutions and the solvent transferred to Ostwald capillary viscometer which is placed in 25°C controlled temperature water bath .The flowing time of the solvent and the polymeric solutions was determined respectively using a simple

stopwatch, the values of relative, reduced, and specific viscosity were calculated, The intrinsic viscosity  $[\eta]$  was graphically determined from the relationship between the concentrations C(gm/mol) and the  $[\eta_{sp}/C]$ . Mark-Houwink equation used to convert Interinsic viscosity to the viscosity average molecular weight ( $\overline{M}_v$ ).

$$[\eta] = K[Mv]^a \quad (1)$$

Where K and a are constants[39]

$$K=5.45 \times 10^{-4}, a = 0.73 \text{ in } \text{CHCl}_3$$

## DISCUSSION

### Synthesis of PLA using microwave irradiation

Many experiments with different conditions of microwave power and temperatures were made in order to set up ideal circumstances for the synthesis of PLA. It was found that when power set at 300 w and the temperature gradually raised to 170°C the produced polymer was white crystal with high molecular weight and good thermal stability. Synthesis of PLA using microwave irradiation by ROP was carried out in two stages: The first was condensation reaction occurred by losing water molecule and produces a low molecular weight polymer (oligomer), which undergoes a back biting reaction end with a formation of the cyclic dimer (lactide)[40]. The second was ROP of the lactide in the presence of ZnO as a polylactic acid with high purity. As shown in Figure1.

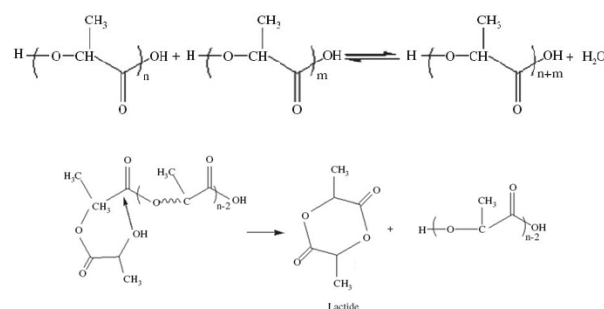


Figure 1. General scheme for ring opening polymerization.

Generally, in polycondensation reaction, the removal of water under vacuum is a basic condition since the esterification reaction is reversible. Nevertheless, the presence of water can act as a chain transfer agent leading to depolymerization reaction which afforded stereo-irregular and low molecular weight polymer[41]. As the temperature increased under

constant power and vacuum a cyclic dimer (Lactide) formed. This procedure involves two equilibria: dehydration and chain-ring equilibrium[42]. Zinc oxide considered being one of the highly effective metal oxides in ROP of the lactide, this is related to the active site in zinc oxide which appear to be wider and leading to formation of longer polymeric chains[43]. Furthermore, ZnO has approved by FDA to be used in medical applications such as drug delivery, tissue engineering due to biocompatibility and low toxicity [44-45].

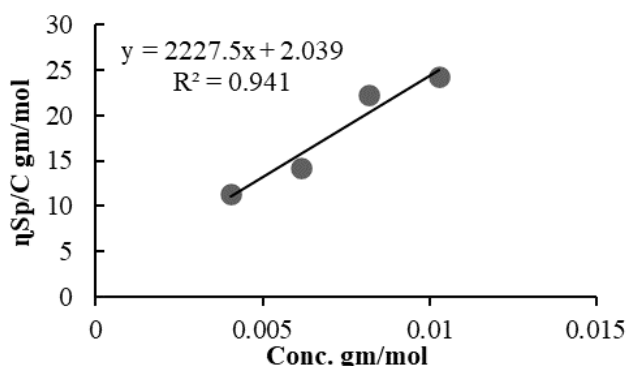
### Determination of the viscosity average molecular weight ( $\overline{M}_v$ ) for synthesized PLA

The values in Table 1 represent the relative, specific and reduced viscosity respectively; the intrinsic viscosity  $[\eta]$  was graphically determined by using the data of reduced, specific viscosity. The viscosity average molecular weight ( $\overline{M}_v$ ) of the obtained polymer was calculated using Mark-Houwink equation (Figure 2). The values of intrinsic viscosity and the viscosity average molecular weight ( $\overline{M}_v$ ) were 2.039,  $7.8 \times 10^3$  gm/mol respectively.

**Table1.** The values of relative, specific and reduced viscosity respectively.

| Conc. gm/mol | T    | $t_0$ | viscosities  |             |              |
|--------------|------|-------|--------------|-------------|--------------|
|              |      |       | $\eta^a$ Rel | $\eta^b$ Sp | $\eta^c$ Red |
| 0.00405      | 40.3 | 38.5  | 1.046        | 0.046       | 11.358       |
| 0.00618      | 41.9 | 38.5  | 1.088        | 0.088       | 14.239       |
| 0.00816      | 45.5 | 38.5  | 1.181        | 0.181       | 22.181       |
| 0.00103      | 48.1 | 38.5  | 1.249        | 0.249       | 24.174       |

$t^a$ =flow time of solvent,  $t^b$ =flow time of polymer, solutions  $\eta^a$  Rel= $t^b/t^a$ ,  $\eta^b$ Sp = Rel-1,  $\eta^c$ Red=Sp/C



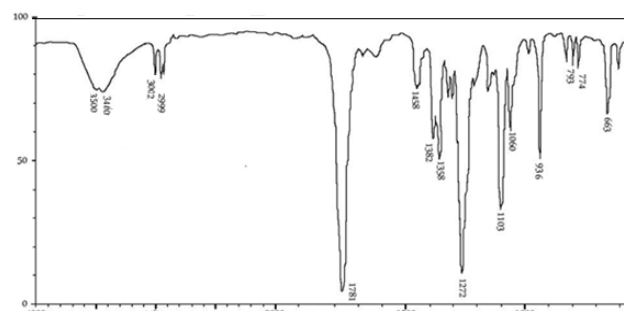
**Figure 2.** Mark-Howeink equation.

### FTIR Characterization

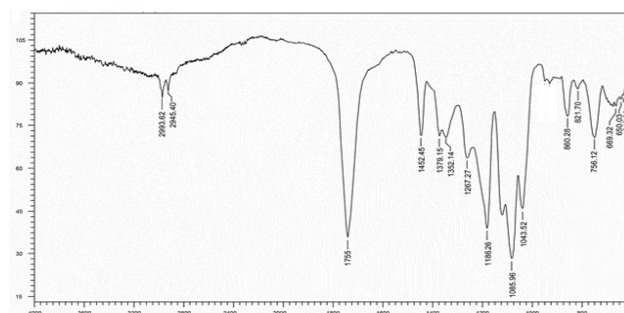
The spectrum of L-Lactide given in Figure 3 shows broad peaks at 3460-3500  $\text{cm}^{-1}$  related to hydroxyl group which is common in the spectrum of prepared L-lactide due to the residual fragment of the oligomer after

cyclization (back biting reaction)[46]. Stretching absorption at 2999  $\text{cm}^{-1}$  and 3002  $\text{cm}^{-1}$  is originated from symmetric and asymmetric valence vibrations of C-H from  $\text{CH}_3$ , respectively. Sharp peak at 1781  $\text{cm}^{-1}$  related to C=O stretching vibration of the lactide ring, peaks at 1458  $\text{cm}^{-1}$  and 1382  $\text{cm}^{-1}$  represent asymmetric and symmetric bending vibration of C-H from  $\text{CH}_3$ , respectively, and peak at 1272  $\text{cm}^{-1}$  represent asymmetric vibrations of C-O-C in the ring. A characteristic absorption peak appears at 936  $\text{cm}^{-1}$  indicate the formation of L-Lactide (ring breathing mode)[45].

The spectrum of PLLA given in Figure 4 revealed peaks at 2993  $\text{cm}^{-1}$  and at 2945  $\text{cm}^{-1}$  related to asymmetric and symmetric valence vibrations of C-H from  $\text{CH}_3$  in the aliphatic chain respectively. stretching vibration of C=O of the aliphatic ester is shifted to 1755  $\text{cm}^{-1}$  in the polymer, This difference in the peak shifting may be related to the arrangement of molecules in the polymer chain backbone [47]. Peaks at 1452  $\text{cm}^{-1}$  and 1379  $\text{cm}^{-1}$  represent asymmetric and symmetric bending vibration of C-H from  $\text{CH}_3$ , respectively.



**Figure 3.** FTIR spectrum of L-Lactide.



**Figure 4.** FTIR spectrum of PolyL-Lactic Acid.

Both asymmetrical and symmetrical vibrations of C-O-C appear at 1186  $\text{cm}^{-1}$  and 1085  $\text{cm}^{-1}$  respectively. Peak at 1267  $\text{cm}^{-1}$  represent the overlap between C-O-C stretching vibration and C-H bending vibration. However, as expected peak at 936  $\text{cm}^{-1}$  for (C=O)-O ring of lactide did



not observed in FTIR spectrum of PLLA which is confirmed that the ring opening polymerization of lactide was undoubtedly occurred.

### Nuclear Magnetic Resonance (NMR)

#### Proton Nuclear Magnetic Resonance ( $^1\text{H}$ NMR)

The  $^1\text{H}$  NMR spectrum of PLA given in Figure 5 shows signals at 1.6 ppm ( $d, 3\text{H}, -\text{CH}_3$ ) and 5.1 ppm ( $q, 1\text{H}, -\text{O}-\text{CH}(\text{CH}_3)\text{CO}$ ), corresponding to methyl and methine protons, respectively. A weak quartet resonance at 4.4 ppm ( $q, 1\text{H}, \text{HO}-\text{CH}(\text{CH}_3)\text{CO}$ ) related to the proton neighbor of the hydroxyl end group. A signal at 1.5 ppm ( $d, 3\text{H}, \text{HO}-\text{CH}(\text{CH}_3)\text{CO}$ ) was assigned to methyl end group of PLA.

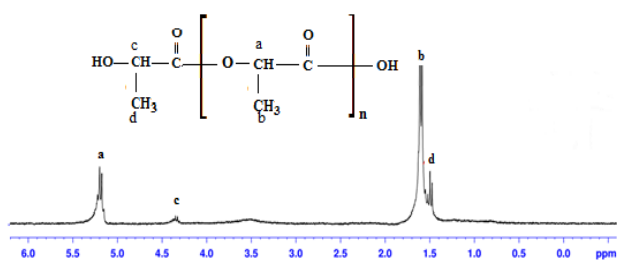


Figure 5.  $^1\text{H}$ NMR of synthesized PLLA under microwave irradiation.

#### Carbon Nuclear Magnetic Resonance ( $^{13}\text{C}$ NMR)

The spectrum shown in Figure 6 represent the ( $^{13}\text{C}$  NMR) of the obtained PLLA, signals at 69.2 (C, CH), 16.8, (C,  $\text{CH}_3$ ), 169.5 (C,  $\text{C}=\text{O}$ ) confirmed the structures of synthesized.

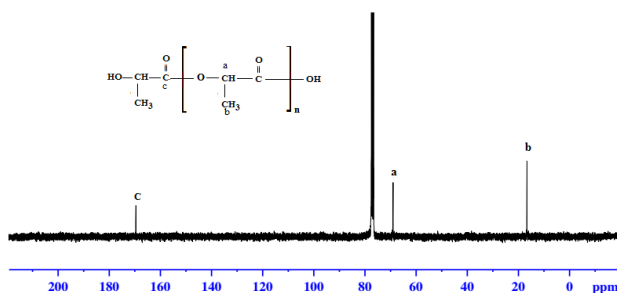


Figure 6.  $^{13}\text{C}$ NMR of synthesized PLLA under microwave irradiation.

### Thermal analysis

TG curve of Poly Lactic acid has been shown in Figure 7. The thermal stability of the polymer obtained by microwave irradiation system which examined by determining the weight loss of a sample at a programmed heating rate of ( $10^\circ\text{C}/\text{min}$ ) under a stream of air. It appears that up to  $160^\circ\text{C}$  there was no obvious weight losing in the sample. The thermogram has shown one

thermal decomposition step at  $265^\circ\text{C}$  with a major weight loss within the range of  $180\text{--}320^\circ\text{C}$ . This is related to session and volatilization of the polymer components. The thermogram shows zero mass left at  $600^\circ\text{C}$ .

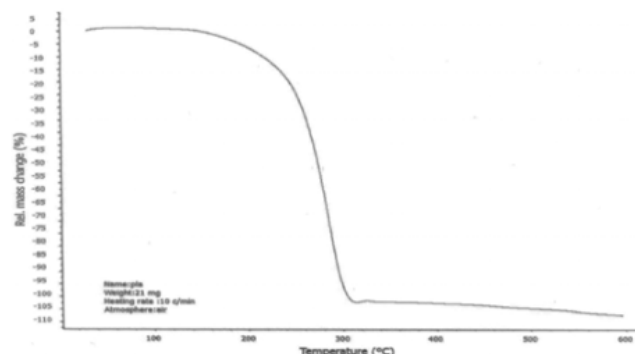


Figure 7. TGA of the synthesized PLLA.

The DSC thermogram (Figure 8) exhibited two endothermic transitions at  $115\text{--}132^\circ\text{C}$  due to melting of the polymer chains the thermal gram also shows clear exothermic transition at  $267^\circ\text{C}$ . This in agreement with TGA result that showed clear decomposition occurred at about  $265^\circ\text{C}$  under air condition.

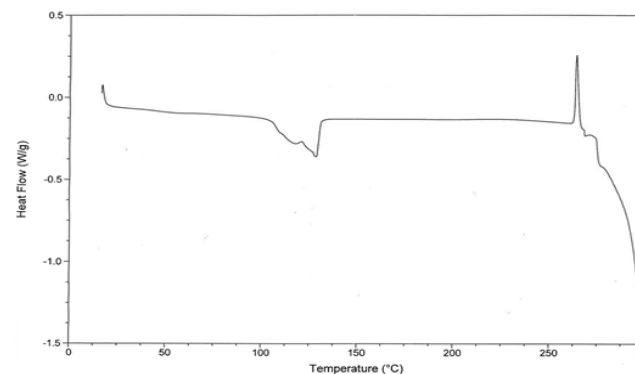
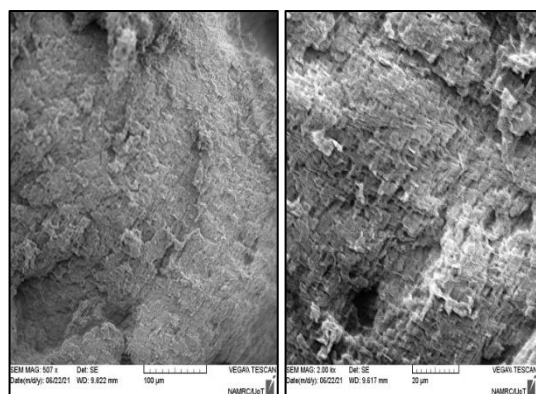


Figure 8. DSC of the synthesized PLA under microwave irradiation.

### Scanning Electron Microscope (SEM):

The morphology of the obtained PLA in Figure 9 presents homogenous surface compact and dense structure, with no separate entities was observed at the surface of the polymer sample. Such material can be used in bio active encapsulation [48].



**Figure 9.** SEM micrograph of the obtained PLLA.

## CONCLUSIONS

In this research, PLA has been successfully synthesized by ROP under microwave irradiation without using sealed tube as used in microwave assisted polymerization; the produced polymer was crystalline, thermally stable and with high molecular weight. The use of unconventional heating source (microwave) lead to a remarkable reduction in time and temperature degrees required for polymerization in classical routes.

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