Nature of Invention: Chemical molecule and synthesis route

**Applicant:** CHEMINOVA

**Inventors:** LAKSHITA GOPALANI (210555) , AVNI GOUR ( 210240)

**Chemical Formul**a: (C3H4O2)n

**Chemical Name**: Polylactic acid

**Chemical synthesis routes:**

## PREPARATION OF POLY LACTIC ACID (PLA) USING LACTIC ACID:

### RAW MATERIALS :

- Lactic acid
- Thionyl chloride
- Methanol
- Stannous Octoate
- Chloroform
- Methanol

## STEP 1: Production Of Lactide from Lactic acid:


- **Yield: >90%**
- **Purity = 99.5%**

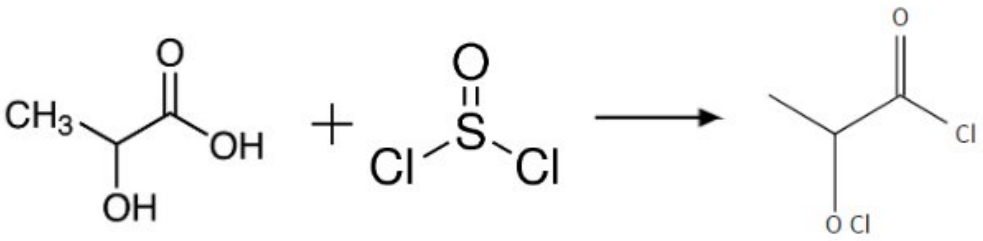
Steps:-

**Purification of Lactic Acid**: Lactic acid is purified to remove impurities that can interfere with the reaction by distillation

1. **Formation of Lactoyl Chloride**: Purified lactic acid + thionyl chloride-> lactoyl chloride.

- Catalyst - pyridine
- Temperature = -10°C - 20°C.
- Time - 1-2 hours.

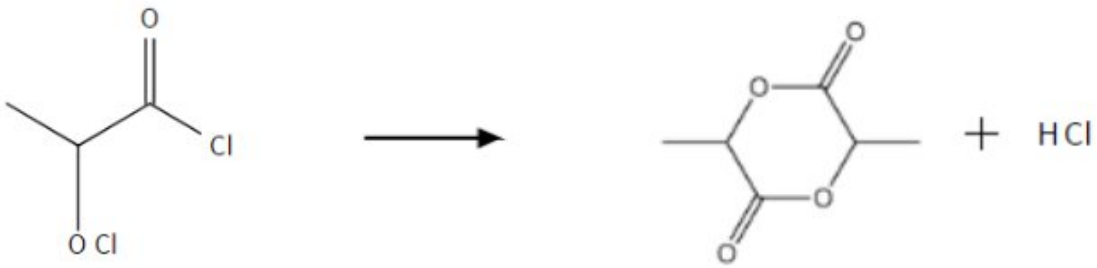
CH3CH(OH)COOH + SOCl2 → CH3CH(OCl)COCl + SO2 + HCl




2. **Formation of Lactide**: Lactoyl chloride is then heated to form lactide.

- carried out under vacuum for removal of HCl byproduct.
- Temperature = 100°C - 170°C,
- Time = 1-10 hours.

2CH3CH(OCl)COCl → (CH3CHCO)2O + 2HCl

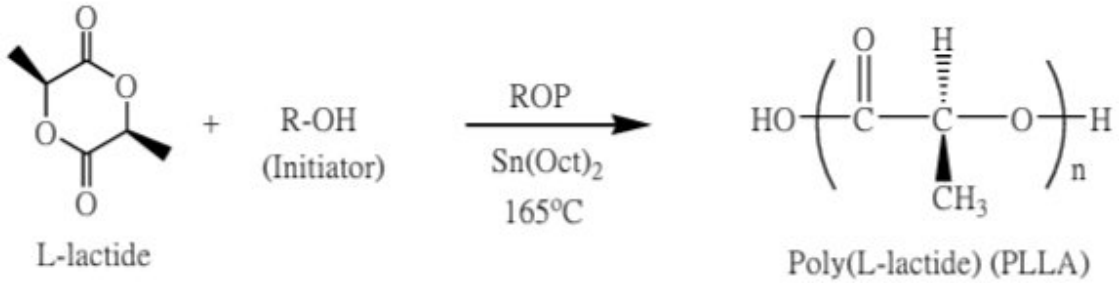


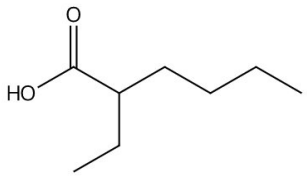

3. **Purification of Lactide**: The lactide is purified by distillation to remove any impurities.

- Reduced pressure(P = 50-200 mbar)
- Temperature = 80°C - 110°C

## STEP 2: Ring Opening Polymerisation of Lactide to produce PLA

Source:
https://iopscience.iop.org/article/10.1088/1757-899X/213/1/012022/pdf




- 1/2 Sn(Ⅱ)

Catalyst (Stannous octoate)

- **Reactant -** Lactide
- **Initiator** - Methanol
- **Catalyst -** Stannous Octoate
- **Solvent for purification -** Chloroform
- **Solvent for precipitation-** Methanol

- **Exothermic Reaction**
- **Yield** : ***89%***

**Steps:-**

1) Lactide -> Stirr under vacuum to remove any residual moisture. *(45degC 12h)*

2) Heat the reaction mixture*(Lactide + methanol(Initiator) + Catalyst)*(nitrogen atmosphere)-*(165degC for 4 hrs)*

3) Cool the mixture to room temperature.

4) Dissolve the resulting PLA in Chloroform and filter the solution to remove impurities.

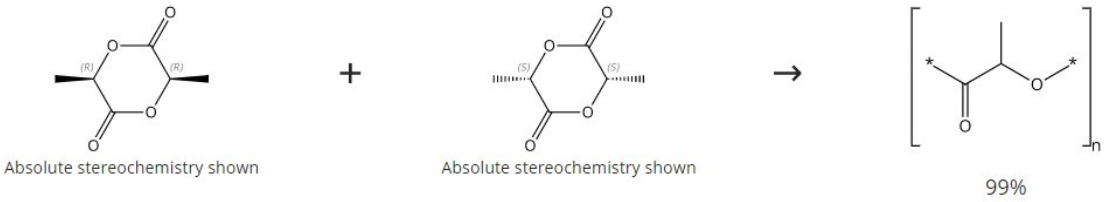
5) Add the polymer solution to methanol while stirring to precipitate the PLA

6) Collect the precipitated PLA by filtration *(in vacuum)* and wash it with methanol to remove any residual catalyst and oligomers.

7) Dry the purified PLA under vacuum at (*50degC for 24 h*)

*Note: Nitrogen atmosphere is used to prevent side reaction (oxidation of the lactide monomer during heating)*

## ALTERNATE REACTION: PREPARATION OF PLA USING L-LACTIDE & D-LACTIDE




**RAW MATERIALS :**

- L-Lactide
- D-Lactide
- Thiourea
- Sparteine
- Benzyl alcohol
- Dichloromethane
- Benzyl Alcohol
- Benzoic Acid
- Methanol

**Source:**

https://scifindern.cas.org/searchDetail/reaction/641ed5ea2e94d03e953f8663/reactionDetails
https://pubs.acs.org/doi/pdf/10.1021/nl0726813?src=getftr

- **Yield**: 99%
- **Reagent**: L-Lactide & D-Lactide
- **Catalysts**: Thiourea, Sparteine & Benzyl alcohol

- **Solvents**: Dichloromethane.
- **Other chemicals Required**: Benzyl Alcohol, Benzoic Acid, Methanol

STEPS:-

1. Stir benzyl alcohol initiator, lactide monomer and thiourea catalyst (131 mg, 0.35 mmol).
2. Dissolve the mixture in methylene chloride (~ 5g).
3. Add (-)-sparteine co-catalyst (82 mg, 0.35 mmol) to the mixture.
4. React the mixture for 4 hours.
5. Add an excess of benzoic acid to deactivate the catalyst.
6. Precipitate the polymer in methanol.
7. Dry the precipitate in a vacuum oven until no further weight loss is observed.

**References:**


- https://iopscience.iop.org/article/10.1088/1757-899X/213/1/012022/pdf
- https://scifindern.cas.org/searchDetail/reaction/641ed5ea2e94d03e953f8663/reactionDetails
- https://pubs.acs.org/doi/pdf/10.1021/nl0726813?src=getftr


**List the contributions of each author:**

LAKSHITA GOPALANI (210555) carried out the literature search and find the reaction steps, and product yield as well as found necessary separation steps to achieve desired product purity for preparation of PLA from method one. She also contributed in literature search of method 2

AVNI GOUR ( 210240) carried out the literature search and found the reaction steps, and product yield as well as found necessary separation steps to manufacture PLA from method two .

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