Synthesis of Poly (Lactic-co-glycolic) acid and its micro fabrication by Centrifugal force melt spinning Technique

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ABSTRACT: Polylactic glycolic acid (PLGA) is most important polymer in biomedical applications because we can modify the degradation rate by co-polymerization ratio, processing. PLGA synthesis done by convectional method, with study of various parameter such as time, temperature, monomer and catalyst ration. PLGA can be synthesized by polycondensation (convectional) method at 130°C, for 25 hours. Important characteristics such as melting temperature, glass transition temperature, and degradation temperature was determined by DSC and TGA analysis, it was obtained as 168.44°C, 55.76°C and 87.61°C respectively. Chemical structure was studied by FTIR and NMR. These results helped to study the effect of monomer, catalyst on reaction and determining the parameters for melt and electro spinning. Because of good biocompatibility and biodegradability, they can be used in various areas, such as long-term release systems and the tissue engineering.
Centrifugal force spinning is emerging versatile process for the formation of high surface area nanofibrous scaffolds. In Forcespinnig, the angular velocity of the spinneret forces the solution or polymer melt to elongate through the designed orifices around the spinneret releasing the material out to create fibers in the nano, submicron, or micron scale. With smaller pores and higher surface area than regular fibers, nano-fibers have been successfully applied in various fields, such as, nano-catalysis, tissue engineering scaffolds, protective clothing, filtration, biomedical, pharmaceutical, optical electronics, healthcare, biotechnology, defence and security, and environmental engineering.

I. INTRODUCTION

Plastic waste management may generate toxic air pollution, landfill sites are limited, and recycling methods for waste are expensive and often energy intensive. In addition, petroleum resources are becoming limited [1]. Plastic recycling cannot reduce the plastic pollution, it is necessary to develop bio-degradable polymer that recycle with help of microorganism without any consumption of thermal energy. Depletion of fossil resource in near future leads to development of biodegradable polymer from renewable resources [2].

PGA, PLA, and their copolymers, poly(lactic-co-glycolic) acid (PLGA) are a family of linear aliphatic polyesters have been demonstrated to be biocompatible and degrade into non-toxic components with a controllable degradation rate in-vivo and have FDA (US Food and Drug Administration) approval for clinical use [3]. The degradation of polymeric materials can be altered by changing their structural composition and fabrication techniques. The rate of degradation of polyesters is dependent on the exposed surface area, crystallinity, the ratio between hydroxyl ions and the monomers, configurational structure, copolymer ratio, molecular weight, morphology, stresses, amount of residual monomer, porosity and site of implantation [4], [5]. To achieve intermediate degradation rates between PGA and PLA, various lactic and glycolic acid ratios are used to synthesize PLGA [6].

PLGA is Colorless, odorless, and hygroscopic crystalline solid, with high solubility in water. PLGA can be dissolved by a wide range of common solvents, like chlorinated solvents, tetrahydrofuran, acetone or ethyl acetate [7]. It can be processed into any shape and size, and can encapsulate biomolecules of any size. An additional methyl group renders lactic acid more hydrophobic than glycolic acid, and varying the lactide to glycolide ratios in PLGAs causes them to possess a wide range of hydrophilicity and degradation profiles [8] [9].

The Tg depends on the mobility and flexibility of the polymeric chains. Intermolecular Forces, Chain Stiffness, Cross-Linking, Bulky and flexible pendent group, Plasticizer, and Molecular weight. The Tm is increased if the double bonds, aromatic groups, bulky or large side groups are present in the polymer chain, because they restrict the flexibility of the chain [10] [11].

The branching of chains causes the reduction of melting point, as defects are produced because of the branching. As the GA content increased, Tg of the copolymers decreased and Tm of the copolymers increased [12], [7]. Various factors affect the degradation such as the copolymer composition, the molecular weight and the sequence structure of the copolymer. The degradation of PLGA copolymer occurs by hydrolysis of ester bonds in the presence of water. Lowest molecular weight shows faster rate of degradation [13]. The absolute value of the biodegradation rate of PLGA constants are rise with increasing the GA content. This process happens over four stages: hydration, initial degradation, constant degradation, and solubilization [14].

Nanofibers have the ability to create a very powerful mesh with a wide range to large volume that enhances its performance for various applications such as water filters, tissue engineering scaffold, wounds, fiber blends, drug extracts and protective coatings. The use of fibers is gaining more attention in biomedical use, due to their ability to mimic the external matrix of native tissues and to more accurately regulate the release of biomolecules in order to improve the process of regenerating damaged tissue [15] [16]. Centrifugal force spinning is emerging versatile process for the formation of high surface area nanofibrous scaffolds. The process of centrifugal force spinning is sometimes called rotary jet spinning, force spinning, centrifugal jet spinning or high-speed rotary spinning [17]. This setup consists of a rotating spinneret located in the center of a cylindrical collector. The spinneret has side nozzle holes, which rotates at high speed (generally in the range of 2000-13000 rpm). The polymer solution is pushed to the spinneret, which is ejected in the form of fibers (usually in the range of micro- to nanofibers) due to the centrifugal force generated as a result of high rotation speed. While the polymer solution is ejected, it undergoes a stretching process due to air friction force followed by fast air solidification due to solvent evaporation and finally fibers formation [18] [19]. It also provides the advantage of producing nanofibers from materials with a low dielectric constant. The process has distinct advantages over conventional techniques to fabricate nanofibers [20]. The surface area of a fibre is proportional to the diameter, whereas the fineness of the fibre is proportional to the square of the diameter, and the flexural rigidity of the fibre is proportional to the fourth power of the diameter [21] [22] [23].

M. Kakiage et.al., prepared UHMW-PE fiber with high tensile strength by the combination of melt-spinning and melt-drawing [24]. N. Leelakanok et.al., used 5-fluorouracil (5-FU) chemotherapeutic agent in PLGA fabrication. Incorporation of 5-FU into PLGA matrices has proven to be an effective way to provide sustained release of 5-FU in vivo and has demonstrated promise as a local delivery-based therapy in preclinical studies and in clinical trials [25]. G. Dadol et.al., used a precursor solution with a concentration of 10 % (w/v) PLGA in acetone and CO_2 flow rate of 13 standard cubic feet hour (SCFH) to fabricate the PLGA nanofibers by solution blow spinning [26].

II.   MATERIALS AND METHODS
PLGA Synthesis:

Materials: Lactic acid 90% pure was purchased from Lobachemie, used as monomer. 97% pure Glycolic acid was purchased from Sigma Aldrich, as monomer. Stannous Octoate 99% pure was purchased from Sigma Aldrich and used as initiator and catalyst (it acts as both). Stearyl alcohol and Lauryl alcohol (reagent grade) from SD Fine Chemicals Pvt. Ltd used as co-catalyst. Silicon oil used in thermocouple pocket.

Apparatus: Heating mental, Overhead stirrer, stirring rod with Teflon blade, Vacuum setup, Nitrogen balloon, oil tube, thermometer, Temperature sensor, Thermocouple, thermometer pocket, four necked round bottom flask, nitrogen (gas) purging tube, Weigh machine, beakers, conical flask, pipette, water condenser, separating funnel, vacuum adaptor, stand, clamp, rubber bulk, join and bends, connector, stopper, Nitrogen balloon stand (ring clamp), fiber resistance fabric,

Petri dish, Funnel, Spatulas, Glass stirring rod, Buchner funnel, wood blocks, rubber tube, etc.,

Procedure:

Arrange apparatus as shown in figure. Nitrogen gas was purged to maintain neutral environment. Stirrer speed was maintained about 500 RPM. Vacuum should apply at interval of time after temperature reaches to 110°C. Thermocouple place in thermometer pocket with silicon oil. After arranging the whole setup secure the connection properly before heating started and also check the water inlet and outlet. Lactic acid added before glycolic acid because glycolic acid has higher reactivity compare to lactic acid. Add stannous octoate and/or Stearyl alcohol/ lauryl alcohol (co-initiator) after one hour (oligomerization). To check the progress of rection we determine acid value and/or saponification value. As reaction proceeds acid value decreases. Also determine acid value of monomer and mixture after adding initiator and/or co-initiator.

PLGA synthesis setup

Parameters of PLGA

Batch	Monomer Ratio	Time	Temperature	Catalyst	Co-catalyst
1	**50:50**	23	130	1.2	1.0
2	**55:45**	23	130	1.2	1.0
3	**60:40**	23	130	1.2	1.0
4	**75:25**	23	130	1.2	1.0
5	60:40	23	**90**	1.2	1.0
6	60:40	23	**110**	1.2	1.0
7	60:40	15	**150**	1.2	1.0
8	60:40	5	**180**	1.2	1.0
9	60:40	**15**	130	1.2	1.0
10	60:40	**18**	130	1.2	1.0
11	60:40	**20**	130	1.2	1.0
12	60:40	**25**	130	1.2	1.0
13	60:40	23	130	**0.5**	**0.3**
14	60:40	23	130	**0.8**	**0.6**
15	60:40	23	130	**1.0**	**0.8**
16	60:40	23	130	**1.5**	**1.3**

PLGA purification

1. Vacuum drying: Synthesized product PLGA was dissolved in chloroform/ acetone, and dried in vacuum at 46°C and 55°C. After it keep for cooling.

Vacuum dryer

2.By using Desiccator: The product PLGA was cooled down and then dissolved in chloroform and subsequently precipitated into diethyl ether. The precipitated mixture was collected in petri dish and dried in desiccator using vacuum. Vacuum applied by vacuum pump.

Desiccator and Desiccator connected through vacuum

3.Büchner funnel: The polymer PLGA was purified by first dissolving in chloroform followed by precipitating in methanol under the action of mechanical stirring. And the precipitate dry using Büchner funnel in which vacuum is applied to the arm.

Büchner funnel

Acid value:

Materials: Alcoholic Potassium hydroxide/ sodium hydroxide solution (0.5 N), Phenolphthalein indicator, methyl ethyl ketone (Solvent)/ benzene alcohol neutral mixture, oxalic acid solution of known normality (0.5 N), distilled water.

Apparatus: Burette, pipette, conical flask, volumetric flask, beaker, measuring cylinder, rubber bulk etc.,

Preparation of reagents:

Alcoholic Potassium Hydroxide Solution (0.5 N): Dissolve Potassium Hydroxide (28.05 g) in little amount of distilled water (approx. 250 mL) and dilute it to 1000 mL in a volumetric flask using alcohol (Ethanol).

Phenolphthalein Indicator:

Dissolve Phenolphthalein powder (0.5 g) in 50 mL ethanol and dilute the solution to 100 mL using ethanol. (Also, can use Phenolphthalein solution available in market.)

Procedure:

A.Standardization of KOH solution:

1.Prepare exactly 0.5 N oxalic acid solution using distilled water.

2.Titrate KOH solution (approximately 0.5 N) with 25 mL of oxalic acid solution using phenolphthalein indicator.

3.Find out the exact normality of KOH solution and use the same for further calculation of acid value.

B.Determination of Acid Value:

1.Weigh accurately 2 to 4 gm of sample depending upon the expected acid content.

2.Dissolve it in 10 mL of Benzene alcohol neutral mixture (or any other water miscible suitable solvent) at room temperature.

3.Add 2-3 drops of phenolphthalein indicator to the solution.

4.Fill burette with alcoholic KOH solution.

5.Titrate the solution against the standard alcoholic KOH solution until pale pink color appears permanently (at least for 15 seconds).

6.Record the volume of alkali consumed as "A" mL.

Centrifugal Melt Spinning

Apparatus: Melt spinning instrument, PLGA

Procedure: PLGA was dried in oven for 40 min. to remove moisture from polymer. About 200 grams of material PLGA used for fabrication process. Material was added to the hopper. RPM and temperature were set. Optimized RPM (1200-1500) and Temperature (100-140°C) according to requirements. Polymer material is added through hopper, material was melted in single screw extruder where heating plates are attached. Material will pass to the spinneret which attached with high-speed rotatory motor. PLGA melted in barrel then it passes through the nozzle to spinneret which rotate at high RPM.

Centrifugal melt spinning setup

Spinneret setup with heater and spinneret respectively.

Characterization of PLGA and fibers:
Fourier Transform Infrared (FTIR)
The infrared absorption spectra were collected at 20°C from 4000 - 650 cm–1. The spectra were recorded on a Bruker spectrometer operating in the ATR (Attenuated Total Reflectance) mode.
Nuclear Magnetic Resonance (NMR)
NMR samples have been prepared by dissolving the co-polymers in CDCl3 from Aldrich containing TMS at 0.05%. 1H spectra were obtained at 400 MHz. Measurement has been performed at 300 K on a Bruker spectrometer. TMS was used as internal reference.

Differential Scanning Calorimetry (DSC)
The DSC equipment used, TQ instruments DCS Q100, was programmed to first heat the samples from room temperature -10°C to 200°C at a rate of 10°C/min. An unsealed aluminium sample vessel was used with nitrogen as the carrier gas at a

flow rate of 20 mL/min. The mass of the analysed sample varied from 5 to 10 mg. The DSC curve was the reference for determining the glass transition temperature (Tg) and phase transition temperature (Tm).

Thermogravimetry (TGA)
TG analysis was carried out to measure change in mass with increase in temperature, thermal stability, and maximum degradation temperature for the samples. The test was conducted at a heating rate of 10°C/min from 20°C to 500°C in an unsealed sample vessel under nitrogen atmosphere with a flow rate of 20 mL/min. The equipment used was a PerkinElmer Pyris 1. The mass of the analysed samples varied between 5 and 10mg.

Optical microscopy
Fiber diameter determined by using Olympus BX41 microscope with lens power of 20x and 50x.

Degradation test
Soil burial test
The material is buried in soil beds prepared in the laboratory using standard sieved soil; often a commercial soil-based compost. The soil beds containing the samples are incubated at a constant temperature for between 28 days and 12 months. The moisture content is normally set at 20–30%. Samples are removed for assessment of changes or to check weight loss and to look for the presence and nature of microbial growth

CO2 release test
When polymer degrades it release Carbon dioxide, which react with KOH solution. Carbon dioxide is acidic in nature and potassium hydroxide is a strong base. Hence, carbon dioxide reacts with potassium hydroxide to form potassium carbonate and water as $2KOH + CO_2 \longrightarrow K_2CO_3 + H_2O$. Thus, the mass increases. This increase in the mass gives the mass of CO_2 produced. (ASTMD 6022)

Solubility Test
Small amount of purified PLGA was dissolved in Solvents to check the solubility. Mainly Acetone, Chloroform, THF, Ethanol, Ethylacetate was used.

III. RESULTS AND DISCUSSIONS

Synthesis of PLGA
Parameters Study for PLGA synthesis:

Batch	Monomer Ratio	Time	Temperature	Catalyst	Co-catalyst	Yield	Acid value
1	**50:50**	23	130	1.2	1.0	76	98.2
2	**55:45**	23	130	1.2	1.0	81	85.9
3	**60:40**	23	130	1.2	1.0	92	78.5

4	**75:25**	23	130	1.2	1.0	86	93.5
5	60:40	23	**90**	1.2	1.0	63	179.2
6	60:40	23	**110**	1.2	1.0	77	150.4
7	60:40	15	**150**	1.2	1.0	76	102.5
8	60:40	5	**180**	1.2	1.0	45	98.8
9	60:40	**15**	130	1.2	1.0	68	205.8
10	60:40	**18**	130	1.2	1.0	73	200.7
11	60:40	**20**	130	1.2	1.0	79	172.9
12	60:40	**25**	130	1.2	1.0	83	96.3
13	60:40	23	130	**0.5**	**0.3**	72	124.5
14	60:40	23	130	**0.8**	**0.6**	78	130.5
15	60:40	23	130	**1.0**	**0.8**	84	89.5
16	60:40	23	130	**1.5**	**1.3**	76	94.8

PLGA successfully synthesized at 130°C temperature, in the ration of Lactic acid: Glycolic acid in 60:40, using stannous octoate as catalyst and Lauryl alcohol as co-catalyst in the 1.0 wt.% and 0.8 wt.% respectively. Reaction proceeds for 23 Hr at mechanical stirring of 500 RPM.

Optimized parameter of PLGA

Monomer ratio (60:40)	Lactic acid = 7.5 g Glycolic acid = 1.9 g
Sn(Oct)$_2$ (1.00%)	0.08 mL
Lauryl alcohol (0.8%)	0.05 mL
Time	23 h
Temperature	130°C
Acid value of lactic acid	561.00 mg of KOH/ g of sample
Acid value of glycolic acid	728.81 mg of KOH/ g of sample
Acid value at start	563.50 mg of KOH/ g of sample
Acid value after 23 H	96 mg of KOH/ g of sample
Yield	89%

Calculations
A.   0.5 N Alcoholic KOH from pallets:
56.11 g in 1000 mL is equivalent to 1 N alcoholic KOH solution

χ g in 1000 mL is equivalent to 0.5 N alcoholic KOH solution

$\chi = \frac{56.11*0.5}{1} = 28.05$

28.05 g of KOH dissolved in small quantity of distilled water and it diluted to 1000 mL using ethanol (alc.) in volumetric flask.

B.   0.5 N HCl solution:
For 0.5 N 500 mL HCl solution from 32% conc. HCl acid,
Normality = 0.5 N
Volume = 0.5 Lt

Molar mass of HCl = 36.46 g/mol, Equivalent wt. = 36.46 g
Specific gravity = 1.189
gm of compound required = 0.5*36.46*0.5 = 9.115
volume of conc. Acid = $\frac{9.115}{0.32*1.189}$ = 23.95 mL
Take 23.95 mL of 32% conc. acid and dilute it by using distilled water in 500 mL volumetric flask.

C.   Acid value:
Acid value = $\frac{56.1*\text{volume of KOH required during titration}*\text{Normality of KOH (0.5 N)}}{\text{weight of sample in grams}}$
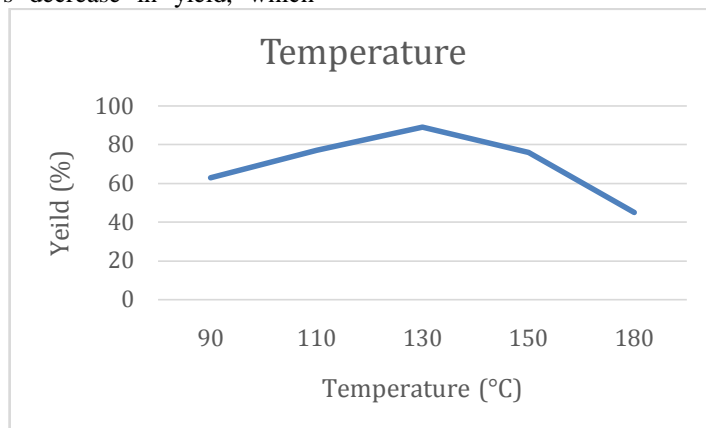
Study the effect of parameter on reaction
1. Effect of temperature
        The reaction was performed at 90°C, 110°C, 130°C, 150°C and 180°C respectively. Monomer ratio LA:GA was 60:40 and time 23 Hr,

Catalyst 1.0%, Co-catalyst 0.8% was used for the reaction. It shows the yield increasing initially but after 150°C, it shows decrease in yield, which indicates polymer degradation at higher temperature.


Effect of temperature
63%, 77%, 89%, 76% and 45% yield was obtained at 90°C, 110°C, 130°C, 150°C and 180°C respectively. Maximum yield was obtained at 130 °C.

2. Effect of time
The reaction was performed for 15, 18, 20, 23 and 25 Hr. Monomer ratio LA:GA was 60:40 and temperature 130°C, Catalyst 1.0%, Co-catalyst 0.8% was used for the reaction. It shows the yield increasing initially but after 25 Hr, it shows decrease in yield, which indicates polymer degradation due to longer prolongation of heat.
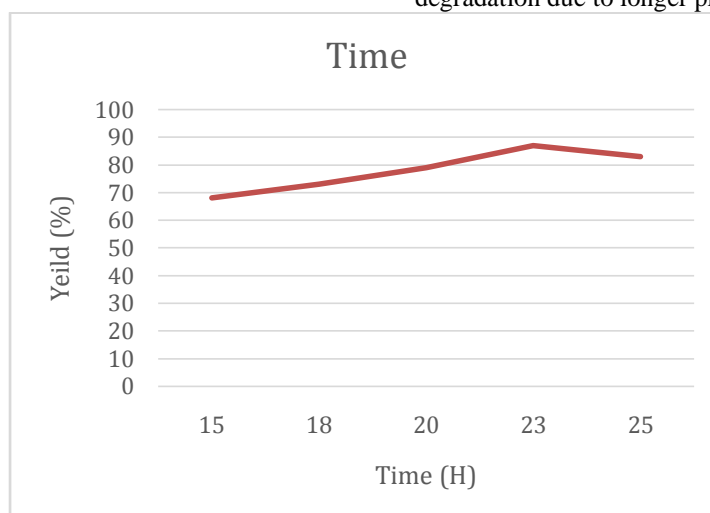
Effect of time
68%, 73%, 79%, 87% and 83% yield was obtained at 15, 18, 20, 23 and 25 Hr respectively. Maximum yield was obtained at 23 H.

3. Effect of catalyst
The reaction was performed in presence of 0.5, 0.8, 1.0, 1.2 and 1.5 wt.% catalyst of batch size. Monomer ratio LA:GA was 60:40 and temperature 130°C, time 23 H, Co-catalyst 0.8% was used for the reaction. It shows the yield increasing initially but after 1.0 wt.%, it shows decrease in yield, due to many active sites generated at once.
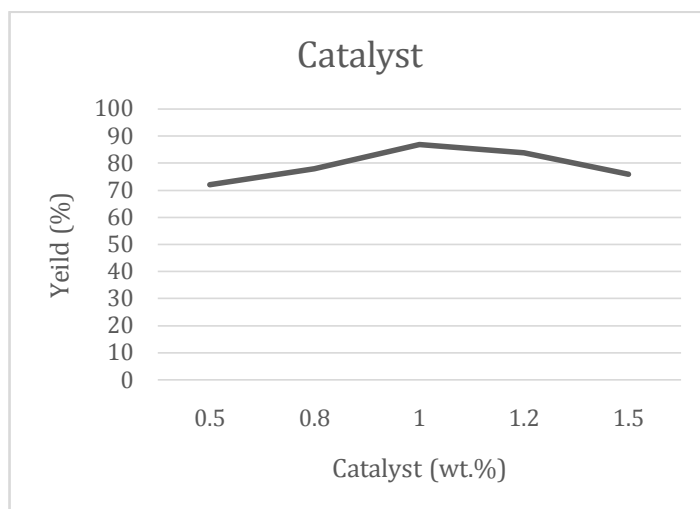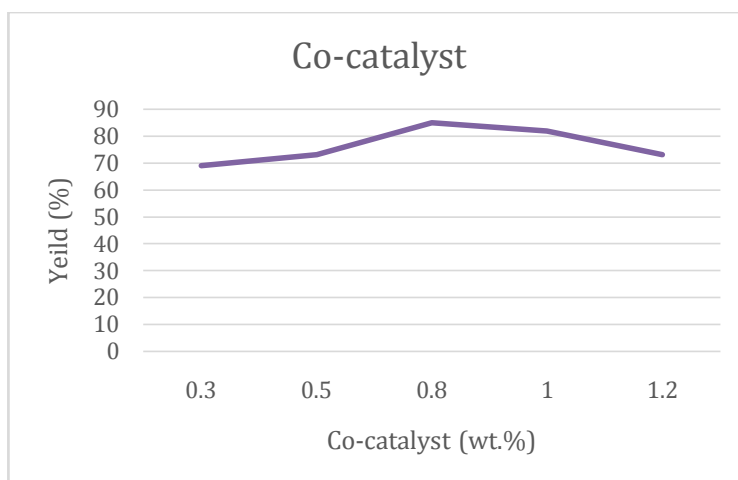
Effect of Catalyst

72%, 78%, 87%, 84% and 76% yield was obtained at 0.5, 0.8, 1.0, 1.2 and 1.5 wt.% catalyst of batch size respectively. Maximum yield was obtained at 1.0 wt.% catalyst.

4. Effect of co-catalyst

The reaction was performed in presence of 0.3, 0.5, 0.8, 1.0 and 1.2 wt.% co-catalyst of batch size. Monomer ratio LA:GA was 60:40 and temperature 130°C, time 23 H, Catalyst 1.0% was used for the reaction. It shows the yield increasing initially but after 0.8 wt.%, it shows decrease in yield, due to many active sites generated at once.


Effect of Co-catalyst

69%, 73%, 85%, 82% and 73% yield was obtained at 0.3, 0.5, 0.8, 1.0 and 1.2 wt.% of batch size respectively. Maximum yield was obtained at 0.8 wt.% co-catalyst.

5. Effect of monomer ratio

Monomer ratio of LA:GA studied was 50:50, 55:45, 60:40 and 75:25. Temperature 130°C,

time 23 H, Catalyst 1.0% and 0.8% co-catalyst was used for the reaction. It shows the yield increasing initially but after 60:40 it shows decrease in yield. As GA content increases hydrophilic nature of co-polymer also increased, LA have a methyl branch attached to main chain which provide good steric hindrance.
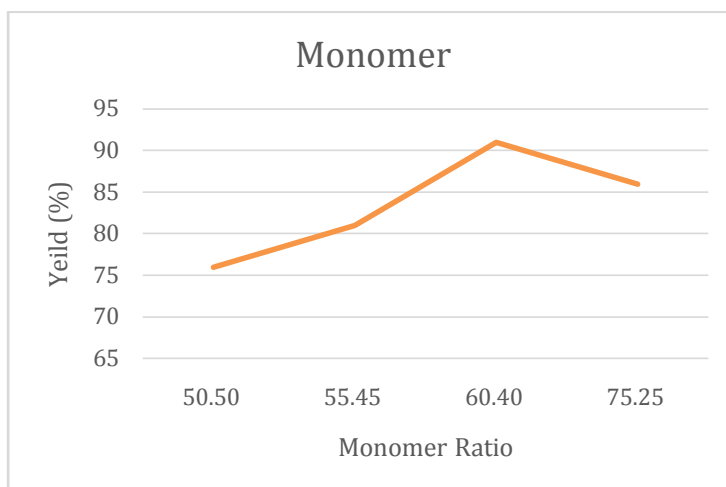
Effect of Monomer ratio

76%, 81%, 91% and 86% yield was obtained at 50:50, 55:45, 60:40 and 75:25 monomer ratio of LA:GA respectively. Maximum yield was obtained at 60:40 ratio.

Characterization of PLGA and fibers
Fourier Transform Infrared (FTIR)

The FTIR shows Characteristic bands of symmetrical and asymmetrical stretching's of CH_2 and CH_3 groups are presented between 2980 and 2850 cm^{-1}. Similarly, bands of asymmetrical deformation of CH_3 are presented in 1375 cm^{-1} and CH_2 in 1450 cm^{-1}, with little decrease in intensity per decrease in mass quantity of IR. The 1760 cm^{-1}, in compounds with PLGA present, is seen in an acute and intense band, caused by the C=O bond stretching of the esters. The intensity is maintained in any composition and, is not visible in 100% IR. The 1185 cm^{-1} and the 1090 cm^{-1} are found to be bands relative to the C-O stretching's of aliphatic polyesters. The vibration of the IR C=C bond appears discrete at 1663 cm^{-1}.
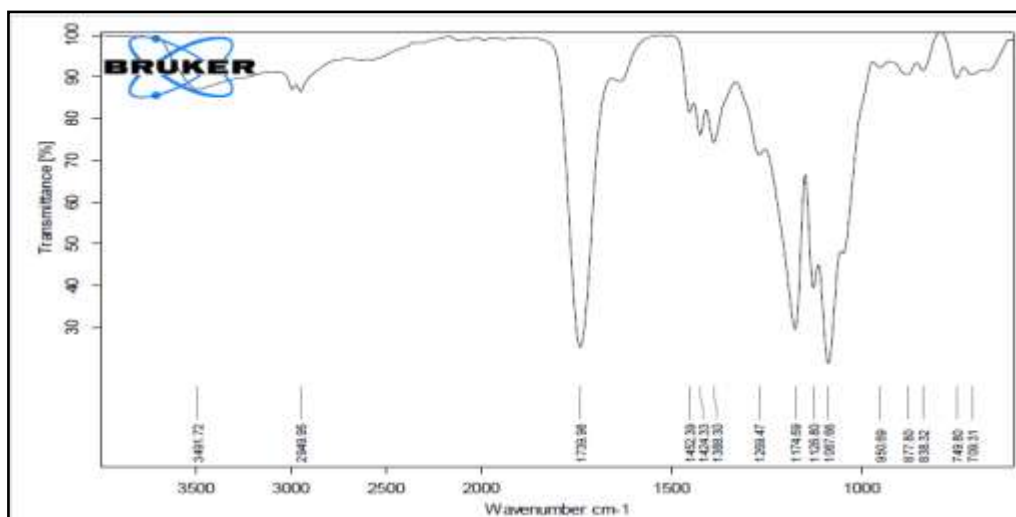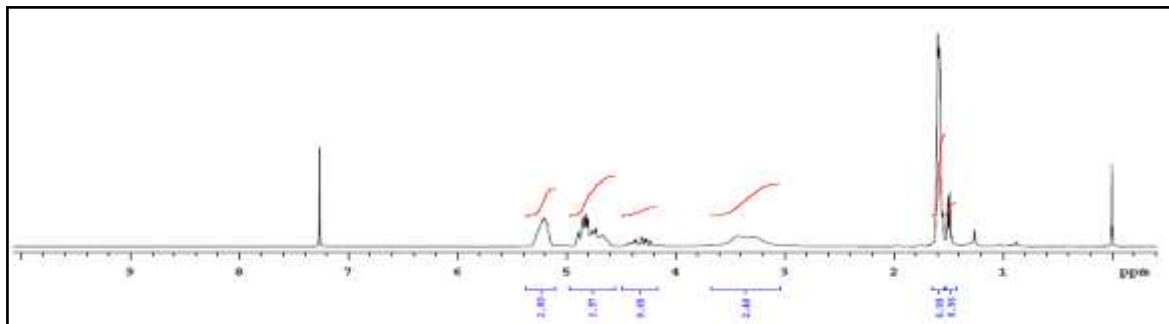

FTIR spectra for PLGA 60:40

Nuclear Magnetic Resonance (NMR)

The methyl group (CH_3) was adopted as a reference for the lactic acid monomer, with a peak of 1.66 ppm. With regard to the glycolic acid, the reference adopted was the peak observed for the methylene group (CH_2), at 4.99 ppm.

¹H NMR spectrum of PLGA 60:40

Differential Scanning Calorimetry (DSC)

The glass transition temperature was observed at 55.76°C and Melting temperature at 168.44°C. In cooling cycle recrystallization temperature noted at 87.61°C.
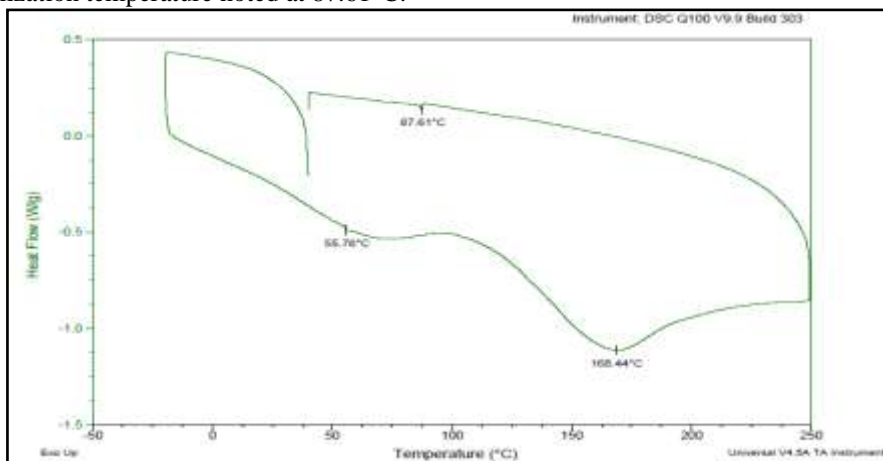
DSC curve of PLGA 60:40 heating from -40 to 250°C

Thermogravimetry analysis (TGA)

Weight loss was 100% for PLGA and the respective values of T_{onset} and $T_{deg.max}$ was observed at 288.64°C and 325.60°C, heating was done from 30°C to 500°C.
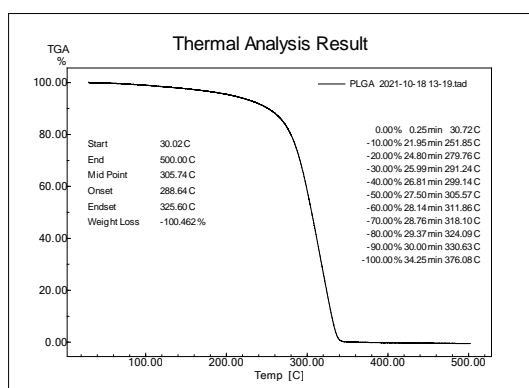
TGA curve of PLGA 60:40 heating from 30 to 500°C.

Optical microscopy

Fiber diameter noted in the range of 21 µm. to 27 µm.

Fiber diameter under lens power of 20x and 50x respectively.

Degradation study
Soil burial test
Weight decrease in PLGA after buried in soil

Days	Weight of sample (gm)
0	5.000
1	5.000
2	5.000
5	5.000
10	4.089
15	4.081
30	4.079
60	4.037
75	4.008

As time increases weight loss of PLGA can be observed, it indicates degradation of polymer. PLGA degrades to release CO2 and water, showing reduction in mass.

CO_2 Release test

When polymer degrades it release Carbon dioxide, which react with KOH solution. Carbon dioxide is acidic in nature and potassium hydroxide is a strong base. This increase in the mass gives the mass of CO_2 produced.

Only soil, Soil and polymer after 5 days, Soil and polymer after 25 days with KOH solution placed in it, and sealed respectively.

Only soil form 7.6 gm of salt, whereas soil and polymer for 5 days form 8.2 gm solids, since solid weight increases polymer must release CO_2 gas which reacts with KOH solution. Soil and polymer for 25 days form 9.8 gm of salt, which shows sample was degrading.

Solubility test

Higher amount of lactic acid polymers can be dissolved using chlorinated solvents whereas higher glycolic acid content materials will require the use of fluorinated solvents such as HFIP. PLGA dissolved dichloromethane, chloroform, and by water miscible solvents, like acetone or tetrahydrofuran, Ethylacetate.

IV. CONCLUSION AND FUTURE WORK

Variety of parameter variations, such as molecular weight, monomer ratio, amount of catalyst etc. can be used to full fill demand required or to achieve desire polymer properties. Ring opening polymerization gives high molecular weight but monomer required for this are expensive, Condensation polymerization is cheap comparative to ROP but it gives very low molecular weight. PLGA, PLA and PGA are easily degraded into environment friendly product and their degradation rate can also be modified. They have tunable properties, featured with excellent characteristics, such as biodegradability, biocompatibility, mild undesirable host reactions, three-dimensional and directional porous structures, PLGA, is broadly studied and used as different biomaterials.

Poly (lactic-co-glycolic) acid was successfully synthesized at 130°C temperature, in the ration of Lactic acid: Glycolic acid in 60:40, using stannous octoate as catalyst and Lauryl alcohol as co-catalyst in the 1.0 wt.% and 0.8 wt.% respectively. Reaction proceeds for 23 Hr at mechanical stirring of 500 RPM. The reaction progress check by acid value. As polycondensation reaction occurs, acid value start decreasing, from 650 to 120-170 KOH/gm of sample.

The structural characteristics of PLGA were done by FTIR and NMR. Thermal properties were measured by DSC and TG. Glass transition temperature was obtained at 55.76°C and Melting temperature at 168.44°C. In cooling cycle recrystallization temperature noted at 87.61°C. Tonset and Tdeg.max was observed at 288.64°C and 325.60°C, heating was done from 30°C to 500°C.

The extra methyl group in the PLA repeating unit (compared with PGA) makes it more hydrophobic, reduces the molecular affinity to water, and leads to a slower hydrolysis rate. The morphology and crystallinity strongly influence PLA rate of biodegradation and mechanical properties. As temperature and time increased polymer starts degrading.

Melt spinning is one of the most versatile and widely used methods in the production of polymeric fibers. Centrifugal spinning is very advantageous process because of rate of fiber production and can be performed without use of solvent, but the major disadvantage is precise control of temperature. Fiber was successfully fabricated at 1200 RPM and Temperature 110°C in the range of 21 µm. to 27 µm.

Current developments are focused on obtaining nano- or submicron-filaments using a melt spinning technique. This technique requires high temperature between the polymeric melt within the extruder and a metallic collecting device. Furthermore, utilization of new polymers and multicomponent spinning, specifically bicomponent systems, are expected to grow in the future. Spinning of new polymers would present new challenges, and further developments are envisaged in the field of post spinning operations such as quenching and drawing. Improvement of the filament quality at high spinning speeds, integration of quality assurance devices with the fast production speeds, and ensuring stability in spinning of finer filaments are some of the research challenges for the years ahead.

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