Preparation and Characterization of Phthalic Acid-Propane-1, 2-Diol-Glycerol Co-Polyester as A Biodegradable Polymer

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Abstract: Phthalic acid propane-1,2 diol-glycerol co-polyester (PPGC) is synthesized from phthalic acid and propane-1, 2-diol with 5% glycerol of total weight as a crosslinking agent using Dean-Stark apparatus with Ferric Chloride (Approximately 0.4% of the total weight) as catalyst and o-xylene as the reaction medium at temperature 137-141° C for about 5 hours. The co-polyester is characterized by its molecular weight, elemental analysis, solubility tests in common organic solvents, swelling behavior in water and ethanol, melting point, density, pH test, IR-spectra and degradation test. Its probable structure is assigned. Molecular weight is determined by end group analysis and viscosity method. The degradation study of the co-polyester is carried out in different solutions of pH values 1.70 - 9.16. At room temperature (30°C) the co-polyester remained intact in the acid solutions (1.70 - 5.80 pH) but degraded in basic solutions (7.14 - 9.16 pH). In normal soil, it is almost 100% degradable within 60 days.

Keywords: Phthalic acid propane-1, 2-diol glycerol co-polyester, dean-Stark apparatus, synthesis, crosslinking agent, ferric chloride, O-xylene, biodegradable, polymer.

1. INTRODUCTION

The polymer which are being produced synthetically are not erodible in nature. For non-degradability they are stable for long time and are imparting various inconveniences, producing environmental pollution.

Recently considerable interest is being focused on the development of biodegradable polymers [1, 3] for specialized applications. Such as controlled release formulation, insecticide and pesticide carriers as well as non-toxic surgical implant materials. Many of these polymers have a built in self-destruct mechanism by which they undergo slow hydrolytic and microbial degradation, releasing the impregnated materials at controlled rates. The modification of synthetic polymers [4, 5] is one of the potential routes outlined for achieving biodegradability.

Moreover, most of the commercial available polymers are virtually resistant to microbial attach and disposal of such synthetics is becoming problem and creating ecological imbalance [6, 8].

Our aim is therefore to develop novel commercially viable polymers [9, 10], specifically designed to degrade under controlled biological conditions [11, 12]. In this connection, we have chalked out to synthesized biodegradable polymers from phthalic acid, propane-1, 2-diol and glycerol.

The present work, which suggests synthesizing biodegradable polymers can be divided into three parts:

Phthalic acid-propane-1, 2-diol-glycerol co-polyester will be synthesized [13, 14] and then characterized by determining their molecular weights, IR spectra, elemental analysis, solubility test in common organic solvents, melting point density and swelling behaviors in water and in ethanol.

Phthalic acid contains two carboxyl groups, propane-1, 2-diol contains two hydroxyl groups and glycerol contains three hydroxyl groups (used as crosslinking agent). Therefore, their esterification will produce co-polyester.

The polymer to be synthesized if exhibit suitable biodegradability [15, 16] may be used as coating materials for the controlled release of bioactive agent.

2. MATERIALS AND METHOD

2.1. Chemicals & Apparatus

(i) Phthalic acid, propance-1, 2-diol

(ii) Glycerol

(iii) Ferric Chloride

(iv) Dean Stark apparatus

(v) Round bottom flask

(vi) Heating mantle

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E-ISSN: 2311-8717/14

(vii) Thermometer (Temperature range 0 to 360˚C)

(viii) Reaction vessel with vacuum pump

(ix) Vacuum desiccator and

(x) Condenser

2.2. Required Solution Preparation

- Alcoholic 0.1 N KOH solution
- Standard succinic acid solution
- Phenolphthalein solution
- Hydrochloric acid
- Sodium carbonate solution

2.3. Materials and Their Purification

The various materials used in typical polymer synthesis needed to be purified, since chemical purity is especially important to produce polymer of good quality.

- Monomer and catalyst: Phthalic acid was a laboratory reagent grade chemical from E. Merck Limited, Mumbai. Propance-1, 2-diol was an A.R. grade chemical from E. Merck Limited, Mumbai and ferric chloride used as catalyst was an A.R. quality chemical from E. Merck Limited, Mumbai, were used as such.

- Solvents: Distilled water was redistilled from alkaline potassium permanganate solution by an 'all glass' distilled apparatus. The other solvents acetone, benzene, chloroform, toluene, diethyl ether etc. were A.R. quality. But solvent ethanol was produced by Cerew & Co. (Bangladesh) Ltd. The impure ethanol was refluxed with quicklime and then distilled at 78˚C. Other solvents were also distilled before use.

2.4. Procedure of Polymer Synthesis

Phthalic acid, propance-1, 2-diol, 5% glycerol together with ferric chloride as catalyst (Approximately 0.4% of the total weight) were taken in a 250 ml round bottom flask which was connected to a dean stark apparatus for eliminating the byproduct water azeotropically with O-xylene [17, 18]. The reaction mixture heated at 137-141˚C for 5 hours. When eliminating of water subsided, the reaction mixture was sticky at room temperature [19, 21], which was stored in a vacuum desiccator. In this way, five different co-polyester of phthalic acid, propane-1, 2-diol along with 1, 2, 3, 4 and 5% of glycerol on the bases of total weight respectively were used to obtained five polymer samples (I, II, III, IV, V). Here 5% glycerol was used as a cross linking agent [22, 24].

2.5. Purification of Polymer

The polymer formed maybe contaminated with the unreacted monomer, initiator, and lower molecular weight of polymer, solvent and hence need to be isolated and purified.

The synthesized polymer is dissolved in a suitable solvent forming a solution and precipitating it using a non-solvent can do purification of the polymer [25, 27]. To get the best result, the polymer solution is diluted to 2-5% concentration and added a large volume of non-solvent under vigorous stirring. The non-solvent should be freely miscible both with the solvent and the monomer. On precipitating from the solution with water [28, 30], the polymer was separated out as solid lumps. Re-dissolve and re-precipitating the polymer a couple of time improved the purity of the polymer. After purification the polymer sample was then vacuum dried at 50˚C and finally stored in vacuum desiccator [31, 33].

2.6. Characterization of Synthesized Polymer

The co-polyester was insoluble in water but soluble in common organic solvent e.g. acetone, ethyl acetate, acetic acid, rectified sprit, ethanol, chloroform and mixed solvent (toluene: ethanol, 1: 3). Molecular weight determination was carried out by end group analysis and viscosity method. Viscosity method was applied by knowing the molecular weight of different fractions of co-polyester by end group analysis. The polymer was characterized [34, 35, 36, 37] by IR spectra, solubility in common organic solvent, melting point, elemental analysis, hydrolytic test, soil degradation test and equilibrium swelling behavior in water and ethanol. Hydrolytic test was carried out in acid and basic medium.

3. RESULTS AND DISCUSSION

Phthalic acid is a di-carboxylic acid, propance-1, 2-diol is a diol and glycerol is a triol, the co-polyester was obtained brown in color, which was sticky at room temperature.

3.1. Solubility

Solubility of this co-polyester in various organic solvents at the ambient temperature is presented in Table **1**. From the Table it can be seen that acetone, ethyl acetate, acetic acid and mixed solvent (toluene: ethanol, 1: 3) are good solvent for this polymer. Ethanol, rectified sprit, chloroform are poor solvent and diethyl ether, xylene, benzene, carbon tetra chloride and water are non-solvent for this co-polyester.

Table 1: Solubility Behavior of Phthalic Acid, Propane-1, 2-Diol- Glycerol Co-Polyester at 31˚C

Solvent	Solubility
Acetone	++
Acetic acid	++
Benzene	-
Chloroform	+
Carbon tetra chloride	-
Diethyl ether	-
Ethyl acetate	++
Ethyl alcohol	+
Formic acid	-
Mixed solvent (Toluene: Ethanol)	++
Rectified sprit	+
Water	-
O-xylene	-

In the Table (++), (+), (-) indicates that, high solubility, low solubility and insoluble respectively.

3.2. Swelling Behavior

Equilibrium swelling value of the co-polyesters I, II, III, IV and V in water and ethanol at the ambient temperature are present in the Table **2** which shows that the equilibrium swelling value of the sample-V in each of the two solvents is the lowest. It is observed that the equilibrium swelling value are in the order I>II>III>IV>V. Again for a series of cross linked polymers which are chemically similar, the higher the cross link density in the network the lower is the swelling in a given solvent at a particular temperature. So the results of swelling study indicate that the cross link density as well as molecular weight follows the order V>IV>III>II>I. Again for each polymer sample the equilibrium swelling values in the two solvents are in the order: Ethanol> Water. This probably because of the better solvent property of ethanol than water.

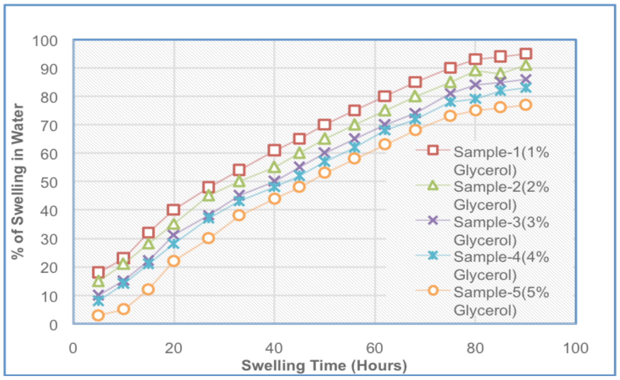

Figure 1: Swelling behavior of Phthalic acid-propane-1, 2-diol-glycerol co-polyester in water at the ambient temperature.

Table 2: Swelling Behavior of Phthalic Acid, Propane-1, 2-Diol-Glycerol Co-Polyester in Water at the Ambient Temperature

Swelling time (Hrs.)	% Swelling				
	Sample-I P1: P2= 1: 1 & Glycerol 1%	Sample-II P1: P2= 1: 1 & Glycerol 2%	Sample-III P1: P2= 1: 1 & Glycerol 3%	Sample-IV P1: P2= 1: 1 & Glycerol 4%	Sample-V P1: P2= 1: 1 & Glycerol 5%
3	15.12	12.30	8.50	5.50	2.50
9	25.00	21.50	15.00	12.70	5.00
15	32.50	28.00	25.45	20.50	13.50
21	39.50	36.50	33.50	29.12	22.50
27	46.50	42.00	40.00	36.50	30.50
33	54.00	51.00	46.50	43.50	38.00
39	59.50	58.00	54.00	49.50	44.00
45	67.00	63.00	60.00	55.00	48.50
51	73.00	68.00	64.00	59.00	55.50
57	77.50	72.50	67.50	64.50	60.50
63	82.00	76.00	72.50	68.30	67.00
69	85.50	82.50	79.00	73.50	70.00
75	88.00	84.50	82.50	77.50	73.50
81	91.50	86.50	84.00	79.50	75.56
87	93.00	87.00	84.50	82.00	76.54
93	94.30	89.50	86.50	83.50	77.52

Table 3: Swelling Behavior of Phthalic Acid, Propane-1, 2-Diol-Glycerol Co-Polyester in Ethanol at the Ambient Temperature

Swelling time (Hrs.)	% Swelling				
	Sample-I P1: P2= 1: 1 & Glycerol 1%	Sample-II P1: P2= 1: 1 & Glycerol 2%	Sample-III P1: P2= 1: 1 & Glycerol 3%	Sample-IV P1: P2= 1: 1 & Glycerol 4%	Sample-V P1: P2= 1: 1 & Glycerol 5%
3	32.50	28.50	22.45	18.50	12.45
9	45.00	39.50	31.50	27.00	19.50
15	55.00	46.50	39.50	34.50	27.50
21	65.50	57.50	50.00	43.00	34.00
27	75.50	67.50	58.00	52.50	42.50
33	83.50	76.50	67.00	60.50	52.25
39	90.50	83.50	75.50	69.50	60.53
45	97.50	90.50	84.50	79.50	67.55
51	100.50	95.50	89.50	85.50	76.08
57	103.50	98.50	94.50	91.50	81.50
63	106.50	103.50	98.00	93.50	83.50
69	107.50	104.50	100.00	94.50	84.50
75	108.50	105.00	101.00	95.50	85.56
81	109.50	106.50	102.45	95.50	85.50

Table 4: Comparison Between Equilibrium Swelling of Phthalic Acid Propane-1, 2-Diol-Glycerol Co-Polyester in Water and Ethanol at Ambient Temperature

Polymer Sample	Acid/Diol Mole ratio	% of Glycerol	% of Swelling at ambient temperature in	
			Water	Ethanol
I	1: 1	1	94	109
II		2	89	106
III		3	86	102
IV		4	83	95
V		5	77	85

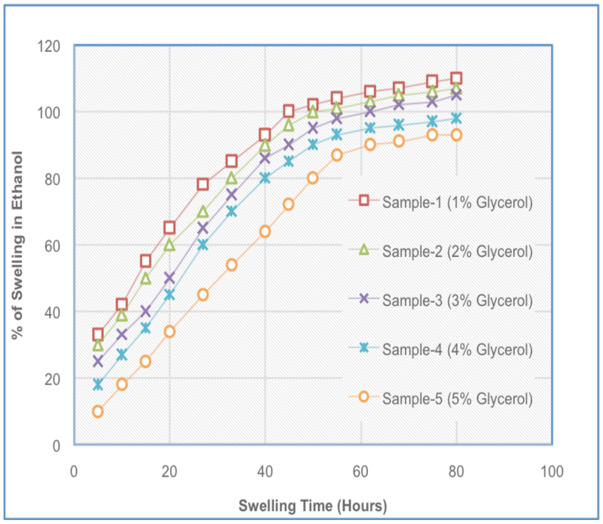

Figure 2: Swelling behavior of Phthalic acid-propane-1, 2-diol-glycerol co-polyester in ethanol at the ambient temperature.

3.3. Fractionation

The polymer sample-V was fractionated by fractional temperature at (20 ± 0.50)˚C using acetone as solvent and water as non-solvent carried out the precipitation technique. The result obtained from the experiment was given in the Table **4**.

The above Table (Table **5**) shows that, the fraction no.-3 (Molecular weight- 20375 gm.) is the highest in quantity in un-fractionated polymer sample.

3.4. Elemental Analysis

The elemental analysis of phthalic acid propane-1, 2-diol-glycerol co-polyester is given below:

a. Calculated from the structure : C=60.96%, H=5.11% , O=33.93%

Table 5: Molecular Weight Distribution of Fractioned Phthalic Acid Propane-1, 2-Diol-Glycerol Co-Polyester in Un-Fractionated Polymer

Fraction No.	Wt. Percentage in gm.	Molecular Wt. by end group analysis (M_n)
1	10.20	25150
2	16.50	22344
3	36.10	20375
4	22.20	18250
5	8.30	14750

Table 6: Comparison Between Molecular Weights Obtained by End Group Analysis and it Obtained by Viscosity Method

Fraction No.	Molecular Wt. by end group analysis (M_n)	Molecular Wt. by viscosity method (M_n)	Degree of polymer, P
1	25150	27207	132
2	22344	25138	122
3	20375	22184	107
4	18250	19907	97
5	14750	16476	80

b. Found from the sample : C=61.78%, H=5.20% , O=28.03%

It is found that, the percentage composition of the sample obtained by calculation matches with the obtained by analysis. The product is accordingly confirmed.

3.5. Density & Melting Point

Density of the prepared polymer at 31˚C is 1.42 gm./cc

The melting point of the co-polyester is 83-89˚C.

Table 7: pH Stability Test for Phthalic Acid Propane-1, 2-Diol-Glycerol Co-Polyester in Hydrochloric Acid Solution at 30˚C. (Initial pH = 4.24)

Reading	Time (Hours)	pH of HCl solution	pH of Polymer + HCl solution
1	0	4.24	4.24
2	1	4.24	4.15
3	2	4.23	4.12
4	3	4.23	4.08
5	4	4.21	4.05
6	5	4.20	4.02

Table 8: pH Stability Test for Phthalic Acid Propane-1, 2-Diol-Glycerol Co-Polyester in Hydrochloric Acid Solution at 30˚C. (Initial pH = 7.92)

Reading	Time (Hours)	pH of HCl solution	pH of Polymer + HCl solution
1	0	7.92	7.92
2	1	7.91	7.67
3	2	7.89	7.58
4	3	7.89	7.50
5	4	7.87	7.41
6	5	7.85	7.40

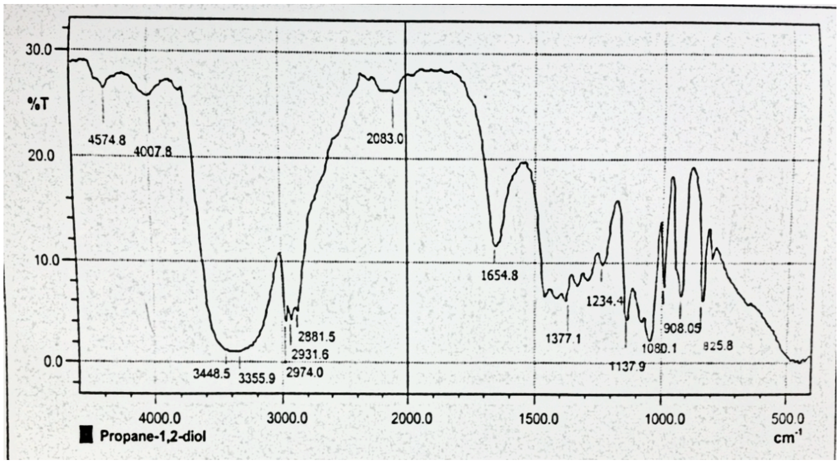

Figure 4: IR Spectra of Propance-1, 2-diol.

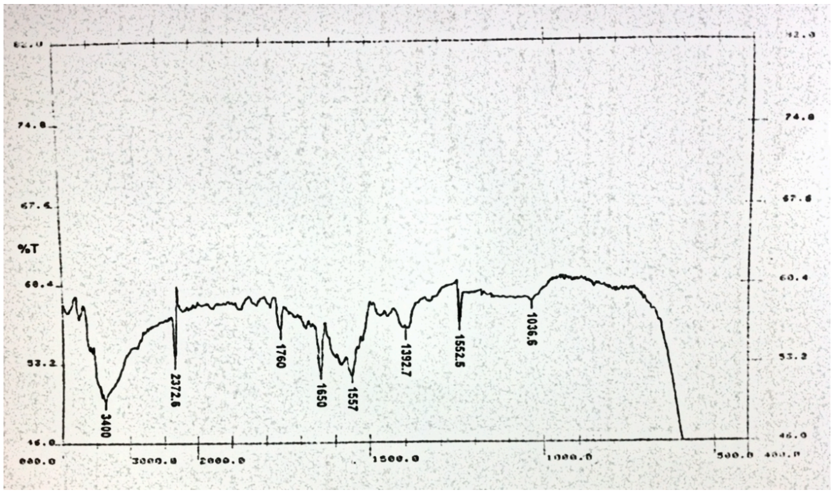

Figure 5: IR Spectra of Phthalic acid-propane-1, 2-diol-glycerol co-polyester.

3.6. pH Stability Test

pH stability were tested of prepared co-polyester in the different pH of hydrochloric acid solution with time as shown in the Table **6** and Table **7**.

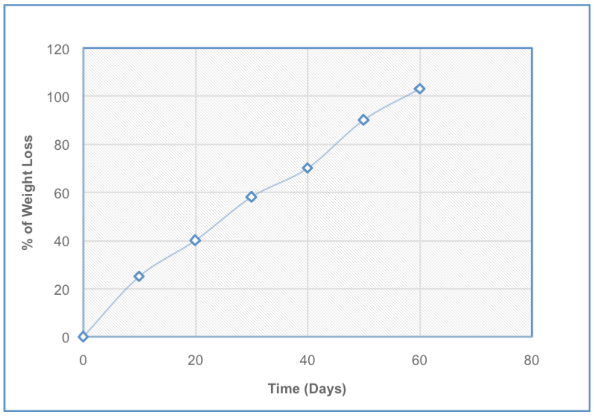

Figure 3: Soil degradation of Phthalic acid-propane-1, 2-diol-glycerol co-polyester at normal weathering condition.

3.7. Degradation Test

Biodegradability test was done in soil with different time interval for the prepared co-polyester as shown in the Figure **3**.

3.7. IR Spectra

The broad band representing the -OH group at the region 3100- 3200 Cm^{-1} in the spectrum of the diol (Figure **4**) and at the region 3300-3500 Cm^{-1} in the spectrum of the triol which disappeared in the spectrum of the polymer.

The >C = O stretching frequency at the 1678-1685 Cm^{-1} region (Figure **5**) of the spectrum of the di-acid shifted to 1642-1650 Cm^{-1} region and a new band representing the ester linkage appeared at 1252.5 Cm^{-1} region (Figure **5**) of the polymer. All these indicate the reaction between -OH and -COOH groups forming ester linkage.

4. CONCLUSION

Now a day's biodegradable polymer plays an important role in pharmaceuticals and many other industries in the world. Our research target was to synthesize a biodegradable polymer in a convenient way. From the degradation study it was found that, phthalic acid-propane-1, 2-diol-glycerol co-polyester remained intact in the gastric fluid (pH = 1.2) but gradually degraded in intestinal fluid (pH= 7.4). Due to inadequate facilities and shortage of manpower we have considered only preparation and along with some studies were performed. But there are some positive future opportunities to study about this biodegradable polymer. This polymer may can use as enteric coating material. So we want to extend our research in future about this polymer as an enteric coated material. Enteric coating material resists the release of the drug from the core tablet in the gastric environment but it aids drug release in the intestine. If this polymer can use as enteric coating material, then it will play an important role in the pharmaceutical sector.

ACKNOWLEDGEMENT

The research work was investigated in the Polymer Research Laboratory, Department of Applied Chemistry & Chemical Technology, and University of Rajshahi. I am also grateful to all respectable members of Central Science Laboratory University of Rajshahi for their cordial help and constant activities during analysis time.

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Received on 26-02-2015 Accepted on 01-03-2015 Published on 31-12-2014

DOI: http://dx.doi.org/10.12974/2311-8717.2014.02.02.4