RESEARCH ARTICLE

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Synthesis and Spectral Characterization of Bio-compatible Copolyester using 1, 4 Cyclohexane Diol

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This paper solely describes about novel aliphatic polymer prepared from polyethyleneglycol (PEG 400) moieties by melt poly condensation of 1, 4- cyclohexanediol and dodecanedioic acid. The structural elucidation of the polymer obtained, were thoroughly investigated by various techniques such as FTIR, NMR, DSC, TGA, XRD. The polycondensation reaction was carried out by the presence of Titanium (IV) isopropoxide as catalyst. We have also discussed about the biological properties like antioxidant and biocompatibility of the polymers with its non-toxic effect.

Introduction

India stands tall in the production of plastic wastes, in which tonnes and tonnes are littered and uncollected. The persistant increase in plastic usage pollutes the environment. These piled wastes created a need for biodegrable polymers which are easily recycled [**1,2**]. Today industries concentrate more on the manufacture of aliphatic biodegradable polymers due to its harmless by products. The polymers with ester groups can be used as a starting material to produce biodegradable and biocompatible polymers which are commercially used for internal and external consumption in the human body. Hence with this information, we come to a conclusion that aliphatic polyesters can be used as biodegradable material for various biomedical and industrial requirements. Poly (ethylene glycol) PEG is nontoxic, hydrophilic, and flexible polymer which is widely accepted and utilized as a biocompatible polymer. The hydroxyl groups available at both the ends provide chemically active sites to change the hydroxyls to other higher activity groups [**3,4**].

In this proposal, the synthesis of aliphatic polyesters were prepared from dodecanedioic acid, PEG 400, and 1, 4 cyclohexane diol by melt polycondensation technique using the catalyst titanium (IV) isopropoxide. The synthesized copolyesters are characterized by spectral studies and thermal analysis. The antioxidant behavior of the copolymer is determined by DPPH method. Further the biocompatibility studies are also discussed.

Experimental

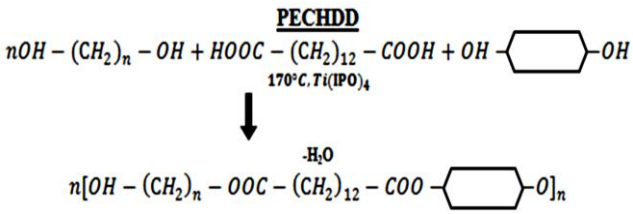
Materials used

PEG 400, dodecanedioic acid and 1, 4-Cyclo hexane diol.

Preparation of the polymer

Polyethyleneglycol dodecanate- co - 1, 4- cyclohexane dodecanate (PECHDD)

A round bottom flask (3 necked with provision for connecting nitrogen inlet and guard tube) need to be charged using 3.5 ml (0.01 mol) of PEG 400, 1.1616 g (0.01 mol) of 1, 4 – cyclohexane diol and 4.606.8 g (0.02 mol) of dodecanedioic acid. The reaction is initiated after nitrogen purging using the catalyst Ti (ipo)$_4$ (0.3ml/0.1mmol) to eliminate water molecule traces. The mixture is heated between 130°C and 140°C in the presence of an oil bath and the same is increased gradually till 170°C (10°C increased every 10 minutes during the process). The formation of methanol will be distilled during the process. Using acetone, the obtained copolymer (viscous slurry) ***Polyethyleneglycoldodecanate- co - 1, 4-cyclohexane dodecanate (PECHDD)*** is dissolved in ice cold methanol to form the precipitate. The process of filtering, washing and drying of precipitate are carried out at room temperature and low pressure [**5**].



Polymer characterization

The IR data was measured through FTIR spectro-photometer using KBr pellets. The proton nuclear magnetic resonance spectra were measured with the help of deuterated TFA (Trifluro acetic acid) at 25°C. The nuclear magnetic spectra was calculated using TMS (Tetra Methyl Silane) as standard to determine the chemical shifts. The Differential Scanning Calorimeter (DSC) study quantifies the thermal transitions of copolymers at inert atmosphere.

Antioxidant activity

DPPH scavenging method uses stable free radical to determine the antioxidant activity for synthesized compounds.

Biocompatible study

Biocompatibility test was carried out for the prepared compounds against human fresh blood with slight modifications [**6**].

Results and discussion

Spectral studies

Fig. 1 indicates the copolymers PECHDD's FTIR spectrum. The copolymers exhibit stable absorption band at 1736 cm-1, corresponding to the vibrations of C=O group [**7**].

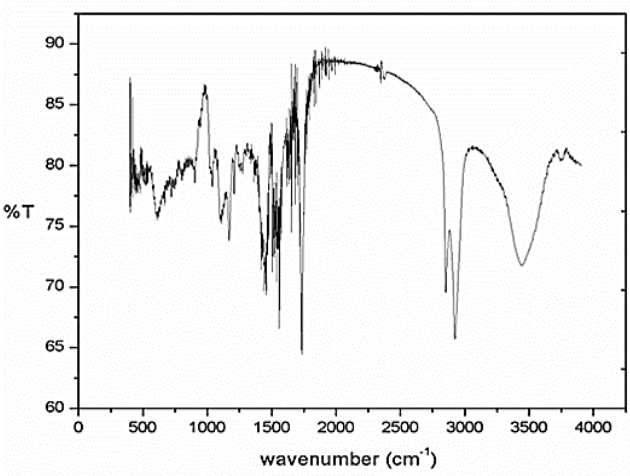


Fig. 1. FTIR Spectrum of PECHDD.

1H NMR spectral data of copolymer PECHDD

Fig. 2 shows the copolymer ^{1}H NMR spectrum. The peaks that exists between 1.73 ppm and 1.78 ppm indicates the presence of methylene protons of 1,4 – cyclohexane diol [**8**].

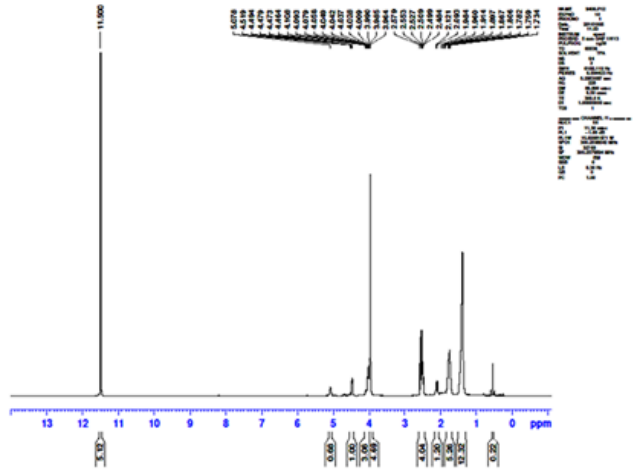


Fig. 2. 1H NMR spectrum of PECHDD.

Thermal analysis

Fig. 3 indicates that the glass transition temperatures (Tg) for PECHDD which falls at -25°C, which helps in possessing the chain flexibility property. The low transition temperature allows the copolymers to use effectively in drug release behavior [**9**].

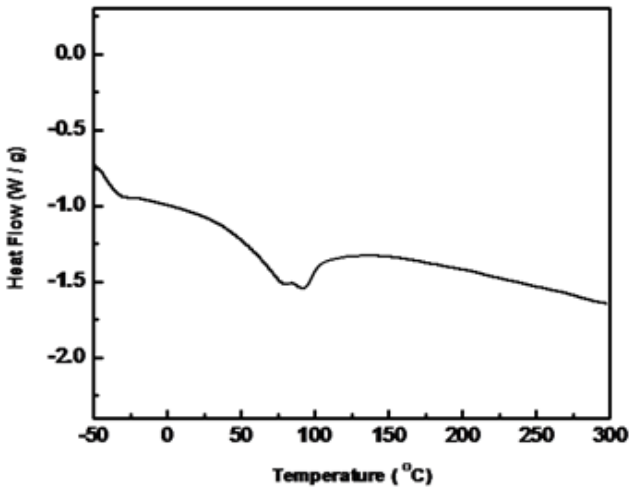


Fig. 3. DSC Spectrum of PECHDD.

Antioxidant results

Fig. 4 shows In vitro DPPH analysis to determine the antioxidant activity of PECHDD. The discoloration rate exposes the scavenging potential and indicates that hydrogen is donated to synthesized antioxidant copolymer and absorbance is decreased from the DPPH radical [**10**]. From the results of **Table 1**, it is significant that PECHDD has produced only moderate radical scavenging activity.

Table 1. *In vitro* DPPH Activities of synthesized compounds.

S.No	Conc. of Compounds (µg/mL)	PECHDD
1.	1000	71.19
2.	500	67.41
3.	250	59.12
4.	125	46.73
5.	62.5	39.56
6.	31.25	23.91
7.	15.62	10.20
8.	IC $_{50}$ (µg/mL)	211.43

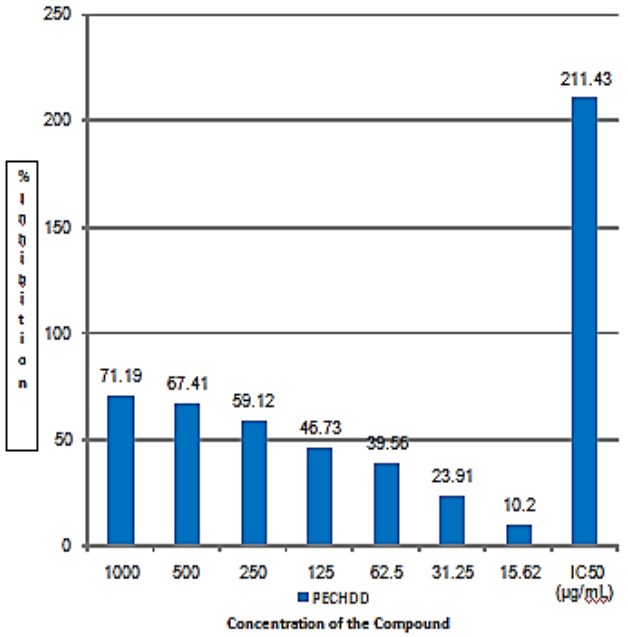


Fig. 4. *In vitro* DPPH analysis of PECHDD.

Biocompatible studies

Cellular Cytotoxicity Method is a basic biocompatibility test performed using human blood to indicate *In vivo* biodegradation. The results revealed that the copolymer (PECHDD) do not have any toxicity against the human blood solution displayed in **Table 2**. This proves that the copolymer can be used in both medical and pharmaceutical applications [**11**].

Table 2. Biocompatibility analysis of synthesized compounds on Red Blood Corpuscles (RBCs).

Concentration of compounds (µg/mL)	Incubation Periods	Name of the compound (PECHDD)
250	15 min 30 min & 60 min	No toxicity
500		No toxicity
1000		No toxicity

Summary

The direct melt polycondensation method was used to synthesize Polyethylene glycol dodecanate-co-1, 4-cyclohexane dodecanate a biocompatible copolyester) by applying titanium tetra-isopropoxide as catalyst. The dissolvability characteristics of the solvents decide the copolyester application. Using the NMR and IR spectral data, the structure of the synthesized copolymers were confirmed. The Differential Scanning Calorimetric (DSC) study exhibits low glass transition temperature which induce the polymeric chain flexibility property. The antioxidant study reveals less activity for the synthesized copolymers. Further the biocompatible studies confirm that the copolymers were found to be non-toxic among human blood solutions.

Keywords

Polycondensation, biocompatible and antioxidant.

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