Journal of Material Science and Mechanical Engineering (JMSME)
Print ISSN: 2393-9095; Online ISSN: 2393-9109; Volume 2, Number 8; April-June, 2015 pp. 26-28

http://www.krishisanskriti.org/jmsme.html

Polymeric-composite Films from Polycaprolactone with Waste Poly (Ethylene Terephthalate) Monomers for Industrial Applications

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Abstract—*New polymeric composite films were synthesized by blending, via solvent evaporation method, biodegradable polycaprolactone and terephthalic acid. Terephthalic acid was synthesized from waste poly(ethylene terephthalate) and it was characterized by fourier transform infrared, thermogravimetric analysis, and differential scanning calorimetry. The resulted terephthalic was used to develop the polymeric composite film. The polymeric composite films developed will be useful in packaging and other (biomedical, light weight polymeric materials for energy) applications.*

Keywords*: Terephthallic acid, waste poly(ethylene terephthalate), biodegradable polycaprolactone, composite films.*

1. INTRODUCTION

Polymers utilization has its attraction in several fields, due to their significant characteristics, such as: lightweight, low cost than metals and easy design by customers to desired shape etc [1]. Theses specific properties make them attractive for use in the fields of packaging and bottling, etc. However, day-by-day, polymer consumption is on the increase in the humans' daily life [2]. At the same time, polymers wastes constitute very serious problem to the environment. In order to solve this problem, many researchers have been working on how to recycle waste polymers and their re-applicability with suitable polymers in technical applications [3].

The present work involves the synthesis of terephthalic acid form waste poly(ethylene terephthalate) oil bottles and the development of polymeric-composite films from polycaprolactone with terephthalic acid. The resulting monomer and films were characterized with fourier transform infrared spectroscopy, thermogravimatric analysis and differential scanning calorimeter.

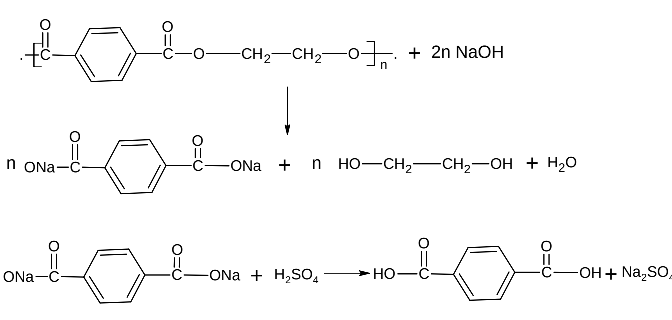
2. EXPERIMENTAL SECTION

2.1 Materials

Waste poly(ethylene terephthalate) (PET) flakes were obtained from waste oil bottles in Concepcion, Chile. Polycaprolactone, ethylene glycol, sodium hydroxide chemicals were purchased from Sigma-Aldrich, Inc. and used without further purification.

2.2. Terephtalic acid synthesis from waste PET oil bottles

Temperature technique was used for synthesis of terephthalic acid. In this process, waste PET from the oil bottles was cut into small pieces and thoroughly washed with soap water and distilled water. The clean PET pieces was dried in an oven for 48 h at 80^{o}C



Scheme 1: Terephtalic acid synthesis from PET

For the synthesis of terephthalic acid (Scheme1), 2.7 g of NaOH was dissolved in 12 g of ethylene glycol at 70^{o}C for the dissolution of NaOH in order to achieve aqueous solution. After that, 5 g of waste PET was added to the aqueous solution in 250 mL of beaker and the temperature was slowly increased to 200±2^{o}C for 10 min in order to completely dissolve the PET in the ethylene glycol, which is continuously stirred with a steel rod. To this solution, 100 mL of distilled water was added slowly, then the solution was cooled at room temperature and then H_2SO_4 was add drop-wise to this solution for the formation of a white precipitate at a of pH 2.4. The resulting precipitate was washed, several times, with distilled water in order to remove the (salt and ethylene glycol) unwanted elements. Finally, it was filtered and dried at room temperature

2.3. Preparation of composite films

Polymeric composite films were prepared by solvent evaporation method, using biodegradable polycaprolactone and terephthalic acid that was obtained from the above process.

2.4. Characterizations

FTIR spectra of the PET waste and the terepthalic acid were obtained from a Perkin Elmer, UATR two, FTIR spectrometer (Beaconsfiled, Bucks, UK) in the wavelength range of 4000-500 cm^{-1}. Signal averages were obtained from 25 scans at a resolution of 1 cm^{-1}. Thermal characteristics were determined from the thermogravimetric analysis (TGA) data, using the TGA Q 50 thermal analyzer (T.A. instruments-water LLC, Newcastle, DE, USA), at a heating rate of 10°C/min and passing nitrogen gas at a flow rate of 100 ml/min. DSC thermograms of waste PET and terephthalic acid (TA) were recorded using a DSC 882e (Mettler Toledo) instrument at a heating rate of 10°C/min under a constant nitrogen flow (100 ml/min) in the temperature range of 40-300°C

3. RESULTS AND DISCUSSION

The selected poly(ethylene terephthalate) waste of oil bottles was depolymerized by temperature (heating) technique. Form this technique, therephtalic acid was obtained and characterized using the FTIR, TGA and DSC.

The FTIR in Fig. 1 shows that the therephtalic acid obtained. The characteristic peaks at 2551-3200, 1679 and 1574-1425 cm^{-1} are related to –COOH, -C=O and aromatic ring, respectively. The peaks in the region 1281-1000 cm^{-1} indicate the –C-OH, -C=O, -C-CH, -C-H groups that are present in the terephthalic acid [4]. The band at 700 to 800 cm^{-1} is indicative of the aromatic ring of the terephthalic acid [5].

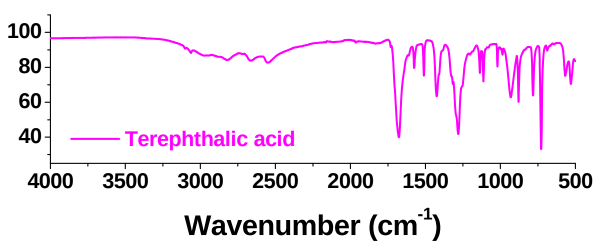


Fig. 1: FTIR spectra of Terephthalic acid

The thermal behaviour of the monomer is usually investigated by the TGA and DSC. The terephthalic acid TGA curves are shown in Fig. 2. The initial decomposition temperature of terephthalic acid occurs at 246°C. The second decomposition occurs at around 400°C. This is due to the evaporation of few organic and interlayer water, dihydroxylation and the decomposition of terephthalate, respectively.

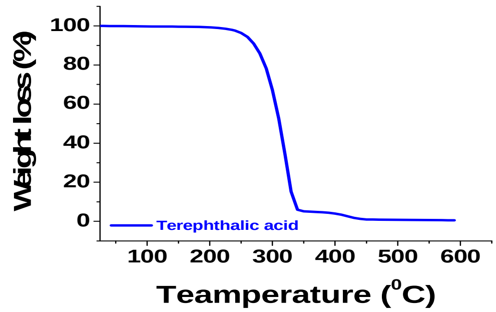


Fig. 2: TGA curves of the terephthalic acid

The DSC curve of terepthalic acid sample is illustrated in Fig. 3. The PET exhibits its melting point at 258 °C where as TA cannot exhibit any peak in the range of 25 to 300 °C [6].

The polycaprolactone film and polycaprolactone-terepthalic acid films (Fig. 4) were obtained via solvent evaporation method. Fig. 5 shows the Fourier transform infrared spectroscopy spectra of polycaprolactone and polycaprolactone-terephthalic acid films. The polycaprolactone shows peaks at 3341 and 1716 cm^{-1}, which indicates the hydroxyl and ester groups, respectively.

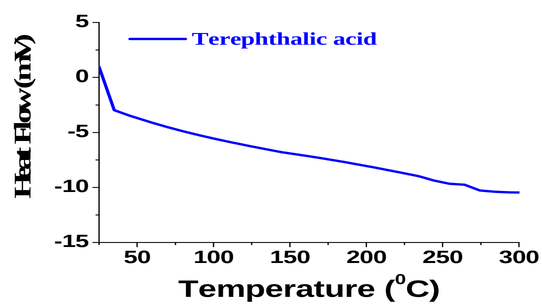


Fig. 3: DSC curves of terepthalic acid

Fig. 4: Polycaprolactone/terephthalic acid composite film

On the other hand, in the case of polycaprolactone-terepthalic acid films, they peaks are shifted to 3423 and 1726 cm^{-1} and the new peaks are observed at 1574, 1430 and 787 cm^{-1}, corresponding to the aromatic ring of terepthalic acid, which confirms clearly a composite that have functional groups of terepthalic acid and polycaprolactone.

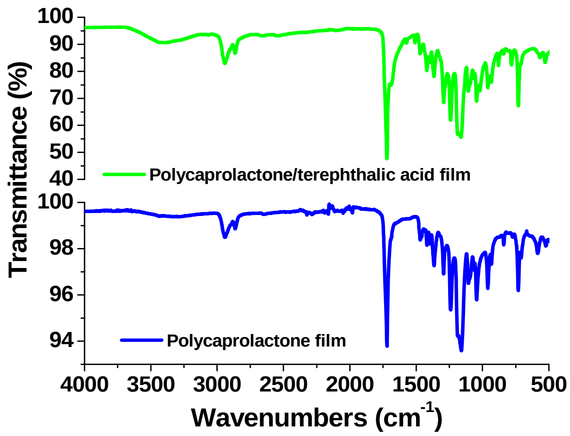


Fig. 5: FT-IR of polycaprolactone/terephthalic acid composite and polycaprolactone film

The biodegradable polycaprolactone/terephthalic acid composite films developed are transparent and they are suitable potential candidates for industrial packaging applications.

4. ACKNOWLEDGEMENTS

The author, Kokkarachedu Varaprasad, wishes to acknowledge the CIPA, CONICYT Regional, GORE BIO-BIO R08C1002 and PAI proyecto 78130211 Conicyt, Chile.

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