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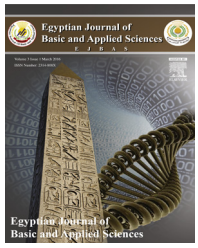

Full Length Article

Spectroscopic studies and thermal properties of PCL/PMMA biopolymer blend

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ABSTRACT

Polycaprolactone/Polymethylmethacrylate (PCL/PMMA) biopolymer blend can be prepared by casting technique. Structural, optical and thermal properties of the blend have been studied using X-ray diffraction (XRD), infrared spectroscopy (FTIR), ultraviolet-visible spectroscopy (UV-Vis) and thermogravimetric analysis (TGA). XRD show two diffraction peaks at $2\theta = 21.4°$ and 23.8° which attributed to the planes (110) and (200) that represent crystallographic planes of semi-crystalline PCL, respectively, where PMMA revealed a broad amorphous hump observed around $2\theta = 15°$. The FTIR spectra showed some variations in the position and intensity of some absorption bands which reveal an interaction and good miscibility between the two polymers. TGA suggested that the thermal stability increases with increasing PCL concentration; this indicates the incorporation of PCL into the host. Approximately the pure PCL mass loss remains constant until complete decomposition occurs at about 430 °C, whereas PMMA complete decomposition occurs at about 400 °C. So, decomposition temperature of PCL is higher than PMMA by nearly 30 °C.

1. Introduction

Blending between two or more polymers can modify the structural and physical properties of polymers to specific requirements. So, the attention of material researchers has been attracted to polymers blend [1–4]. It involves physical mixing of biopolymers, leading to creation of a new material having some desirable properties that are superior to any one of the component polymers [5–7]. Manifestation of these properties depends on the miscibility of homo-polymers at the molecular scale, so that miscibility leads to variation of the blend's morphology, ranging from a single phase system to a two phase or multiphase systems [6]. The basis of biopolymer miscibility may arise from several different interactions, like charge transfer complexes for homo-polymer mixtures, hydrogen bonding and dipole–dipole forces [5]. Polycaprolactone (PCL) is a semi-crystalline aliphatic polymer with the chemical formula $(C_6H_{10}O_2)_n$. It has a low melting point (60°C) and glass transition temperature equals –60 °C. PCL also have suitable properties such as good biocompatibility, good biodegradability, good mechanical strength and remarkable toughness. Owing to its

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hydrophobic nature PCL has a good solubility in chloroform and tetrahydrofuran (THF). As the molecular weight increases, PCL crystallinity tends to decrease [8–11]. Blend compatibility has made PCL a continuous research focus in the biomedical application. Polymethylmethacrylate (PMMA) is a linear thermoplastic polymer with the chemical formula $(C_5H_8O_2)_n$. It has a melting point equal to 160 °C and a glass transition temperature of 85 °C. PMMA has been widely used as a biomaterial in medical applications and in some optical systems (manufacturing of contact lens as transmit light up to 93%) [12]. It also can be used as denture base material due to its favorable properties, including ease of handling and repair, as well stability in the oral environment [13]. PCL is selected as one of the blend components in view of its miscibility with PMMA to introduce a modern category of biopolymer blends with a simple method of preparation suitable for being used in some biological application. This work aims to investigate both compatibility and phase behavior of PCL/PMMA bioblends to obtain a critical concentration with the best overall good properties to use later with some addition of organic or inorganic materials for some application.

2. Experiment and method

2.1. Materials

The chemicals used were Poly ε-caprolactone pellets (M_w~80,000, Sigma-Aldrich and Lot No. MKBP7389V), polymethylmethacrylate (M_w~120,000, Sigma-Aldrich and Lot No. MKBB7676) and chloroform solution as a solvent (HPLC grade, Fisher Chemical and Lot No. 1229720).

2.2. Sample preparation

Polycaprolactone/Polymethylmethacrylate bioblends prepared via casting technique and mixtures of the PCL/PMMA blend films were dissolved in a glass beaker by chloroform using a magnetic stirrer for 45 minutes until complete miscibility [14] occurred and placed in an 8-cm diameter Petri dish (The Petri dishes were cleaned with chloroform and dried in an oven at 60 °C for 20 minutes). After evaporation of the solvent, bioblend films were kept to dry at room temperature for one day.

2.3. Physical measurements

The X-ray diffraction (XRD) scans were obtained using PANalytical X'Pert PRO XRD system using the radiation of Cu K_α X-ray where the tube operated at 30 kV, Bragg's angle 2θ in the range of 3–60°, λ = 1.54 Å. FTIR absorption spectra were achieved utilizing the single beam Fourier transform-infrared spectrometer (FTIR-Nicolet is10). FTIR spectra of the samples were obtained in the spectral range of 4000–500 cm^{-1}. UV-Vis absorption spectra were measured in the wavelength region of 200–600 nm using spectrophotometer (T80+, UV/Vis. spectrometer, PG Instrument Ltd.) to retrace the structural changes due to different blend concentrations and their optical properties. A Perkin–Elmer (US, Norwalk, CT) TGA-7 was used for the thermogravimetric analysis of the samples. For the data analysis a small amount of (mg) sample was taken and the samples were heated at room temperature of 500 °C at a rate of 10 °C/min in nitrogen atmosphere.

3. Results and discussions

3.1. X-ray diffraction analysis

The XRD diffraction scans of the biopolymer blend are also used as a criterion to determine its overlap and homogeneity. If two biopolymers have low biocompatibility and are immiscible, then each biopolymer would have its own crystal region in the blend films. Fig. 1a shows two characteristic peaks at angles 2θ = 21.4° and 23.8°, corresponding to the (110) and (200) crystallographic planes of semi-crystalline nature of PCL biopolymer, respectively [15]. The observed scans of pure PMMA as shown in Fig. 1a exhibits a characteristic broad amorphous hump observed around 2θ = 15°, 30.2° and also a weak hump observed at about 2θ = 42.2° [16,17].

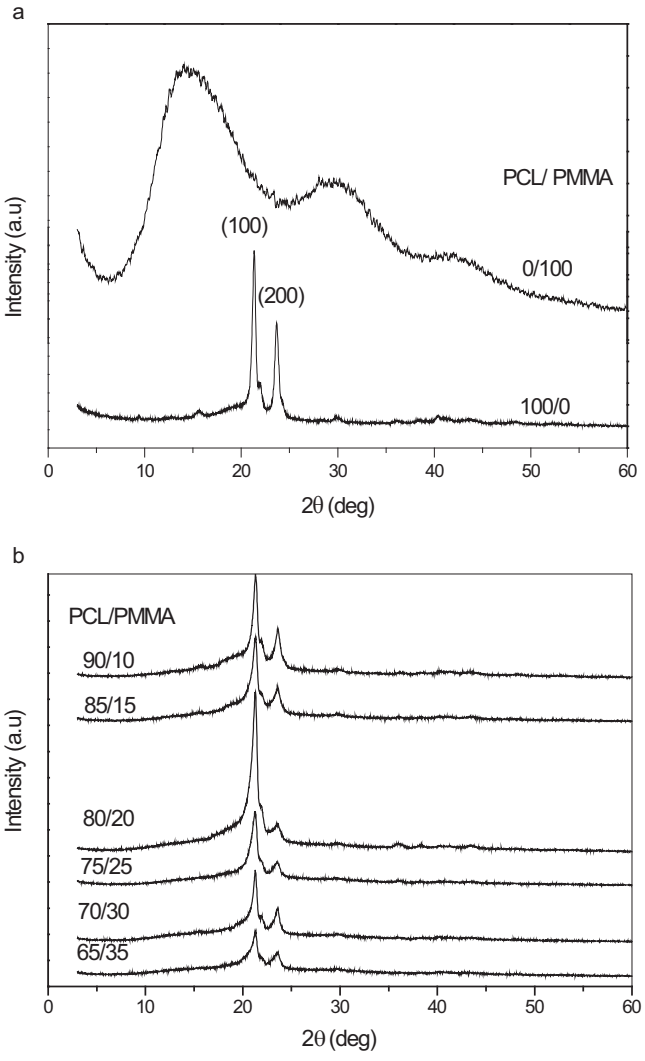

Fig. 1 – (a) X-ray diffraction scans of pure PMMA and pure PCL. (b) X-ray diffraction scans of PCL/PMMA blend films.