



Mechanical, crystallisation and moisture absorption properties of melt drawn polylactic acid fibres

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ABSTRACT

Poly(lactic acid) (PLA) fibres were produced with average diameter ranging from 11 to 38 μm via a melt drawing process employing increasing take-up velocities. The PLA fibres exhibited smooth surfaces and uniformity in diameter as determined by scanning electron microscopy (SEM) and optical microscopy (OM). Fourier Transform Infrared Spectroscopic (FTIR) analysis using the dichroic ratio demonstrated alignment of PLA chains with the draw direction, where the lower diameter PLA fibres exhibited a higher degree of chain orientation during the high speed melt drawing process. The crystallinity of the fibres also increased up to 34% with decreasing fibre diameter due to strain-induced crystallisation. The room temperature tensile strength and modulus of the smaller PLA fibres with an average diameter of 11 μm revealed values of 213 MPa and 4.8 GPa, respectively. These fibres revealed a significant decrease in their tensile strength (by 29%) when tested at 37 °C compared to the room temperature value. Comparatively larger diameter PLA fibres did not show any significant change in their mechanical properties at 37 °C. The variation in diameter of PLA fibres also revealed a noticeable influence in moisture absorption at various humidity levels believed to be due to the effect of crystallinity on water absorption.

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1. Introduction

Poly(lactic acid) (PLA) is widely used in the field of tissue engineering for biomedical applications such as for surgical sutures, implants, scaffolds, fracture fixation devices and sustained release polymers for drug delivery systems [1–6] due to its biocompatibility, biodegradability, superior transparency and suitable thermal and mechanical properties [7,8]. PLA can be produced via ring opening polymerisation of the lactide monomer which can be derived from

renewable sources such as corn, potato starch and molasses [9]. As part of its appeal, thermoplastic PLA can be formed into various architectural forms including films [5,10], plates [11], scaffolds [12,13], fibres [14], screws [15] and rods [16]. It can also be processed *via* solvent casting or by melt compounding processes (such as hot compression, injection and extrusion moulding) [7,9] and can be produced into fibrous structures via melt spinning and/or electrospinning with distinct fibre properties [14,17–19]. For example, melt spinning is employed to produce microfibrils whilst the electrospinning process has been used for nanofibre to submicron fibre production.

Properties of melt drawn PLA fibres produced using different processing conditions with varying L-lactide to D-lactide ratios have been reported in the literature [17,20–25]. Kim et al. [25] produced melt spun PLA fibres

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with a 1–9 mol% D-lactide content and investigated the mechanical, thermal and crystallisation properties of uniaxial elongated PLA monofilaments at varying draw ratios (DR), from 1 to 8, in the temperature range between 25 °C and 65 °C. They reported that the tensile strength and modulus of control PLA monofilaments (4.2 mol% D-lactide content) decreased from 71 MPa to 18 MPa and 2.4 GPa to almost zero, respectively, with increasing temperature due to chain mobility of the polymer in the glass transition region. The glass transition (T_g) temperature of PLA fibre drawn at higher ratios (DR = 8) was seen to shift to 68 °C, which was approximately 12 °C higher than the undrawn fibre, suggesting a higher degree of chain orientation due to strain induced crystallisation during the fibre drawing process. Chain orientation ascertained in terms of inverse dichroic ratio (measured using polarised Raman spectroscopy) within the PLA fibres was also reported to increase with an increase in the draw ratio of the spun fibres.

Okuzaki et al. [26] investigated melt spun PLLA fibres (processed at 185 °C) with a 394 μm diameter and reported that further drawing (DR = 10.5) of the as-spun fibre at 90 °C increased the crystallinity of the PLLA fibre by 30% when compared to undrawn fibres (crystallinity 7.4%). In addition, drawing the fibres (DR = 10.5) increased the tensile strength from 16 MPa to 275 MPa, whilst the modulus increased from 1.8 GPa to 9.1 GPa. They further suggested that the improved properties were due to the increased orientation factor of the polymer chains within the fibre as a direct result of drawing at 90 °C. PLLA fibres with diameters ranging from 11 to 14 μm obtained at 5000 m min^{-1} take up velocity and at 233 °C [23] were shown to display a maximum crystallinity, tensile strength and modulus of 42%, 385 MPa and 6 GPa, respectively.

Another useful property of PLA is that it can be hydrolysed by water and as such, its degradation is accelerated in a moist environment and by the presence of water during its service life. The moisture absorption capacity of the fibre depends on the nature of the PLA, such as hydrophilicity (ester groups), diffusion coefficient, thickness and the surrounding environment. *In vitro* degradation of PLLA fibres with varying diameter ranging from 113 to 148 μm in phosphate buffered saline (PBS) media at 37 °C was investigated by Yuan et al. [27]. They reported that the tensile moduli and ultimate strength of the fibres was not significantly altered during 35 weeks of degradation except for PLLA fibres with the smaller diameter of 113 μm . These smaller fibres exhibited a significant decrease in ultimate strength by 20% with respect to their initial value (500 MPa). The authors suggested that formation of micro-cracks on the degraded fibre surface resulted in the decrease of their mechanical properties.

The PLA fibres investigated in the literature as reported above [23–26] were produced via a melt drawn process using an extruder to feed them onto the winder drum and the fibre properties were further improved using cold and/or hot draw processes [25,26]. The PLA fibres investigated in this study were manufactured using a gravity induced melt drawing process employing various take up velocities up to 400 m min^{-1} . The aim was to improve their crystallinity and chain orientation properties during the fibre production process while avoiding the need for a

further cold drawing process. This would reduce required processing steps whilst still increasing mechanical properties. The morphology, chain orientation, thermal, crystallisation and moisture absorption properties of the PLA fibres produced at various take-up velocities were characterised. The effect of temperature (37 °C) and moisture absorption at various humidity levels on the mechanical properties of the PLA fibres are also reported.

2. Materials and methodology

2.1. Manufacturing process of PLA fibres

PLA fibres were produced via a melt spinning process. PLA beads (NatureWorks LLC, Ingeo™ Grade 3251D, average $M_w \sim 90,000\text{--}120,000 \text{ g mol}^{-1}$, density = 1.24 g cm^3) dried at 50 °C for 48 h were melted at 180 °C in air using a cylindrical steel mould fitted with a band heater. Polymer was allowed to exit through a base hole (2 mm) under gravity. The molten polymer was collected on a rotating drum (with a 1 m circumference using a collector distance of approximately 50 cm) utilising various take-up velocities (of 100, 200, 300 and 400 m min^{-1}). The PLA fibres produced were coded as PLA 11, PLA 20, PLA 29 and PLA 38 with the number indicating the average fibre diameter in μm .

2.2. Scanning electron microscopy (SEM)

The surface morphology of the PLA fibres was characterised using scanning electron microscopy (SEM – Philips XL30, FEI, USA) operated at an accelerating voltage of 10 kV and a working distance of 10 mm. A sputtered coating of platinum was used to avoid image distortion due to charging.

2.3. Optical microscopy (OM)

Fibre diameters were measured using a calibrated optical microscope (20 \times magnifications) and Image Pro Plus software was used to measure the diameter of the fibres in microns. The average fibre diameter is presented and the errors were calculated from the measurement of at least 35 random fibres obtained utilising the varying take-up velocities.

2.4. FTIR-ATR analysis and chain orientation

Identification of the functional groups of the PLA fibres were analysed using FTIR spectroscopy (Tensor-27, Bruker) and the spectra were collected with a resolution of 4 cm^{-1} by averaging 32 scans using a standard pike attenuated total reflectance (ATR) cell (Pike Technology, UK). To investigate the chain orientation of the PLA fibres with varying diameters via dichroic ratio calculation, two different FTIR-ATR spectra for each type of PLA fibre were taken by placing the fibres in the machine in the (parallel) and subsequently transverse (perpendicular) direction to the FTIR beam source as described elsewhere in the literature [25].

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