Enhancing the Mechanical and Molecular Properties of Polycaprolactone Nanofibers with Heat-Setting

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Introduction: Electrospun polycaprolactone (PCL) nanofibers show promise in a wide range of biomedical application due to their strength and biocompatibility. Typically, tuning the nanofiber material properties is done at the electrospinning stage. However, this does not allow for fine control of the rate and degree of crystallization of the fibers, which limits the mechanical properties that can be achieved. Here we demonstrate that heat-setting and quenching in water increases the percent crystallinity, molecular alignment, and mechanical properties in distinct, but predictable ways.

Materials and Methods: Electrospun PCL nanofibers were collected on an automated parallel track collection and processing system and drawn to a draw ratio (DR) of 3. Samples were incubated in water baths ranging from 22 to 75 °C for various lengths of time before being transferred to a room temperature quenching bath. Molecular alignment and crystallinity were assessed with FTIR and XRD. Fiber alignment, diameter, and morphology were determined with SEM. Mechanical properties such as ultimate strength, modulus of elasticity, elongation to failure, and toughness were calculated from tensile tests.

Results: Alignment, crystallinity, and strength were all affected by heat-setting. Alignment increased for temperatures up until 50 °C, while crystallinity peaked at 70 °C (Figure 1), and ultimate tensile strength peaked at 55 °C. Isothermal crystallization kinetics were determined for 70 °C, which showed that the bulk of crystallization occurs within in 30 seconds. There was a strong relationship between crystallinity and alignment ($p < 5.0e\text{-}5$), which demonstrates the tradeoff of alignment for crystallinity at higher incubation temperatures. The 55 °C incubation increased the ultimate tensile strength of the fiber by 25% compared to the room temperature treatment. Young's modulus was affected, but to a much smaller degree, and the fibers became more brittle, demonstrating a decrease in toughness and elongation at failure.

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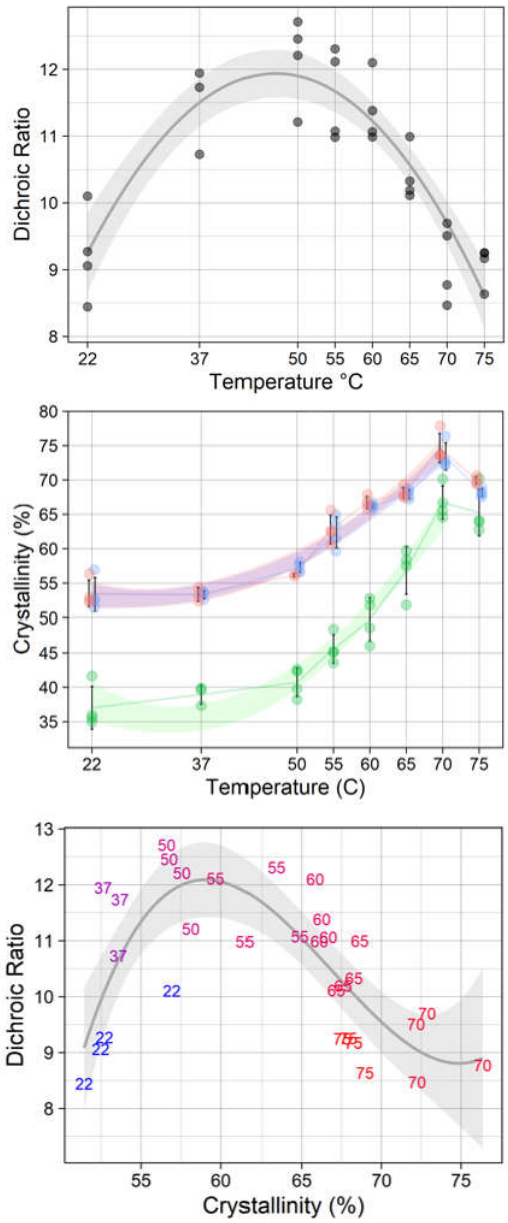

Figure 1: The effects of temperature on crystallinity and dichroic ratio. a) Dichroic ratio follows an inverted – U curve as a function of temperature ($p < 5.0e\text{-}10$, $R^2 = 0.7885$), with a maximum at 50 °C. b) Crystallinity increased for both orientations and overall ($p < 5.0e\text{-}13$, $R^2 > 0.92$ for all). The perpendicular orientation underwent the greatest increase in crystallinity. c) Dichroic ratio and crystallinity are also related by an inverted – U curve ($p < 5.0e\text{-}5$, $R^2 = 0.523$), with temperatures ranging from 55 °C to 65 °C striking a balance between DR and crystallinity.