Solventless High Throughput Manufacturing of Poly(butylene terephthalate) Nanofibers

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Supplementary Information

Experimental Details

1.1 Scanning Electron Microscopy (SEM)

A thin layer of fibers was attached to the SEM sample holder with double sided carbon tape and sputter coated with Au/Pd to minimize charging. The coated samples were imaged by Hitachi S-4500 SEM at an accelerating voltage of 15 kV. Several low and high magnification images were taken from different areas of the sample. Fiber diameters of 200 to 250 fibers were measured from 15 to 20 SEM randomly sampled images using image analysis software (Image J, NIH). The average diameter and standard deviation of the fibers were reported and a histogram was constructed for each fiber sample.

1.2 Differential Scanning Calorimetry

Melting and crystallization behavior of the fiber samples was investigated by differential scanning calorimetry (DSC-1, Mettler Toledo). About 2 mg of fiber sample was used for each run and two runs were conducted at each condition to ensure data reproducibility. The sample was held at 25 ˚C for two minutes, and then heated to 300˚C at a rate of 10˚C/min. The sample

was held at 300 ˚C for two minutes before cooling down to 25˚C at a rate of 10˚C/min. The sample was again held at 25 ˚C for two minutes and heated to 300˚C at a rate of 10˚C/min. The peak values of the heating and cooling scans were taken as the melting and crystallization temperature, respectively. Percentage crystallinity was estimated from the area of melting endotherm using 140 J/g as the heat of fusion for 100% crystalline PBT.

1.3 Rheometry

An AR2000ex rheometer (TA instruments) was used to study the effect of temperature on the rheological properties of the bulk material. Nitrogen gas was used at all times to avoid sample degradation. After conducting a strain sweep experiment to determine the linear viscoelastic region, oscillatory shear experiments were conducted on PBT resin at 10% strain with frequency ranging from 0.1Hz to 100 Hz. Experiments were conducted at multiple temperatures (260, 280, 300 and 320 ˚C) using 25 mm diameter steel parallel plates.

1.4 Optical Microscopy

Molecular orientation in fibers was analyzed using an optical microscope (Olympus BX 60) with crossed polarizers. Fibers sandwiched between a glass slide and a cover slip that was placed on a hot stage and the birefringence behavior imaged at different temperatures with an associated Nikon camera.

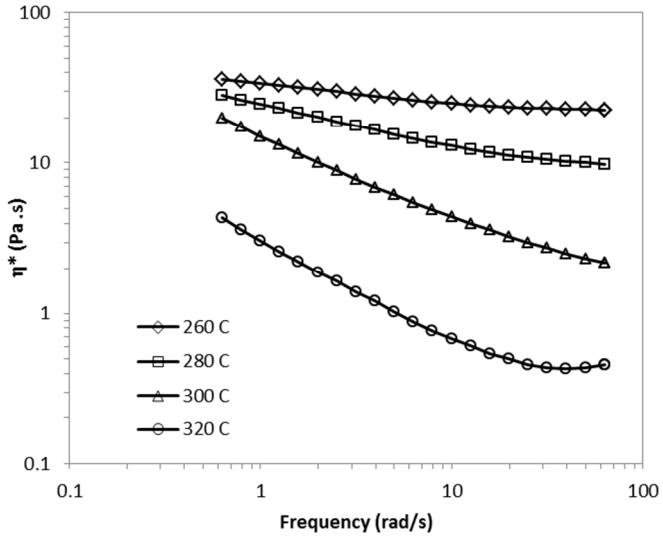

Figure S1: Complex viscosity of PBT resin at different temperatures as a function of frequency of oscillation.

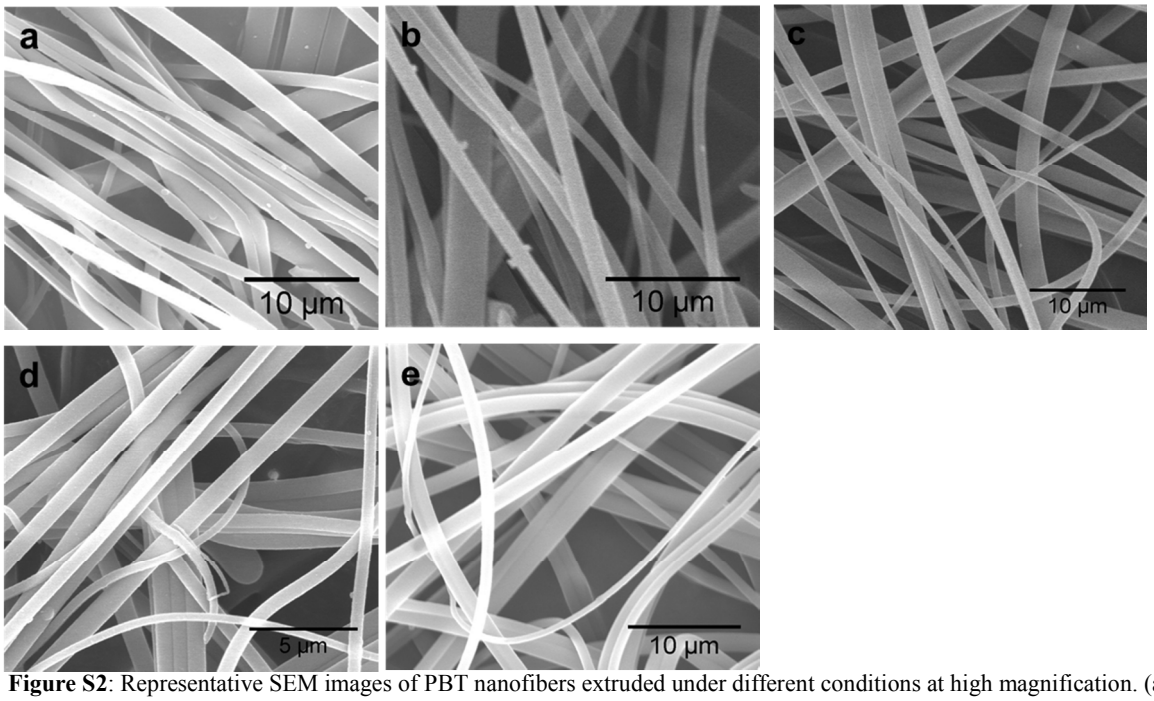

Figure S2: Representative SEM images of PBT nanofibers extruded under different conditions at high magnification. (a) 10000 rpm at 300 °C (b) 12000 rpm at 300 °C (c) 15000 rpm at 300 °C (d) 12000 rpm at 320 °C (e) 12000 rpm at 280 °C.

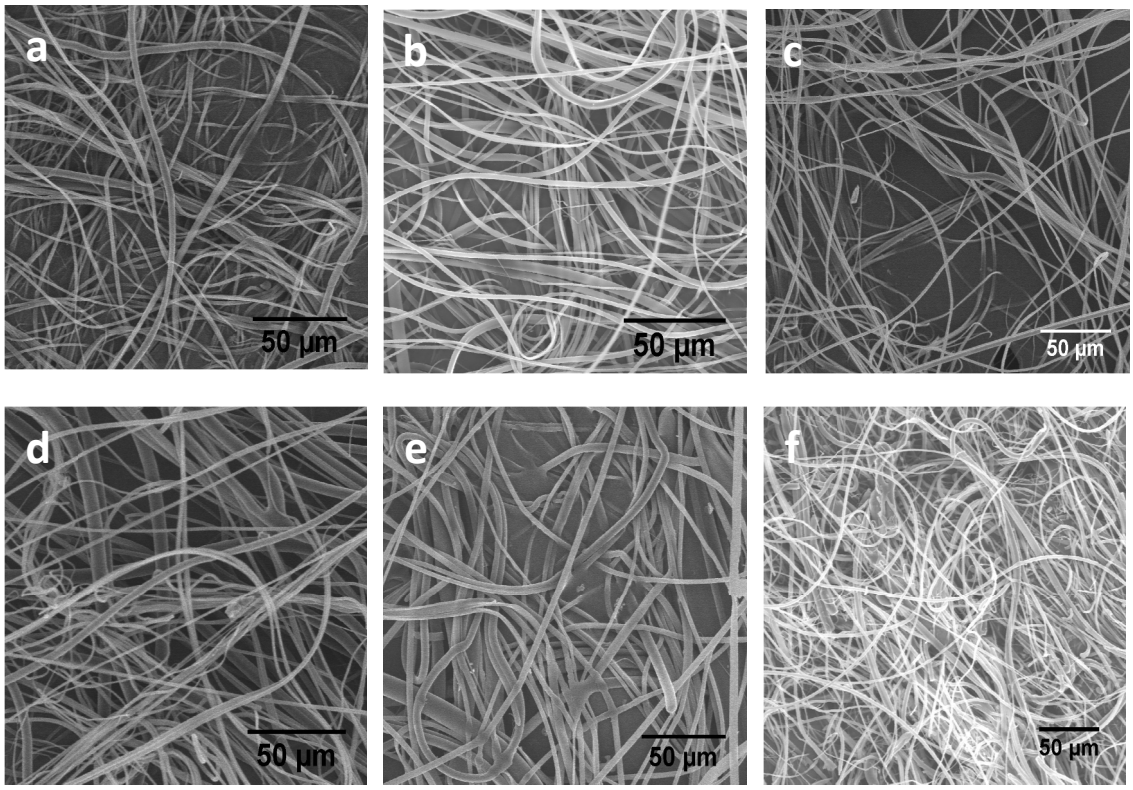

Figure S3: Representative SEM images of PBT nanofibers extruded under different conditions at low magnification. (a) 10000 rpm at 300 °C (b) 12000 rpm at 300 °C (c) 15000 rpm at 300 °C (d) 12000 rpm at 320 °C (e) 12000 rpm at 280 °C (f) fibers made at 12000 rpm at 300 °C after immersing in hot toluene at 60 °C for 24 hours.