Supplementary Material (ESI) for Journal of Materials Chemistry

Supplementary Information

Fabrication of thermoset polymer nanofibers by co-electrospinning of uniform core-shell structure

C. S. Reddy, A. Arinstein, R. Avrahami, E. Zussman

Experimental

Polycaprolactone (M_n = 80,000), chloroform, dimethylsulfoxide, bisphenol A ethoxylate dimethacrylate (*BPAEDMA*, *M_n = 1700*), *2, 2-Dimethoxy-2-phenylacetophenone and 2, 2, 2-trifluoroethanol* were purchased from Aldrich and used without further purification. Polycaprolactone was dissolved in 9:1 (wt./wt.) ratio of chloroform and dimethylsulfoxide to obtain 9 wt.% concentrated solution, used as shell component (shear viscosity 600cP). As a core component, 98 wt. % BPAEDMA and 2 wt. % *2, 2-Dimethoxy-2-phenylacetophenone dissolved in BPAEDMA was used* (shear viscosity 450cP): All experiments were conducted at room temperature and a relative humidity of $50-60\%$.

To produce the core-shell fibers, we used the coaxial electrospinning process. The core capillary was a stainless-steel needle with inner and outer diameters of 0.42 and 0.64 mm, respectively. The shell capillary was a plastic needle with inner and outer diameters of 1.2 and 1.75 mm, respectively. The tip of the core capillary protruded 0.3 to 0.5 mm below that of the shell capillary.

The shell component was also electrospun to obtain PCL shell fiber mat. The core component was placed in between two microscope galls slides adjusted to 1 cm height and exposed to UV radiation to obtain a BPAEDMA film.

The spinning parameters were as follows: shell flow rate was constant at 4 mL/h, the core flow rate was maintained at different rates (*0.5, 0.02, 0.01* mL/h), electrostatic field of about 10.5

kV, distance between the spinneret and the collector wheel was 11 cm. The wheel had a tangent velocity of 600 m/s. The flow rates of both the core and shell solutions were controlled by two syringe pumps. The aligned core-shell fiber mats were placed on aluminum foil supported on a metal disc at a distance of 15 cm from two horizontal 20W high pressure mercury lamps and UV polymerized for 10 min.

The shell from the UV polymerized bulk fiber mat and the individual fibers collected on microscope glass slide and UV polymerized was removed by dissolution in trifluoroethanol (TFE) for 30 min.

For the analysis of fiber morphology the cross-sectioning of the fiber mat was done in liquid nitrogen and the cross-section of the fiber mat was imaged with the help of a Leo Gemini high-resolution scanning electron microscope (HRSEM) at an acceleration voltage of 3 kV and a sample to detector distance of $3-5$ mm. The specimens were coated with a thin gold film to increase their conductivity.

Infrared spectroscopy experiments were done on the fiber mats and film using a NEXUS 870 FTIR (Thermo Nicolet) in ATR mode in a humidity free atmosphere, at room temperature, with 4 cm^{-1} resolution and 32 scan signal average. Spectra were taken from 600 to 4000 cm^{-1} in the absorbance mode using a DTG TEC detector. The IR data are presented in this paper without any correction or modification Dynamic mechanical analysis was carried out with a TA Instruments DMA-Q800 dynamic mechanical analyzer (New Castle, Delaware, USA). The instrument was used in the tension film mode. The samples were subjected to a sinusoidal displacement at $10\,\mu$m amplitude at a fixed frequency of 1 Hz from -80 to 40°C at a heating rate of 2°C/min. The storage modulus, loss modulus and loss tangent were measured on fiber mats before and after UV polymerization and on the cured film in this temperature range.