Complementary characterization data in support of uniaxially aligned electrospun nanocomposites based on a model PVOH-epoxy system

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A B S T R A C T

This paper presents complementary data corresponding to characterization tests done for our research article entitled “Uniaxially aligned electrospun fibers for advanced nanocomposites based on a model PVOH-epoxy system” (Karimi et al., 2016) [1]. Poly(vinyl alcohol) and epoxy resin were selected as a model system and the effect of electrospun fiber loading on polymer properties was examined in conjunction with two manufacturing methods. A novel electrospinning technology for production of uniaxially aligned nanofiber arrays was used. A conventional wet lay-up fabrication method is compared against a novel, hybrid electrospinning–electrospraying approach. The structure and thermomechanical properties of resulting composite materials were examined using scanning electron microscopy, dynamic
mechanical analysis, thermogravimetric analysis, differential scanning calorimetry, Fourier transform infrared spectroscopy, and tensile testing. For discussion of obtained results please refer to the research paper (Karimi et al., 2016) [1].

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**Specifications Table**

| Subject area | Engineering, material science, nanotechnology |
|--------------|---------------------------------------------|
| More specific sub-area | Nanocomposite materials, electrospinning |
| Type of data | Table, image, graph, figure |
| How data was acquired | SEM (JEOL Neoscope JCM-5000, USA), tensile (Instron 4444), DMA (TA Instruments Q800), TGA (TA Instruments Q600), DSC (TA Instruments Q2000), FTIR (Bruker ALPHA series spectrometer equipped with an ALPHA platinum ATR single-reflection diamond ATR module) |
| Data format | Raw, analyzed |
| Experimental factors | No pretreatment on samples was done |
| Experimental features | Tensile tests were done according to ASTM D882. Other tests were performed according to common practices and fully explained in the research paper [1]. |
| Data source location | Lincoln, New Zealand |
| Data accessibility | Data found in this article |

**Value of the data**

- The huge potential of electrospun sub-micron aligned fiber arrays in reinforcing a composite material is demonstrated which can open up new possibilities in various fields.
- The importance of nanocomposite manufacturing practice in final performance of the article is demonstrated.
- The data are useful for comparing purposes when addressing the influence of aligned electrospun fibers for reinforcing articles.

1. **Data**

A schematic representation of nanocomposite fabrication is presented in Fig. 1. SEM micrograph and image analysis results of electrospun fibers are illustrated in Fig. 2. Original micrographs obtained from SEM of fracture surfaces of fabricated nanocomposites are presented to show the material microstructure and quality (Fig. 3).

Tensile test data obtained for all specimens and the corresponding stress–strain diagrams is depicted (Fig. 5). Table 1 is presented a complete range of obtained tensile, DMA and TGA data.

TG and DTG curves are depicted in Fig. 6. DSC curves are presented in Fig. 7. FTIR graphs of all studied materials are displayed in Fig. 8.
2. Experimental design, materials and methods

Poly (vinyl alcohol) being chosen for the fiber, and epoxy resin for the matrix. The materials used in this study were selected based on processability and successful use in the manufacture of macro-scale composites [2]. Two different composite manufacturing methods were explored. Firstly; conventional wet lay-up followed by vacuum consolidation, and secondly deposition of the uncured epoxy matrix into the reinforcement layer-by-layer using electrospaying, followed by vacuum consolidation. For detailed information please refer to the related research paper [1].

2.1. Scanning electron microscopy (SEM)

Specimens were sputter-coated with gold for 240 s to avoid charging. The average diameter of the PVOH fiber was based on the measurement of ~200 fibers by computer image analysis of scanning electron micrographs (Electrospinz SEM Analyser software, Electrospinz Ltd., New Zealand) [3] as depicted in Fig. 2. The SEM of fractured surfaces of fabricated materials are presented in Fig. 3.
Fig. 2. (a) Photograph of aligned fibers before collection (b) SEM of aligned fibers and (c) diameter distribution of obtained fibers.
2.2. Tensile test

Rectangular coupons (80(l) × 6(w) × 0.1(t) mm) were tested at room temperature using a gauge length of 25 mm in accordance with ASTM D882 using a crosshead speed of 50 mm/min. Data were averaged over at least 4 replicates. A pair of 3D printed dumb bell-shaped tensile specimens were also prepared from polylactide to provide a non-slip gripping surface during the testing of the composite films (Fig. 4). The obtained engineering stress–strain curves are presented in Fig. 5.
2.3. Thermogravimetric analysis

Specimens of ~3 mg were placed in platinum pans and heated from 20 to 500 °C at a heating rate of 10 °C/min under a nitrogen atmosphere. Obtained graphs are illustrated in Fig. 6.
2.4. Dynamic mechanical analysis

DMA was carried out within the linear viscoelastic range of the samples in tensile mode using rectangular specimens $(15(l) \times 5(w) \times 0.1(t) \text{ mm})$. The storage ($E'$) and loss ($E''$) moduli were measured at a frequency of 1 Hz over a temperature range of 20 to 150 °C. The temperature ramp rate was 5 °C/min. A summary of obtained data pertaining DMA, TGA and tensile tests are presented in Table 1.

Fig. 6. Obtained TG and DTG curves.
2.5. Differential scanning calorimetry

Specimens of ~1.5 mg were cut to lay flat on the base of the low mass Tzero aluminum pan. To remove prior thermal history, specimens were first heated for 10 min, then cooled, and then reheated in the temperature range of 20–250 °C at a heating rate of 10 °C/min. Superposition of obtained DSC graphs (second heat) is shown in Fig. 7.

![DSC graphs](image)

Fig. 7. Superposition of obtained DSC graphs (second heat).

2.6. Fourier transform infrared spectroscopy

FT-IR was carried out on the electrospun mat, unreinforced epoxy and nanocomposites using a Bruker ALPHA series spectrometer equipped with an ALPHA platinum ATR single-reflection diamond ATR module. Spectra were averaged over 16 scans using a resolution of 2 cm⁻¹ over the mid-IR range (4000–400 cm⁻¹). The data was analyzed using OPUS software (Bruker). Superposition of obtained FTIR graphs is presented in Fig. 8.

![FTIR graphs](image)

Table 1
Summary of Tensile, DMA and TGA data.

| Treatments                          | URE  | WL30 | HEE30 | WL60 | HEE60 | WL90 | HEE90 |
|------------------------------------|------|------|-------|------|-------|------|-------|
| **Tensile**                        |      |      |       |      |       |      |       |
| Tensile strength (MPa)             | 20.21| 38.28| 49.50 | 56.73| 82.15 | 95.38| 140.40|
| Young’s modulus (GPa)              | 0.91 | 1.23 | 1.43  | 1.73 | 1.90  | 2.13 | 2.24  |
| Elongation at break (%)            | 2.92 | 4.28 | 4.52  | 4.88 | 7.92  | 8.12 | 8.36  |
| **DMA**                            |      |      |       |      |       |      |       |
| Storage modulus at 55 °C (MPa)     | 394  | 479  | 579   | 672  | 1068  | 1101 | 1124  |
| Storage modulus at 75 °C (MPa)     | 20   | 32   | 78    | 190  | 340   | 565  | 776   |
| tanδ peak temperature (°C)         | 68   | 71   | 73    | 81   | 83    | 84   | 92    |
| tanδ peak magnitude                | 0.78 | 0.74 | 0.59  | 0.56 | 0.52  | 0.46 | 0.45  |
| **TGA**                            |      |      |       |      |       |      |       |
| Onset of degradation (°C)          | 326  | 328  | 329   | 330  | 332   | 334  | 340   |
| Temperature of maximum decomposition rate (T_max) (°C) | 355 | 357 | 359 | 358 | 360 | 362 | 364 |
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Appendix A. Supplementary material

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.dib.2016.01.046.

References

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Fig. 8. Superposition of obtained FTIR graphs.