Vanadium flow batteries

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On the other hand, another efficient alternative approach is to replace the conventional carbon felt electrodes by an electrospun modified nanofibrous network. This 1D morphology of nanofibers within a 3D porous network has exceptional properties such as high tensile strength, high specific surface area, high porosity, and unique electrical, mechanical and physical properties leading to better performance in flow batteries5,24.

On the other hand, carbon black (CB) filled polymers are widely used in industrial applications owing to their cost advantage over other fillers to improve the mechanical, electrical, or thermal properties of the materials43,44,45. In our recent publication, polyacrylic acid (PAA) was used as a binder for higher CB loading within PAN-based CNFs and the obtained CNFs exhibited promising results as electrodes for VRFBs37.

However, there is no deep understanding from a material point of view when CB is added to the polymeric matrix utilized in the form of nanofibrous mat as electrode of VRFBs. The key properties of carbon blacks are considered fineness (primary particle size distribution), structure (aggregate size/shape), electrical conductivity, porosity and surface chemistry which all depend on the manufacturing process and may have an influence on the CB loading degree within fibers as well as their performance in VRFBs.

This study establishes guidelines for a successful fabrication of free-standing carbon black-loaded 3D nanofibrous mats as electrodes with good electrochemical behaviour in VRFBs using electrospinning. The results will help to understand the role of the carbon black type and its loading within the electrospun nanofibers as well as mild/severe heat treatment on the electrochemical behaviour of an efficient electrode for VRFBs. In the future, an upscaled approach for the fabrication of carbon-filled electrospun materials seems feasible.

The spinning solvent was N,N-dimethylformamide (DMF) and solutions were prepared by stirring polymers in the solvent. First, the polymers (i.e. blend of PAN and PAA) were dissolved overnight in DMF and then, CB was added to the obtained polymeric solution and stirred for another day. Different carbon blacks (detailed in Table S1) with various properties were used: CB-L-1, CB-L-2, CB-H-1, CB-H-2. All sample preparations were carried out under ambient conditions. ES was done using a commercially available unit (EC-DIG, IME Technology).

Thermal heating was used as a post-treatment after in-situ electrospinning to further enhance the

electrochemical properties of the created fibrous electrodes. Two strategies of thermal treatment were pursued to modify the electrospun mats for VRFBs: (i) a mild heat treatment at 300 ?C to produce a flexible polymeric matrix with loaded CBs; and (ii) a severe heat treatment by carbonization at high temperature (1,000 ?C) to make a rigid carbon- nanofibrous matrix.

In the first strategy, the nanofibrous mat was placed in an oven (Linn High Therm, Germany) held at a constant temperature of 300 ?C for 24 h. In the second strategy: the collected electrospun fibers were first stabilized by heating them at 280 ?C with a heating ramp of 1 ?C min-1. The temperature was held for 1 h in an open tube in a conventional furnace (split tube furnace EST 12/300, Carbolite Ltd). The stabilized fibers were then carbonized under a nitrogen flow by heating them to 1,000 ?C at a rate of 5 ?C min-1 and the temperature was held for 1 h.

Raman spectroscopy was performed with a REN-ISHAW via Raman spectrometer with a Leica microscope using 633 nm laser as an excitation source, 60x optical lens and stream-line mode to gain more detailed information about the surface defects of the as-prepared electrospun nanofibers. Deconvolution of the spectra was performed by mixed Gaussian/Lorentzian peaks to describe both the main D- and G-bands and the three minor ones D2, D3 and D4 using Origin Pro 2017.

The thermal stability of the fabricated mats was investigated with thermogravimetric analysis (TGA; Netzsch STA 449F5). The runs were performed in the presence of inert gas, at a heating rate of 10 ?C min-1 and at temperatures ranging from room temperature to 1,000 ?C.

CBs with different morphological and physical properties were in-situ incorporated into the electrospun webs, as described in the experimental section. Two groups of carbon blacks with different functionalities (see Table S1) were used to fabricate CB-loaded nanofibrous composite: group L; low CB content, including L-1 and L-2 and group H; high CB content, including H-1 and H-2.

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Web: https://sumthingtasty.co.za/contact-us/
Email: energystorage2000@gmail.com
WhatsApp: 8613816583346