INTERNATIONAL JOURNAL OF HYDROGEN ENERGY XXX (2017) 1–9

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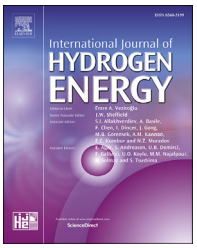

Ionic liquid-SnO_2 nanoparticle hybrid electrolytes for secondary charge storage devices: Physicochemical and electrochemical studies

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ARTICLE INFO

Article history:
Received 12 June 2017
Received in revised form
8 August 2017
Accepted 9 August 2017
Available online xxx

Keywords:
SnO_2-Nanoscale hybrid ionic fluids
Mechanical properties
Temperature dependent electrochemical cycling behaviour
Capacitance retentivity

ABSTRACT

A series of nanoscale hybrid ionic fluids (NHIFs) has been prepared by tethering 1-ethyl-3-(3-trimethoxysilylpropyl)-imidazolium bis(trifluoromethanesulfonyl) imide ionic liquids to the surface of SnO_2 nanoparticle, at different grafting densities. Investigations reveal that SnO_2 nanocores with uniform particle size of 14–17 nm are uniformly dispersed in different NHIF matrices through strong covalent attachment. Thermal stability and mechanical properties of NHIFs are found to improve with increasing grafting density. Temperature dependent electrochemical cycling behaviour of NHIF at different grafting densities are thoroughly investigated. In comparison to pure ionic liquid, the hybrid ionic fluids show substantial enhancement in electrochemical properties, which further improve with increasing grating density. The electrochemical cell containing NHIFs as electrolytes show very good capacitance retentivity (>90%) and long term cyclic stability above room temperature. Results obtained from the study demonstrate the applicability of these hybrid ionic fluids as promising electrolytes in secondary charge storage devices.

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Introduction

Research into new electrolytic materials for secondary charge storage devices such as electrochemical double layer capacitors and lithium ion batteries has received considerable attention in recent years. The use of room temperature ionic liquids (ILs) as potential electrolytes are found to be effective to increase the operative voltage of charge storage devices due to their high thermal stability, wide electrochemical window [1,2], ultralow vapour pressure, and considerably high ionic conductivity [3]. However, practical use of ionic liquids as electrolyte are still under debate due to their poor mechanical properties and lower capacitance retentivity [4]. Thus, further development of this class of materials to overcome the above mention constrains is warranted for their successful use as electrolytes [5].

Recent studies have revealed that functional ionic liquid based hybrid materials formed in combination with graphene-based nanostructured materials [6] or metal oxides nanoparticles such as SiO_2 [7–9], ZrO_2 [10], TiO_2 [11], Al_2O_3 [12] etc. and show improved features with sharing properties of both ionic liquids and nanoscale materials. Specifically, significant synergic properties have been observed in a class of materials, the nanoscale hybrid ionic fluids (NHIFs), formed by tethering or covalent grafting ionic liquid chains on the

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http://dx.doi.org/10.1016/j.ijhydene.2017.08.065

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Please cite this article in press as: Dutta B, et al., Ionic liquid-SnO_2 nanoparticle hybrid electrolytes for secondary charge storage devices: Physicochemical and electrochemical studies, International Journal of Hydrogen Energy (2017), http://dx.doi.org/10.1016/j.ijhydene.2017.08.065

surface of different nanostructured cores [7,10–12]. Due to the interactions between ionic liquids and nanoparticles, thermal, mechanical and electrochemical properties of these NHIFs are found to be superior to those of the ionic liquid alone [12,13]. Thus, the grafting density of the ionic liquid chains on the surface of the core nanoparticles should be one of the key parameters to effectively tune the properties of NHIF.

To extend the research further, the current study is focused on to get insight to the effect of grafting density on the physicochemical and electrochemical properties of different imidazolium ionic liquid based NHIF materials. These NHIF materials are prepared by tethering 1-ethyl-3-(3-trimethoxysilylpropyl)-imidazolium bis(trifluoromethanesulfonyl) imide [b((MeO)$_3$Sip)im][NTf$_2$] ionic liquids to the surface of pre-synthesized tin oxide (SnO_2) nanoparticles at three different grafting densities. The thermal, mechanical and electrochemical properties of these NHIFs are studied. It has been observed that both thermophysical and electrochemical properties of the NHIFs significantly depend upon the grafting density of ionic liquid chains to the surface of SnO_2 cores. The NHIF containing cells show thermally stable wide electrochemical potential window and both specific capacitance of the cell and long-term cycling stability can be substantially improved with increasing grafting density.

Experimental section

Synthesis

Synthesis of SnO_2 nanoparticles

In a typical synthesis procedure appropriate amount of an aqueous solution of oxalic acid was drop wise added to the aqueous solution of Tin (II) chloride ($SnCl_2 \cdot 2H_2O$, Sigma-Aldrich). The solution was stirred for 8 h to get a white precipitation which was washed with water and dried at 100 °C for overnight. Finally the dried powder was calcinated at 800 °C for 1 h to get the SnO_2 nanoparticle powder.

Synthesis of SnO_2-NHIF

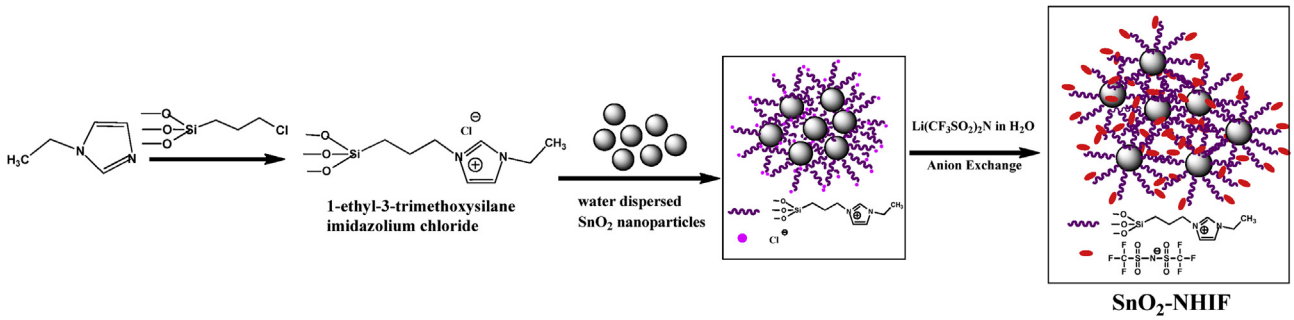
Different SnO_2-NHIF materials were prepared according to Scheme 1. Briefly, 1-ethylimidazole (Sigma Aldrich) and (3-Chloropropyl) trimethoxysilane ≥97% (Sigma Aldrich) were refluxed at 120 °C under nitrogen atmosphere for 2 days. The resultant viscous ionic liquid 1-ethyl-1-propyl imidazolium chloride with the trimethoxysilane tail was collected as honey like orange viscous liquid via liquid extraction in ether. To tether the resulting IL to the surface of the as synthesized SnO_2 nanoparticles, the excess amount (1.5 times) of the IL was allowed to react with the aqueous dispersion of nanoparticles at 100 °C for 12 h under inert atmosphere and subsequently absolute ethanol was added to it. The surface functioned core nanoparticles were collected by repeated washing and high speed centrifugation.

Finally the hybrid ionic fluids with different cores were obtained by substituting Cl^- anion with the $(CF_3SO_2)_2N^-$ by the simple metathesis reaction. In a typical procedure, appropriate amount of aqueous solution of $[SnO_2\text{-IL}]^+[Cl]^-$ and $Li(CF_3SO_2)_2N$ salt were added in 1:1.2 ratio under continuous stirring. Due to the hydrophobic nature of the $(CF_3SO_2)_2N^-$ anion, the $[SnO_2\text{-IL}]^+[(CF_3SO_2)_2N]^-$ NHIF immediately separates from the water phase and settles to the bottom of the container. The upper phase was decanted and NHIF was collected by repeated washing with distilled water until no precipitation of AgCl can be observed within the decant, after the addition of $AgNO_3$. That further confirmed the completeness of the metathesis reaction and absence of any unreacted lithium salt within the material. Finally the NHIF was rigorously dried to remove any residual moisture and preserved in an argon filled glove box.

Three different NHIFs with SnO_2 nanoparticle weight percent of 0.5, 1.0 and 2.0 were prepared accordingly and were designated as 0.5SnO_2-NHIF, 1.0 SnO_2-NHIF and 2.0 SnO_2-NHIF, respectively.

Thermophysical and mechanical characterizations

The X-ray diffraction (XRD) study of the synthesized SnO_2 nanoparticles was carried out on a Rigaku Miniflex 600 X-ray diffractometer operating at 40 kV and 20 mA with a copper target (λ = 1.5418 Å) and at a scanning rate of 0.5°/min. Thermal stability of the NHIFs was analyzed using Netzsch TG 209 F3 Tarsus thermogravimetric analyzer (TGA) at a heating rate 10 °C/min under nitrogen environment. From TGA profiles the grafting density of RTIL chains to different wt% of SnO_2 cores were estimated. The dispersion of core nanoparticles within the NHIF matrices was characterized by high-resolution transmission electron microscopy (HR-TEM) (JEOL JEM-2100, 200 kV). Mechanical properties of different NHIFs at different temperatures were studied in an advanced air bearing rheometer, model Bohlin Gemini 2 (Malvern



Scheme 1 – Synthesis scheme for SnO_2-NHIF.

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