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Helical polysilane coating onto hollow spherical indium oxide: Fabrication, characterization and infrared emissivity property study

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ABSTRACT

A new-type of composite combined by helical polysilane (HPS) and hollow spherical indium oxide (In_2O_3) was designed and fabricated via solution blending. The hollow spheres constructed by In_2O_3 particles were synthesized via a facile hydrothermal route where it demonstrated larger surface area, enabling the combination of HPS and In_2O_3 more easily. Meanwhile, the synergetic effect between functional group of HPS and surface hydroxyl of In_2O_3 improve the reduction of infrared emission. Consequently, the composite shows a superior low infrared emissivity performance at the value of 0.416 (8–14 μm), and the covering of HPS make the composite disperse well in the polar reagent. This work develops a feasible strategy that utilizes metallic oxide to design and synthesize hollow spherical matrix for preparing the low infrared emissivity materials.

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1. Introduction

Morphology design is an effective strategy to fabricate and manufacture hierarchical materials with distinctive optical properties. Therefore, multicomponent composites with a wealth of configurations, including tubular [1], laminated [2] and core-shell structure [3,4], have been substantially developed in past years. Particularly, core-shell structural nanocomposite has triggered great attention in general areas, because of their superior optical performance originating from the promoting synergistic effect between different components and the morphology of functional shell. For instance, Wang et al. prepared core-shell structural SiO_2@DNA-LDH which displayed low infrared emissivity property at the value of 0.458 and demonstrated promising prospect in the application of military equipment or vehicles stealthy [3]. However, challenges emerging when removing the SiO_2 core from LDH shell, the vulnerable LDH hollow structure usually failed to maintain original structure. Unlike DNA-LDH, In_2O_3 particle could withstand harsh treatment and transform to fulfil specific requirement [5–8]. Benefiting from unusual physicochemical and optoelectronic properties, even amorphous In_2O_3 exhibit a certain performance of low infrared emissivity at the value of 0.913 [9]. From this point of view, it is an intriguing target to contrive hollow hierarchical architecture constructed by indium oxide particles to reduce infrared radiation [10].

The intermolecular interactions, such as π-π [11], CH-π [12] and van der Waals forces [13,14], have been widely considered as driving forces in determining the behavior of polymer-wrapping. To be more specific, polysilanes display excellent compatibility with CNTs not only due to their intermolecular noncovalent interactions, but also resulted from show helical conformation of polysilanes and tubular configuration of CNTs [15]. Based on the computational simulation, the helical groove of polysilane could closely stick to the highly curved surface of CNTs which improve the formation of interfacial hydrogen bond [1]. According to the theory proposed by yang et al. [16], hydrogen bond showed close relationship with the unsaturation degree leading to the reduction of the infrared radiation. Compared to Collagen-g-PMMA/In_2O_3, the HPS/In_2O_3 composite constituted by porous hollow In_2O_3 spheres undoubtedly possess higher curved surface to interact with polymers. Furthermore, the helix of HPS backbone could be modulated to adapt the curve of particle surface and the polar functional groups in HPS's sidechain would be well compatible with In_2O_3's hydroxyl group. In other words, the conjugation of HPS and In_2O_3 will be feasible via

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solution blending owing to the self-adjust ability of HPS. Besides abovementioned properties, the heterojunction assembled by n-type In_2O_3 [17] and p-type HPS [18] semiconductors also bring about intriguing property.

In this work, the combinational pattern between In_2O_3 surface group and functional group of HPS was studied, using combinational analyses with Fourier transform infrared spectroscopy (FT-IR), X-ray photoelectron spectroscopy (XPS), X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscope (SEM) and thermogravimetric analysis (TGA). Moreover, the conformational behaviors of single-handed HPS coating on In_2O_3 were spectroscopically probed by the circular dichroism (CD) and ultraviolet–visible spectroscopy (UV–Vis). In addition, computational molecular mechanics and animation softwares were employed to simulate the interfacial interaction between HPS and In_2O_3, connecting with the reduction value of infrared radiation emissivity (IRE).

2. Experimental

2.1. Materials

All the reagents and solvents were of analytic grade purity, bought from J&K or Aladdin Industrial Corporation, and used as received without further purification. Solvents for the polymerizations (tetrahydrofuran, xylene) were refluxed with sodium prior to the experiment. Dichlorodi-*n*-hexylsilane (DCDHS) and Platinum(0)-1,3-divinyltetramethyldisiloxane complex (19.0–21.5% Pt) were purchased from Tokyo Chemicals Industry. L(+)-Lactic acid and allyl bromide were obtained from Adamas-beta. Methyl 2-(allyloxy)propanoate was synthesized according to the procedures of previous literature [19]. Deuterated solvents were purchased from Beijing Chongxi High-tech Incubator Co. Ltd. $In(NO_3)_3$ and glucose were purchased from Sinopharm Chemical reagent Co., Ltd.

2.2. Instruments

^1H NMR and ^{13}C NMR spectra were recorded from samples in $CDCl_3$ using a Bruker AV 300 NMR spectrometer operated at 300 and 75 MHz, respectively. The molecular weight information was calculated using a Polymer Laboratories' PL-GPC220 gel permeation chromatography (GPC) system (with PLgel-5μL-MIXED-C, 300*7.5 mm column); tetrahydrofuran (THF) was used as the eluent at 30 °C (flow rate: 1 mL/min). FTIR spectra were obtained using a Bruker ALPHA FTIR spectrometer. Samples were prepared with the KBr disk method and measured 32 scans with resolution of 1 cm^{-1} at room temperature. The thermal stabilities were measured using a TA Q-600 thermogravimetric analyzer under a nitrogen atmosphere; the sample was first loaded in a Pt cell and then heated from 50 to 800 °C (heating rate: 10 K min^{-1}) under a 60 mL min^{-1} nitrogen flow. A JEOL-2100 TEM operated at an accelerating voltage of 100 kV was used to record transmission electron microscope (TEM) images, high-resolution transmission electron microscope (HRTEM) images, and selected area electron diffraction (SAED) patterns for further characterization. SEM was performed using a LEO-1530VP microscope. XRD was measured on a Rigaku D/MAX-R diffractometer with a copper target at 40 kV and 30 mA. UV–vis and CD spectra were recorded using a SHIMADZU UV-3600 Series spectrophotometer and an Applied photophysics Chirascan Ultrasensitive Spectroscopy, respectively, with a 10 mm quartz cell at room temperature. XPS was performed using a Shimadzu (Amicus, Japan) instrument. Infrared emissivity values of the samples were investigated on a silicon substrate by using an IRE-2 Infrared Emissometer of Shanghai Institute of Technology and Physics, China. The sample solution was dripped on the silicon substrate and then the solvent was evaporated, this process was conducted several times until the sample covered on the plate surface completely. Each sample was measured 20 times from different parts on the plate and then the average value was calculated.

2.3. Synthesis of HPS copolymer (2:3)

DCMMS and sandy sodium was prepared according to the procedure of literature. HPS was prepared using the Wurtz-type condensation reaction of DCMMS (8 mmol) and DCDHS (12 mmol) with sandy sodium in THF (20 mL) for 24 h at room temperature and quenched by 100 mL methanol, and then filtered by membrane with careful successive additions of 2-propanol (5 mL) and methanol (5 mL) to eliminate the higher molecular weight fraction of the bimodal distribution. The insoluble HPS fraction after the addition of methanol was dried in a vacuum. Mn = 10,800, Mw/Mn = 1.1. Yields: 23–58%. ^1H NMR (300 MHz, $CDCl_3$): δ [ppm] = 4.02–3.88 (CH), 3.77–3.62 (CH_3–O), 3.56–3.41 (O–CH'H''–CH), 3.35–3.24 (O–CH'H''–CH), 1.82–1.56 (CH_2–CH_2–CH_2), 1.42–1.32 (CH–CH_3), 0.91–0.77 (Si–CH_3), 1.76–0.92 ($C(CH_3)_2$, Si-(CH_2)5–CH_3, and CH_3–*Si*–CH_2). ^{13}C NMR (75 MHz, $CDCl_3$): δ [ppm] = 104.65 (CH–COO–CH_3), 59.48 (CH_3–CH–O), 45.83 (CH_3–O), 45.91 (Si–CH_2–CH_2), 33.01 (Si–$(CH_2)_2$–CH_2–Pr), 31.52 (Si–$(CH_2)_3$–CH_2–Et), 22.57 (Si–CH_2–CH_2-Bu), 31.62 (CH_2–CH_2–CH_2), 29.64 (CH–CH_3), 16.61 (Si–$(CH_2)_4$–CH_2–Me), 16.19 (Si–CH_2-$(CH_2)_2$–O), 16.12 (Si-(CH_2)5–CH_3), 14.01 (Si–CH_2–CH_2-Bu), −0.07 (Si–CH_3). FT-IR (cm^{-1}, KBr): 2956, 2921, 2857, 1757, 1741, 1571, 1465, 1403, 1379, 1341, 1259, 1186, 1075, 1015, 961, 886, 840, 792, 772, 721, 685.

2.4. Preparation of hollow spherical indium oxide

All reagents used in this work were analytical grade. Carbon spheres [18–20] were synthesized in glucose solutions by a hydrothermal route, and the used carbon spheres had an average diameter of ca. 300 nm. In_2O_3 hollow spheres were obtained by electrostatic absorption of In^{3+} on the carbonaceous spheres and the solid hollow spheres was prepared via hydrothermal reaction. The templates were subsequently removed by calcinations. Firstly, $In(NO_3)_3$ (1 mmol) was dissolved in deionized water and ethanol (50 mL of 1:1) mixed solution. Then the prepared carbon spheres (0.2 g) were dispersed in the solution. After sonicated for 30 min, the mixture was transferred into a Teflon-lined stainless-steel autoclave (100 mL) and maintained at 180 °C for 4 h. Then the precipitate was separated by centrifugation, washed with deionized water and ethanol, and dried at 60 °C. Finally, the obtained sample was calcined at 600 °C for 5 h in oxygen atmosphere.

2.5. Preparation of HPS/In_2O_3 nanocomposites

Solutions of HPS and In_2O_3 were mixed using an ultrasonic cleaner (Endosonic Co. Ltd) in order to make nanocomposite particles well dispersed in the solvent. A solution of HPS in THF (2 mM, 2 mg/mL) was added to a solution of either In_2O_3 in THF (2 mM, 2 mg/mL) or In_2O_3 in $CHCl_3$ (2 mM, 1.787 mg/mL) at a rate of 0.5 mL min^{-1} with continuous sonicating for 6 h at room temperature [15]. After mixing, the solution was vacuum dried to remove the solvent 75 °C for 48 h.

3. Results and discussion

3.1. Synthesis of HPS copolymer

DCMMS with methoxycarbonyl functional group was obtained

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