



Preparation of antistatic polystyrene superfine powder with polystyrene modified carbon nanotubes as antistatic agent



Min Zhang, Chen Zhang, Zhongjie Du, Hangquan Li, Wei Zou*

Key Laboratory of Carbon Fiber and Functional Polymers, Beijing University of Chemical Technology, Ministry of Education, Beijing, 100029, PR China

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ABSTRACT

A facile approach for fabricating antistatic polystyrene (PS) microspheres used in 3D print had been carried out by introducing polystyrene onto the MWCNTs (MWCNTs@PS) surface. Firstly, poly (styrene-co-acryloyl chloride) (PS-co-APCl) was synthesized via free radical polymerization. Then azide-poly (styrene-co-acrylic acid) (PS-co-APAA) were prepared through the reaction between the acylchloride groups of PACl with the sodium azide. Subsequently, the azide groups of PS-co-APAA high produced nitrene under high temperature, reacting with the sp^2 carbon in MWCNTs. MWCNTs@PS thus obtained were characterized using FTIR, EDS, Raman, TAG and TEM, and used as antistatic agent in PS in-situ suspension polymerization. MWCNTs@PS/PS composite microspheres were characterized using TAG, SEM and TEM. Results revealed that PS was covalently bonded onto the surface of MWCNTs, which improved the compatibility with matrix. Thus, the antistatic performances were enhanced by a well-constructed electrostatic discharge channel in the polystyrene matrix. As a result, the surface resistance of the percolation threshold was $3.54 \times 10^8 \Omega$ when the loading of MWCNTs@PS was at 0.5 wt%. The average diameter of MWCNTs@PS/PS microspheres was about 54 μm and the morphology was also very smooth, which could be used in 3D print.

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1. Introduction

3D printing technologies also known as additive manufacturing has caused widely attention in many fields [1–3], because of their high efficiency compared with the traditional methods [4,5]. Selective laser sintering (SLS) [6] as a kind of 3D printing, which used polymer powder as printed material and the diameters of polymer powder was about 30–100 μm [7]. Polymer materials were easily caused electrostatic effect, especially in such a small size. In the process of spread powder easily caused electrostatic effect due to friction, making these tiny powder attached to the drum, resulted uneven spread of powder [8,9]. It had drawn enormous attention from engineers and scientists that how to avoid electrostatic effect [10] in the process of spread powder. So it was necessary to manufacture a kind of special antistatic powder used in SLS. Multi-walled carbon nanotubes (MWCNTs) have been become one of the most striking recent discoveries in chemistry and materials science, owing to their efficient 'network-like' conducting structure [11], large surface area properties and high aspect ratio [12]. Therefore, MWCNTs have been used widely as antistatic modifier materials.

However, non-polar and chemical inert surface made MWCNTs agglomerate easily in matrix. The unevenly disperse MWCNTs in matrix could not form well-constructed electrostatic discharge channel in the matrix to approach percolation sufficiently, unless the added amount of MWCNTs were very high. Therefore, the dispersibility of MWCNTs was very important for fabricating antistatic materials with lower MWCNTs content [13,14].

Both noncovalent [15] and covalent modifications [16–18] of the MWCNTs have been developed to improve its dispersibility and compatibility. Covalent modification [19–24] was the most effective method to modify MWCNTs. Tsang S C [25] introduced carboxylic acid groups at the defect sites of MWCNTs by nitric acid oxidation. Long alkyl chains, polymers and sugars could be grafted on modified MWCNTs surface by esterification and amidation reactions. Mitchell et al. [26] synthesized 4-(10-hydroxydecyl) benzoate-SWCNTs by in situ reaction of organic diazonium compounds, and prepared functionalized SWCNTs/PS nanocomposite. They found that functionalized SWCNTs/PS nanocomposite showed low percolated SWCNTs loading than unfunctionalized SWCNTs/PS nanocomposite. These functionalizations improved the compatibility and dispersibility of MWCNTs but also alter the structure and electronic properties of MWCNTs. Azide/acyl fold nitrogen [27] compounds were important organic intermediates,

* Corresponding author.

E-mail address: zouwei@mail.buct.edu.cn (W. Zou).

which with high reactivity were widely used in the interface chemistry, material chemistry, biology, etc. Azide [28,29] was an efficient and versatile approach for strong covalent bonding of polymers to MWCNTs ways to modified carbon nanotubes.

In this paper, in order to improve the compatibility and dispersibility of MWCNTs, polystyrene was grafted onto MWCNTs surface via cycloaddition reaction [30,31], as illustrated in Fig. 1. Firstly, PS-co-APCl was synthesized via free radical polymerization. Then PS-co-APAA was prepared through the reaction between the acylchloride groups of PACl with the sodium azide. Subsequently, the azide groups of PS-co-APAA high produced nitrene under high temperature, reacting with the sp^2 carbon in MWCNTs [32–35]. MWCNTs@PS thus obtained and used as antistatic agent in PS in-situ suspension polymerization. PS graft on the MWCNTs made it have a better dispersion in matrix. MWCNTs@PS was used as antistatic agent in in-situ suspension polymerization [36–39] to prepare MWCNTs@PS/PS composite microspheres [40]. We use sodium dodecyl benzene sulfonate as second dispersant to reduce surface tension and the average diameter of MWCNTs@PS/PS microspheres was about 54 μm , the morphology was also very smooth, which could be used in 3D print. Thus, the surface resistance of the percolation threshold was $3.54 \times 10^8 \Omega$ when the loading of MWCNTs@PS was at 0.5 wt%.

2. Experimental

2.1. Materials

Purified multi-wall carbon nanotubes (MWCNTs) of acetylene type were supplied by Shenzhen Nanotech Port, China

(diameter 10–30 nm and length 0.5–500 μm , purity >90% reported by supplier). Acryloyl chloride (purity $\geq 96.0\%$) was purchased from Energy Chemical. Polystyrene, Acetone (A.R), 1,4-Dioxane (A.R), and *N,N*-Dimethylacetamide (DMAc) were purchased from Beijing Chemicals Co. of China. Sodium azide (DDN) was provided by Xiya Reagent Co. Azo-bisobutyronitrile (AIBN) was kindly offered by Beijing Chemical Reagent Co. and benzoyl peroxide (BPO), a free radical initiator, were purchased from Fluka. Gelatine as stabilizer and sodium dodecyl benzene sulfonate (SDBS) as a surface active agent for suspension polymerization of styrene, was provided by Tianjin Jinke Fine Chemical Institute.

2.2. Preparation of poly (acryloyl chloride)-co-polystyrene

PACl-co-PS was prepared via free radical polymerization. In a 100 mL dry Schlenk tube equipped with a magnetic stirring bar, the solution of styrene (12.5 mL) and 2,2-azoisobutyronitrile (AIBN, 375 mg) in anhydrous 1,4-dioxane (27 mL) was stirred for 10 min at room temperature. And then, 1 mL acryloyl chloride was injected in Schlenk tube. The mixture was kept vigorously stirring for 24 h at 60 °C under nitrogen atmosphere.

2.3. Preparation of azide-poly (styrene-co-acrylic acid)

In a 100 mL round bottomed flask the solution of PS-co-PACl (250 mg) and acetone (16 mL) were stirred at 0 °C, then the aqueous solution of sodium azide (NaN_3 , 2.5 mL) which mass fraction of 10% was dripped into the round bottomed flask. The mixture was kept vigorously stirring for 4 h at 0 °C. Finally, the mixture was collected by rotary evaporation. The collected solid

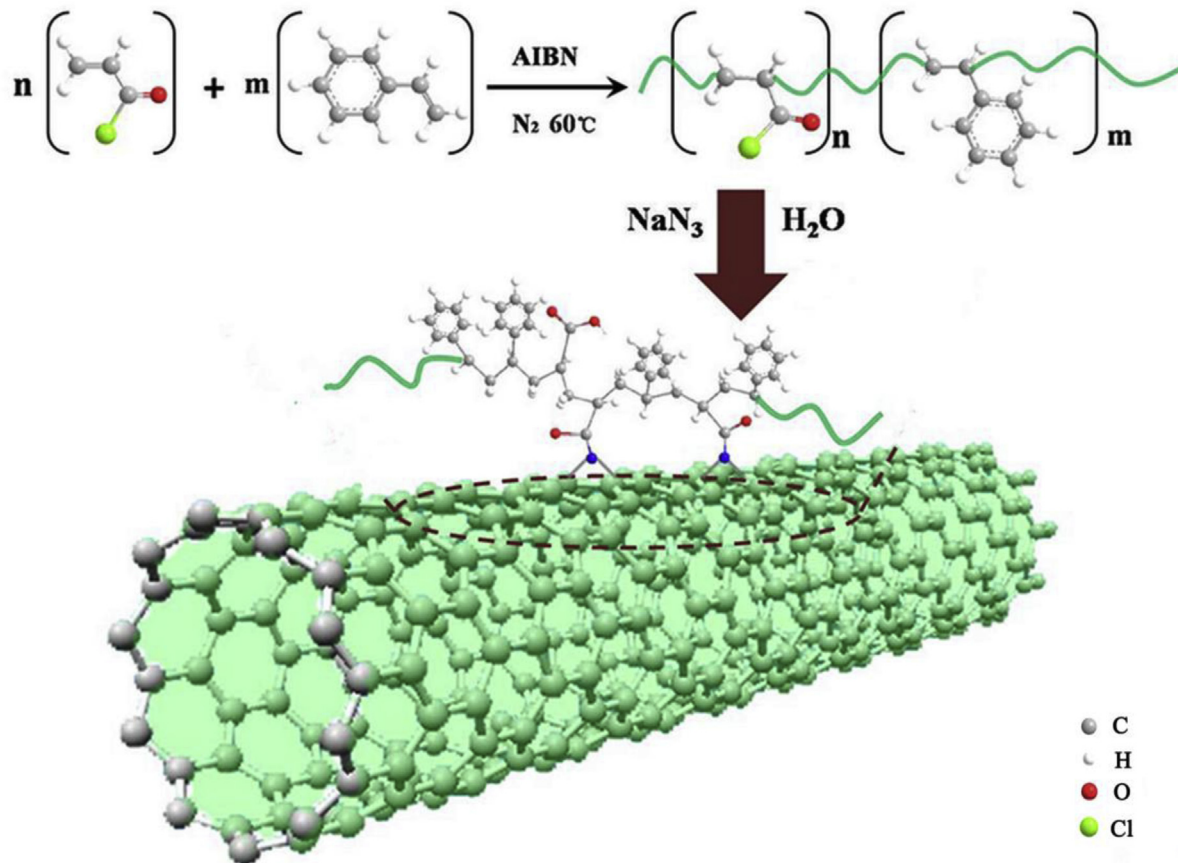


Fig. 1. Schematic presentation of MWCNTs@PS.

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