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Nanocast carbon microsphere flowers from a lanthanum-based template

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ABSTRACT

Hollow carbon microsphere flowers were nanocast from glucose, acrylamide, and acetylene sources. Carbon growth was catalyzed by a lanthanum graft copolymer template using wet acetylene. The resulting spherical shape is beneficial for 3-D porosity, and has a highly graphitic content as indicated by raman spectroscopy (I_D:I_G = 0.99). The carbon has a high surface area of 1000 m^2/g, as well as strong π-π stacking of aromatic carbons.

1. Introduction

Porous carbons are commonly used as catalyst supports, adsorbents, and energy storage materials. Carbons are derived using variety of methods like polymer degradation, polysaccharides pyrolysis, and combustion of gases during chemical vapor deposition (CVD) [1–4]. Recently, a CVD method for the low temperature production of well-formed graphene-like carbon microstructures has been developed by Ryoo et al. [5]. Other structures, such as wrinkled silica nanoparticles were impregnated with lanthanum and used to nanocast carbon [6].

Previously, cerium and lanthanum flower microsphere were synthesized from polysaccharide based graft copolymers [7,8]. In this study lanthanum carbonate nanostructures were used as nanocasting templates for the catalytic formation of graphitic carbon. Carbons with a 3D pore structures are first derived from a lanthanum-ion initiated acrylamide-glucose graft copolymer. Additional graphitic carbon was then nanocast from acetylene, utilizing the catalytic properties of a lanthanum based template.

2. Experimental

2.1. Materials

All chemicals were commercially sourced and used as received. The lanthanum nitrate hydrate (99.9%) and D-(+)-glucose (99%) were obtained from Alfa Aesar. Acrylamide (99%) was purchased from Sigma and ammonium hydroxide (28 wt%) from EMD Chemicals, Inc. The hydrochloric acid (36 wt%) was sourced from Fisher Scientific. Dissolved acetylene and high purity nitrogen were obtained from Airgas.

2.2. Synthesis of $LaCO_3OH/La_2CO_3O_2$ microsphere flowers

Lanthanum microsphere flowers (2–7 μm) were synthesized as previously reported [7]. In summary, 1.08 g lanthanum nitrate hydrate ($La(NO)_3{\cdot}6H_2O$), 0.90 g (D-(+)-glucose), and 0.53 g acrylamide were combined in 40 mL of water and NH_4OH was added dropwise until the pH ~10. The reaction was stirred for 5 h, then placed in a 45 mL teflon lined autoclave at 180 °C for 72 h. The resulting orange product was washed with 50/50 water/ethanol and then dried at 90 °C overnight.

2.3. Synthesis of carbon microsphere flowers

The lanthanum microsphere flowers were placed into an alumina boat inside a horizontal tube furnace. The furnace was heated at a rate of 5 °C/min to 600 °C under a 200 mL/min high purity nitrogen flow. Once the temperature reached 600 °C, 30 mL/min of acetylene and 2 mL/h of water were passed through the furnace for 1.5 h. The water was preheated to 150 ℃ as steam before introduction into the tube. Subsequently, the acetylene and water flows were discontinued and the temperature was increased to 900 °C for 2 h and then cooled. The resulting product was washed with

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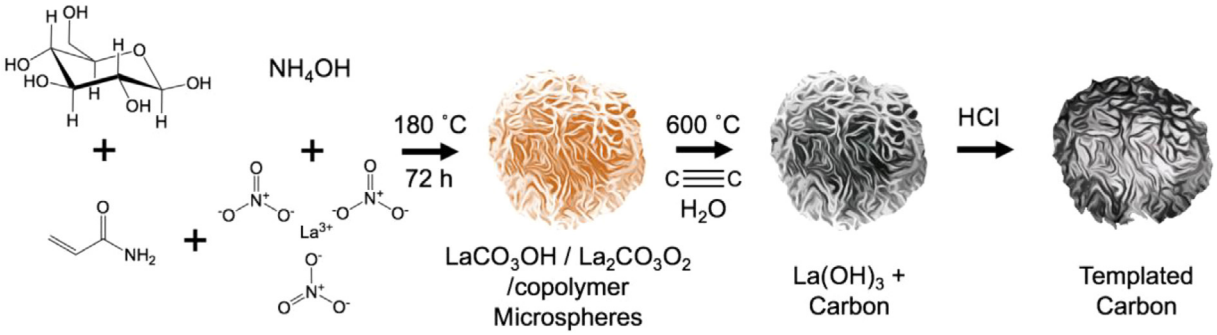

Fig. 1. The cartoon depicts the synthesis of carbon microspheres from a lanthanum carbonate template.

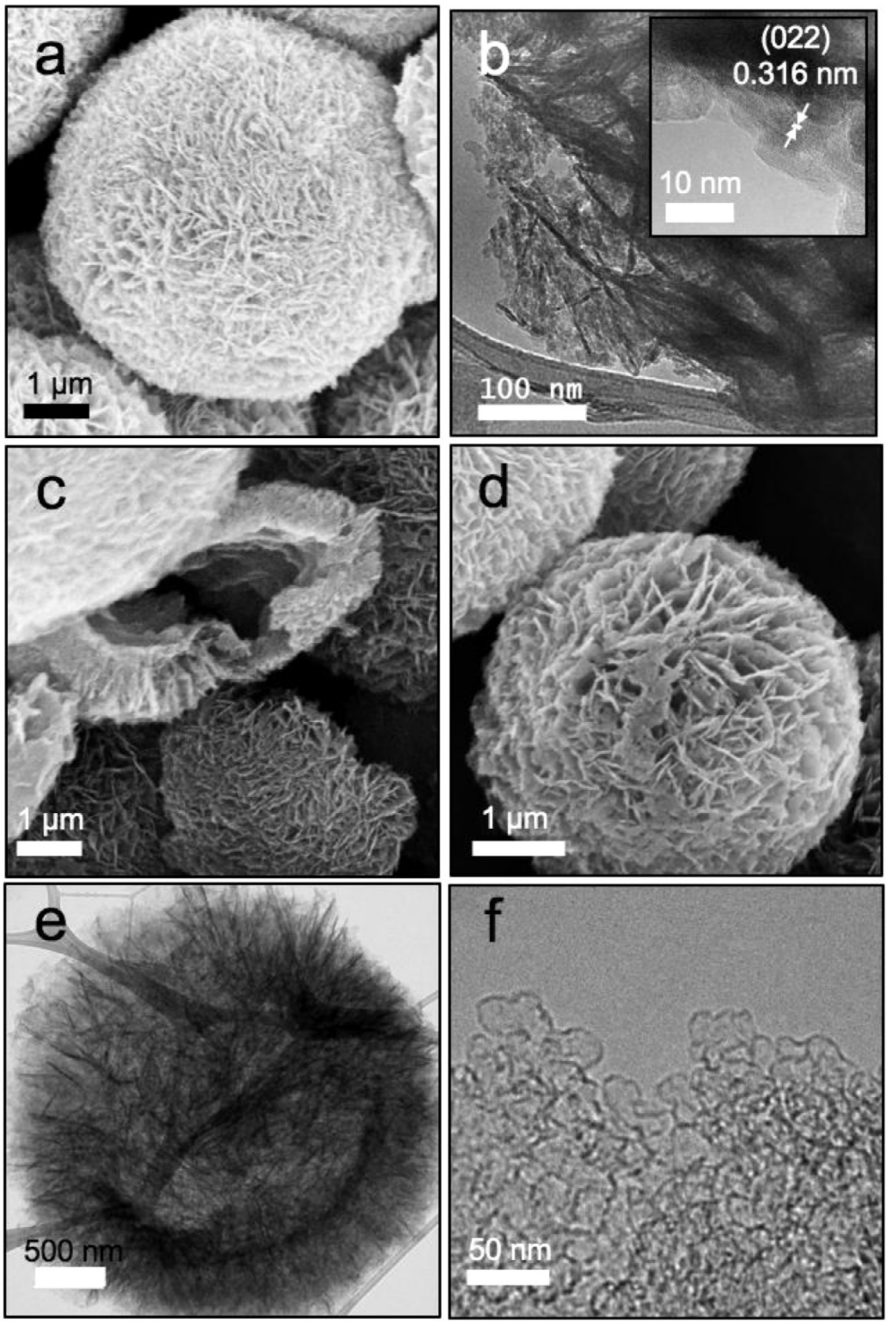

Fig. 2. SEM/TEM images of (a) $LaCO_3OH/La_2CO_3O_2$ microspheres; (b) $LaCO_3OH/La_2CO_3O_2$ microspheres inset of $La_2CO_3O_2$ (0 2 2) lattice fringes; (c) $La(OH)_3$ microsphere post-CVD shows the hollow core; (d and e) carbon microsphere morphology; (f) close-up image of disordered carbon pores.