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Visible light laser-induced graphene from phenolic resin: A new approach for directly writing graphene-based electrochemical devices on various substrates

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ABSTRACT

The cost-effective construction of self-designed conductive graphene patterns on desired substrates is crucial to the fabrication of graphene-based electrochemical devices. Here, we report a new approach for the scalable construction of laser-induced graphene (LIG) patterns on diverse substrates by using phenolic resin (PR) as the precursor. The PR-based LIG, which was produced with smart and inexpensive 405 nm semiconductor lasers under ambient conditions, possesses several interesting properties, e.g., 3D porous structures, low resistance (~44 Ω/sq), good mechanical property and a wide range of applicable substrates, e.g., polymer films, glass slides, metal foils, ceramic plates and plant leaves. The efficient absorption of laser light by PR coatings themselves or dopants such as metal salts and organic dyes is demonstrated critical to the formation of PR-based LIG by visible light lasers. Based on this technique, self-designed and highly conductive graphene arrays can be easily constructed on various substrates to fabricate all-carbon supercapacitors and electrochemical glucose biosensors. The unique properties of PR materials, including easy synthesis, tunable structure and composition, excellent film-formation ability and extremely low cost, thus foresee the promising applications of PR-based LIG in electrochemical fields.

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1. Introduction

Over the past decades, there has been a sharp increase in the synthesis of graphene with high mobility [1,2], high conductivity [3–5], high stiffness [6], superior electrocatalytic properties [7–9] and electromechanical modulation [10], which has promising applications in a wide range of fields such as electrochemical biosensors [11–13], lithium batteries [14–16] and supercapacitors [17–20]. Many of these applications require the construction of conductive graphene films or coatings with desired shapes, sizes and substrates. Therefore, great efforts have been made for the efficient construction of graphene patterns on various substrates, including templated CVD [16,20], etching-precipitation [21] and printing techniques like screen printing [22] and inkjet printing [23]. However, these methods usually have limitations like expensive instruments, complicated operations or need of extra templates.

Laser-induced formation of graphene provides a template-free approach for constructing conductive graphene patterns with high precision, easy processing and low cost [24–26], which has been demonstrated to possess promising applications in constructing graphene-based energy storage devices and sensors [25–27]. Kaner et al. started this trend by laser-induced reduction of graphene oxide (GO) films with a standard LightScribe DVD optical drive, which produced graphene patterns with high electrical conductivity and excellent electrochemical properties [28].

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Latter, Tour et al. developed a more straightforward method for the fabrication of patterned LIG by laser-induced graphitization of high temperature resistant polymers such as polyimide (PI) [25,29,30]. Compared with the GO approach, the PI approach allows the direct writing of highly conductive and porous graphene patterns on insulative PI tapes to form flexible and smart microsupercapacitors (MSCs), which greatly simplifies the fabrication process and reduces cost. Unfortunately, the poor solubility and non-melting property of PI inevitably restrict its doping functionalization and applicable substrates. Therefore, the synthesis or discovery of new precursors with tunable composition and favorable processability is of great significance for the development of LIG-based electronics or functional materials. However, so far only very few works reported the discovery of new LIG precursors such as lignin-containing wood [31] and sulfonated poly(ether ether ketone) (SPEEK) [32].

Phenolic resin (PR), a widely used heat-resistant polymer [33], is frequently employed as precursors for the synthesis of carbon composites and activated-carbon in material fields, which has promising applications in various fields including energy storage [34] and electrochemical sensors [35]. For instance, Yu et al. recently demonstrated that microporous hard carbon prepared from PR exhibited excellent lithium/sodium storage capacity [36]. However, most of previous PR-derived carbon materials are prepared by extremely severe conditions like prolonged high-temperature heating in inert atmospheres. Up to date, only one work reported the carbonization phenomena of Pluronic F127-doped and UV light-treated PR by a CO_2-based infrared (IR) laser but without conductivity test and electrochemical applications [37].

In this work, we demonstrate for the first time that PR is also a promising precursor of LIG. Different from PI, PR can be easily synthesized from phenols and formaldehyde with different physiochemical properties, and may exist in the form of either thermoplastic/thermoset blocks or soluble powder. The latter is soluble in various organic solvents including ethanol and acetone to form solutions with desired viscosity, which is suitable for the construction of PR coatings or doped composite films by various techniques. The laser-induced graphitization of PR doped with various organic dyes or metal salts like ferric chloride ($FeCl_3$) allows the rapid and scalable construction of highly conductive and 3D porous graphene patterns on various substrates at ambient conditions. Moreover, the synthesis of PR-based LIG can be achieved by inexpensive semiconductor-based visible light lasers, thus enabling the facile fabrication of LIG-based devices on desirable substrates with simple instrument and low cost. The resulting PR-based LIG possesses good mechanical and electrochemical properties, which has been successfully applied in capacitors and electrochemical sensors.

2. Experimental section

2.1. Reagents

Ethanol-soluble PR powder was obtained from Zhicheng Plastic Rubber Co. Ltd. (Guangzhou, China). Thermosetting PR plates (thickness, ~0.5 mm) were received from Zhiyou Plastic Rubber Co. Ltd. (Shenzhen, China). Aluminum nitrate ($Al(NO_3)_3$) was purchased from Zhenxin Reagent Factory (Shanghai, China). Calcium chloride ($CaCl_2$) was received from Kermel Chemical Reagent Co. Ltd. (Tianjin, China). Neutral red (NR) was gained from Sanaisi Reagent Co. Ltd. (Shanghai, China). Dimethyl yellow (DMY) was obtained from Beijing Chemical Plant (Beijing, China). Methyl violet (MV) was obtained from Shanghai Specimen Model Factory (Shanghai, China). Methylene blue (MB), glutaraldehyde (GA), anhydrous sodium sulfate (Na_2SO_4), ferric chloride ($FeCl_3 \cdot 6H_2O$), zinc acetate ($Zn(CH_3COO)_2 \cdot 2H_2O$), copper nitrate ($Cu(NO_3)_2 \cdot 3H_2O$), cobalt chloride ($CoCl_2$), nickel chloride ($NiCl_2 \cdot 6H_2O$), potassium ferricyanide ($K_3Fe(CN)_6$) and potassium ferrocyanide ($K_4Fe(CN)_6$) were purchased from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China). Chitosan (CS) was obtained from Ruji Biological Technology Co. Ltd. (Shanghai, China). Glucose oxidase (GOD) was purchased from Sigma. Glucose was got from TCI Development Co. Ltd. (Shanghai, China). Ferrocene formic acid (FcCOOH) was purchased from Fluorochem Ltd. Glass slides and copper sheets were obtained from Xinshenshi Chemical Technology Co. Ltd. (Wuhan, China). Ultrapure deionized water (>18 MΩ cm) was produced on Heal Force (Nison Instrument Ltd., Shanghai, China). Phosphate buffer solutions (PBS, 0.1 M, pH 7.4) were prepared from $Na_2HPO_4 \cdot 12H_2O$ and $NaH_2PO_4 \cdot 2H_2O$ and adjusted to desired pH values with 1.0 M HCl or NaOH by a pH meter (PB-10, Sartorius). Polyethylene terephthalate (PET) films, copper wires, ceramic plates, silicone rubber films, polyvinyl chloride (PVC) films, sticky notes and copper foils were purchased from local markets.

2.2. Instruments

X-ray powder diffraction (XRD) measurements were taken on Bruker D8 Discover equipped with Cu Kα irradiation (k = 1.5406 Å). Transmission electron microscope (TEM) images were characterized by high resolution transmission electron microscope (HRTEM) (FEI tecnai G2 F30, USA). Scanning electron microscope (SEM) images with energy dispersive spectrum (EDS) were collected on a Zeiss sigma field emission system (FEI Nova NanoSEM 450, USA). Raman spectra were obtained on Thermo DXR at an excitation wavelength of 512 nm. X-ray photoelectron spectroscopy (XPS) analysis was performed on Escalab 250Xi. Sheet resistances were measured with a four-point probe sheet resistance tester (ST-21H, China). Fourier transform infrared (FTIR) spectra were collected on a Thermo iS10 spectrometer. UV–vis spectra were performed on a UV 2600 spectrophotometer. A spin coater (KW-4B, China) was used to form various PR films. A 3D printer with laser engraving function (CR-8, Shenzhen Creality 3D Technology Co. Ltd., China) was employed to convert PR into LIG. For comparison, a laser engraving machine equipped with a 50 W CO_2 laser (JL-K3020, Liaocheng Julong Laser Equipment Co., Ltd., China) was also employed for preparing PR-based LIG. All tests of supercapacitors were performed on a CHI 660A analyzer (CH Instruments, Shanghai, China). A Ubbelohde viscometer with an inner diameter of 0.7–0.8 mm and a viscometer constant of 0.0294 mm^2/s^2 at 20 °C was used to test the Kinematic viscosity of PR solutions.

2.3. Preparation of PR solutions

The ethanol solutions of 100, 200, 300, 400, 500 and 600 mg/mL PR were prepared by adding 1, 2, 3, 4, 5 and 6 g of PR powder to 10 mL ethanol, respectively, followed by sonication for several minutes at room temperature to form homogenous solutions. To prepare the doped PR solutions, 20 mg of the dopant was added into 10 mL of PR solution and the solution was sonicated for several minutes. Because of the strong water absorption capacity of $FeCl_3$, the $FeCl_3$-doped PR (PR-Fe) solution should be freshly prepared while the other doped PR solutions can be stably stored under ambient conditions for several days.

2.4. Fabrication of PR-based LIG patterns

The schematic procedures for the construction of PR-based LIG patterns on diverse substrates are shown in Fig. 1a. Briefly, a PET film (4 cm × 4 cm) or other substrate (e.g., glass slides, sticky notes

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