Fabrication and Characterization of Prussian Blue-Derived Iron Carbide-Iron Oxide Hybrid on Reduced Graphene Oxide Nanosheets[†]

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Abstract

This work demonstrates the fabrication of a nanoporous iron carbide-iron oxide/reduced graphene oxide (IC-IO/rGO) hybrid via a controlled one-step thermal treatment of Prussian blue (PB)/GO hybrid at 450 °C under N₂ flow. The PB/GO hybrid is initially prepared through the in-situ deposition of PB nanoparticles on the GO sheets through electrostatic interactions. The morphological analysis of the hybrid reveals the uniform coverage of the rGO sheets by IC-IO nanoparticles and the even distribution of carbon (C), oxygen (O), and iron (Fe) on the rGO nanosheets. As a result of the hybrid composition and controlled morphology, the surface area of the obtained IC-IO/rGO hybrid (~40 m²/g) is significantly enhanced compared to those of the calcined GO sheets and PB nanoparticles (without GO).

Keywords: metal-organic frameworks, porous coordination polymers, metal oxides, metal carbides, graphene oxide

1. Introduction

Metal-organic frameworks (MOFs) or porous coordination polymers (PCPs) have attracted significant interest as porous materials because of their high surface area, large pore volume, controllable composition and pore size, etc. (Wang Z.-L. et al., 2018; Salunkhe R. R. et al., 2016; Kaneti Y. V. et al., 2017). Prussian blue (PB) and its analogues (PBAs) exhibit several attractive characteristics, such as open framework structures, high thermal stability, and good redox activity. These CPs have been employed in numerous applications, including drug delivery (Lin W. et al., 2009), energy storage (Paolella A. et al., 2017), sep-

aration (Bureekaew S. et al., 2008), and so on (Doty F. et al., 2009; Ishizaki M. et al., 2013). Nevertheless, the poor conductivity and low chemical stability of PB presents major challenges for their practical applications (Salunkhe R. R. et al., 2015). Therefore, extensive efforts are needed to enhance the stability and conductivity of PB and its derived materials. To date, PB and PBAs have been employed as novel precursors for obtaining various transition metal compounds, including metal oxides and metal carbides (Azhar A. et al., 2019a). Generally, the calcination of PB in air produces iron oxide (IO) as a result of the removal of the cyano-group and the oxidation of the iron species. Our group previously synthesized PB and completely transformed it into nanoporous iron oxide hybrids (Azhar A. et al., 2019b), such as β -Fe₂O₃ (Machala L. et al., 2013), and mixed crystalline phases of iron oxide (α , γ and β - phases) (Roy X. et al., 2011). On the contrary, when pure PB was heated under inert atmosphere, the formation of metallic iron and iron carbide (IC) (Fe₇C₃, Fe₂C, and Fe₃C) was observed (Zakaria M. B. et al., 2016). Very recently, pure nickel carbide (Ni₃C) was successfully prepared from

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cyano-bridged CPs through a thermal treatment under inert atmosphere at 450 °C (Zakaria M. B. et al., 2019). Despite some progress, the construction of PB-derived metal carbide/carbon hybrids are still rarely reported.

Recently, carbonaceous materials, such as graphite, graphene oxide (GO), reduced graphene oxide (rGO), and carbon nanotubes (CNTs) have been widely used to enhance the functional performance of PB-based materials (Daneshvar F. et al., 2018; Azhar A. et al., 2018). The oxygen-containing functional groups on the surface of the two-dimensional (2D) GO nanosheets can serve as excellent sites for the in-situ growth of PB nanostructures (Lee T. et al., 2015). Our group has demonstrated the fabrication of several cyano-bridged CPs grown on GO nanosheets by a variety of methods (Zakaria M. B. et al., 2019). Herein, we report the synthesis of a hybrid material combining GO sheets with PB nanoparticles which can be subsequently transformed into a nanoporous IC-IO/rGO hybrid under nitrogen atmosphere at 450 °C. The obtained IC-IO/rGO hybrid has been thoroughly characterized in terms of its composition, morphology, thermal decomposition behavior, and textural properties.

2. Experimental

2.1 Chemicals

Sodium ferrocyanide(II) decahydrate ($\text{Na}_4[\text{Fe}(\text{CN})_6] \cdot 10\text{H}_2\text{O}$, $\geq 99\%$) was purchased from Sigma-Aldrich (Japan). The sulfuric acid solution (H_2SO_4 , 98 %) was obtained from Nacalai Tesque (Japan). Potassium hydroxide (KOH), sodium nitrate (NaNO_3 , $\geq 99\%$), and ferric chloride hexahydrate ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, $\geq 98\%$) were sourced from FUJIFILM Wako Corporation (Japan). Graphite nanoplatelets (N008-100-N, thickness ~ 100 nm) were obtained from Angstrom materials (USA). Potassium permanganate (KMnO_4 , $\geq 99\%$) and hydrogen peroxide solution (H_2O_2 , 30 wt.% in H_2O) were purchased from Kanto Chemicals (Japan). All chemicals were utilized as received without further purification.

2.2 Preparation of GO nanosheets

The thin GO nanosheets were fabricated by employing the modified Hummer's approach (Tanaka S. et al., 2017). In the first step, 0.33 g of graphite powder and 0.17 g of NaNO_3 were mixed together and stirred. Following this, 7.67 mL of concentrated H_2SO_4 solution was slowly poured into this suspension and then stirred for 1 h. Next, 1.0 g of KMnO_4 was added into the mixture solution which was placed in an ice bath below 20 °C. The mixture was subsequently stirred at 35 °C for 2 h and distilled water (83 mL) was added into this mixture solution under strong

stirring. After that, 1.67 mL of aqueous H_2O_2 solution (30 % w/w) was added into the suspension. The final GO suspension was washed a number of times with a diluted HCl solution and distilled water. Finally, this GO suspension was sonicated in distilled water to exfoliate the GO sheets. The GO sheets were collected by centrifugation and subjected to repeated washing with distilled water, before being dried at ambient temperature, followed by a final drying in vacuum at 60 °C overnight. The as-prepared GO powder was dispersed in water to prepare an aqueous GO solution (2 mg mL^{-1}) to be used in subsequent steps.

2.3 In-situ deposition of PB nanoparticles on GO nanosheets (PB/GO hybrid) and conversion to iron carbide-iron oxide/rGO hybrid

Typically, 40 mL of 0.299 mM $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ solution was poured into the GO solution (20 mL, 2 mg mL^{-1}) and stirred for 0.5 h. The mixture solution was slowly mixed with 40 mL of $\text{Na}_4[\text{Fe}(\text{CN})_6] \cdot 10\text{H}_2\text{O}$ solution (0.358 mM) and stirred for further 0.5 h, before being aged for two days. The product was isolated via centrifugation and washed multiple times with water and ethanol, and finally dried at room temperature. The porous IC-IO/rGO hybrid was achieved by calcining the PB/GO hybrid at 450 °C for an hour under nitrogen (N_2) flow with a ramping rate of $5 \text{ }^\circ\text{C min}^{-1}$. For comparison, pristine PB nanoparticles without GO sheets were also prepared and heated under the same conditions.

2.4 Characterization

The purity and compositions of the samples were checked by X-ray diffraction (XRD) with a Rigaku RINT 2500X diffractometer utilizing $\text{Cu K}\alpha$ ($1.5406 \text{ \AA}$) radiation. Nitrogen sorption isotherms were collected using a Quantachrome Autosorb at 77 K. To dehydrate the samples, they were subjected to degassing at 250 °C for 16 h prior to the BET measurement. Morphological observations of the products were conducted using both scanning electron microscope (SEM, Hitachi SU8000) and transmission electron microscope (TEM, JEOL JEM-2100F). The infrared (IR) spectra of the samples were collected using a Thermoscientific Nicolet 4700 spectrometer (Waltham, MA, USA). Raman spectroscopy measurements were performed using a Horiba-Jovin Yvon T64000 Raman spectrometer. Thermogravimetry (TG) measurements were carried out with a Hitachi HT-Seiko Instrument Exter 6300 under N_2 atmosphere from 30 to 550 °C at a ramping rate of $5 \text{ }^\circ\text{C min}^{-1}$.

Fig. 1 (a, b) SEM images of (a) GO nanosheets and (b) PB nanoparticles. (c) SEM and (d) TEM images of the as-synthesized PB/GO hybrid.

Fig. 2 Wide-angle XRD patterns of GO, PB, PB/GO hybrid and IC-IO/rGO hybrid. For comparison, the XRD pattern of calcined PB without GO is also shown.

3. Results and discussion

The PB/GO hybrid was obtained by the in-situ deposition of PB nanoparticles on the GO sheets (Azhar A. et al., 2019b). First, the GO sheets were synthesized by the exfoliation of graphite based on the modified Hummer's method (Tanaka S. et al., 2017). The SEM image of the prepared GO sheets (**Fig. 1a**) shows a two-dimensional (2D) crumpled sheet-like structure. The zeta potential measurements reveal the change in the surface charge of the GO sheets from negative to positive charge after the modification with PB nanoparticles. The morphology of the PB/the GO hybrid is depicted in **Fig. 1c**, which shows the wrapping of the PB nanoparticles by the GO sheets (i.e., the surface of the GO sheets is bumpy.). The TEM image further shows that the PB nanoparticles are successfully deposited on the surface of the GO sheets

(**Fig. 1d**). For comparison, pristine PB without GO sheets was also prepared (**Fig. 1b**). **Fig. 2** shows the XRD pattern of the PB/GO hybrid which reveals the diffraction peaks belonging to face-centered cubic (fcc) phase of PB (JCPDS No. 73-0687) (Cao L. et al., 2010). Importantly, after the modification with PB, the primary diffraction peak belonging to the GO nanosheets disappears, possibly due to the nearly complete surface coverage of the GO sheets by the PB nanoparticles (Islam M. N. et al., 2018).

Further compositional analyses of the PB nanoparticles, GO sheets, and PB/GO hybrid were carried out using fourier-transform infrared (FTIR) spectroscopy. The FTIR spectrum of the PB/GO hybrid (**Fig. 3a**) shows the presence of strong bands belonging to the cyano (CN) group of PB ($\text{Fe}^{2+}\text{-CN-Fe}^{3+}$) (Ge C.-X. et al., 2018). The peaks originating from oxygen-containing functional groups (Vermisoglou E. et al., 2014) on the GO surface is significantly reduced following the modification with PB nanoparticles, indicating the complete reduction of GO to reduced graphene oxide (rGO) (Ren S. et al., 2012) and the formation of PB on GO sheets.

The thermal degradation of PB under nitrogen flow was studied by TGA (**Fig. 4**). The TG curve of pure PB shows multi-step weight loss (Sun D. et al., 2019). In the first stage, lattice water and adsorbed water molecules in the structure are removed at temperatures between 30 and 210 °C. The second weight loss (210–420 °C) can be attributed to the loss of the cyano (CN) group present in the PB nanoparticles, and the final weight loss above 420 °C is correlated to the formation of IC. The thermal stability of the pure GO nanosheets under inert atmosphere was also investigated by TG measurements (**Fig. 4**). The evaporation of adsorbed water molecules and the removal of labile oxygen functional groups occur at ~250 °C and no further

Fig. 3 (a) FTIR spectra of GO, PB, PB/GO hybrid, and IC-IO/rGO hybrid. (b) Raman spectra of GO and IC-IO/rGO.

weight loss is observed (Park S. et al., 2011).

As depicted in **Scheme 1**, the thermal treatment of the PB/GO hybrid at 450 °C under N₂ atmosphere results in the formation of the IC-IO/rGO hybrid. The crystal structure of this hybrid after calcination was examined by wide-angle XRD (**Fig. 2**). Some peaks are assignable to iron carbide (Fe₃C, IC) (Fletcher D. et al., 2019), while the other peaks at 35.5° and 62.9° can be attributed to iron oxide (γ-Fe₂O₃) (Zhu K. et al., 2018), suggesting the presence of multiple iron phases after the heat treatment. Compared to the calcined PB (without GO), the relative intensity of the IO peak in the IC-IO/rGO hybrid is increased, which may be due to the reaction of iron species with oxygen functional groups of GO sheets.

Fig. 5a displays the SEM image of the IC-IO/rGO hy-

Fig. 4 TGA data of (a) PB and (b) GO. The measurement was carried out in N₂ atmosphere.

brid, which is composed of irregular and porous structures due to the removal of the organic group after calcination. Also, some IC-IO hybrid nanoparticles can be observed on the surface of the GO nanosheets. The TEM image clearly reveals the uniform decoration of the rGO sheets by IC-IO nanoparticles (**Fig. 5b**). The corresponding elemental mapping images in **Fig. 5c–f** confirm the uniform distribution of carbon (C), oxygen (O), and iron (Fe) on the rGO nanosheets.

The N₂ sorption isotherms of the calcined PB and the IC-IO/rGO hybrid are given in **Fig. 6**. The specific surface area of the IC-IO/rGO hybrid (38.7 m²/g) is significantly larger than those of the calcined GO sheets (1.25 m²/g) (Zakaria M. B. et al., 2019) and calcined PB (18.2 m²/g). This is likely due to the presence of IC-IO nanoparticles which may serve as effective spacers between the GO sheets, thus preventing the stacking of the GO sheets during the thermal treatment.

The FTIR spectrum of the IC-IO/rGO hybrid (**Fig. 3a**) clearly shows the disappearance of many oxygen-containing functional groups, which suggests the successful reduction of GO sheets to reduced GO (rGO) (Liu H. et al., 2015). Additionally, a weak IR band belonging to the CN group is also observed. Raman spectra of the GO sheets before and after the thermal treatment are compared in **Fig. 3b**. The D and G bands are clearly observed. However, the positions of the D and G bands are shifted after the

Scheme 1 Schematic illustration of the in-situ growth of PB nanoparticles on GO sheets (PB/GO hybrid) and the subsequent thermal decomposition to IC-IO/rGO hybrid.

Fig. 5 (a) SEM and (b) TEM images of the IC-IO/rGO hybrid obtained by the thermal treatment of the PB/GO hybrid at 450 °C for 1 h under N₂ atmosphere. (c) HAADF-STEM image of the IC-IO/rGO hybrid and (d–f) the corresponding elemental mapping for (d) carbon, (e) oxygen, and (f) iron.

Fig. 6 (a) N₂ adsorption-desorption isotherms of PB calcined at 450 °C and the IC-IO/rGO hybrid. (b) SEM image of calcined-PB nanoparticles.

thermal treatment. Compared to the I_D/I_G value of pure GO (1.02), the I_D/I_G value of the IC-IO/rGO hybrid is higher (1.12), suggesting the increase of structural defects in the GO sheets after calcination.

4. Conclusions

In summary, we have successfully achieved the in-situ deposition of PB nanoparticles on the surface of GO sheets through the interaction of PB with the oxygen-containing functional groups of GO nanosheets. This PB/GO hybrid can be converted to the IC-IO/rGO hybrid via a one-step thermal treatment at 450 °C under N₂ flow with well-retained morphology. This finding indicates that the presence of rGO during thermal treatment helps to promote the formation of iron carbide at high temperatures. The surface area of the obtained IC-IO/rGO hybrid (~40 m²/g) is superior to those of the calcined GO sheets and the calcined PB without GO. This hybrid material composed of multiple compositions may have many potential applications in supercapacitors and oxygen reduction reaction (ORR).

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