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Synthesis and Catalytic Properties of Polyaniline/Au Hybrid Nanostructure

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Polyaniline (PANI)/Au hybrid nanostructure was synthesized by a simple two-step process without any other templates or additives. Transmission electron microscopy (TEM), field emission scanning electron microscopy (FE-SEM), Fourier transform infrared (FTIR) spectroscopy, and X-ray diffraction (XRD) were employed to study the morphologies and structures of the hybrid. Results indicate that the AuNPs are evenly immobilized on the PANI nanofibers. Notably, the PANI/Au nanostructure exhibits extremely high catalytic activity toward the organic dyes, 4-nitro phenol (4-NP), and methylene blue (MB), which are attributed to stimulation effects of the AuNPs on the electrons transmission of $NaBH_4$ to 4-NP or MB.

Keywords: Polyaniline, Au, Catalytic degradation, Hybrid nanostructure, Nanoparticles

Introduction

In the last decade, One-dimensional (1-D) nanomaterials have drawn considerable attention due to their unique structure, chemical property, unique surface-dominated characteristics, and inherent direct transport path, suitable for catalyst and electricity devise etc. (1, 2). Consequently, the more effective and higher stability 1-D structures have been extensively studied by many professionals. To meet this significant target, various nanostructures, such as organic/metal or organic/inorganic, have been gradually explored. In the current research, a novel one-dimensional nanomaterial with two different combinations was acquired through a two-step process. According to previous literature, the design and fabrication of organic/inorganic hybrid nanostructures with different combinations have demonstrated significant applications in various fields such as electrochemical sensor and catalytic degradation (3).

Due to the highly π-conjugated polymeric chains, polyaniline (PANI) possesses high capacitance, good environmental stability, low cost when combined with other nanomaterial, and exhibits extreme conductivity that is similar to the semiconductor (4). Due to the aforementioned excellent properties, various morphologies of PANI nanostructures have been explored, such as nanospheres and nanorods (5–9). However, the low activity resulting from the poor charge-transport rate led to its low electrochemical and catalytic activities, which can be improved by some functional metal elements. In recent studies, for the higher reduction potential, some novel metal elements with wide ranges of size and morphology can be obtained with the assistance of PANI. More importantly, on the other hand, it has been found that some hybrid structures contain PAIN and novel metals have excellent catalytic activity for organic dyes (10, 11).

Au nanoparticles (AuNPs) have been widely investigated because of their high conductivity and catalytic property that depends on the small size. Loading the AuNPs onto the conducting polymer PANI could stabilize the AuNPs and improve the relative properties (12). AuNP with small size is considered a promising catalyst as it shows good activity during oxygen reduction reaction in alkaline media (e.g., $NaBH_4$) (13). From previous research (14), we know that the PANI exhibit higher reduction potential than Au metal ions; therefore, Au metal ions can be directly reduced to form AuNPs by PANI. On the other hand, thioglycollic acid (TA) was used as a dopant to make a graft polymerization with PANI fibers to form an –SH group that can combine with AuNPs (15). Due to the catalyst of AuNP and the excellent electric conductivity of PANI, the hybrid structure has been demonstrated to be an efficient catalyst toward organic synthesis and organic dyes. In the present investigation, the PANI/Au hybrid nanostructure was synthesized by a simple two-step process without any other templates or additives; the morphology and structures of the hybrid were investigated and characterized. The catalytic properties of the synthesized hybrid nanostructure were demonstrated by the degradation of two organic dyes, 4-nitrophenol (4-NP) and methylene blue (MB).

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Experimetal Section

Materials

Aniline, thioglycolic acid (TA), and hydrochloric acid (HCl) were all purchased from Aladdin Chemistry Co., Ltd. Ammonium persulfate (APS) and chloroautic acid ($HAuCl_4 \cdot 4H_2O$, 99.9 %) were acquired from Shanghai Civi Chemical Technology Co., Ltd, Methyl blue (MB) was purchased from Tianjin Yongda Chemical Reagent Co., Ltd.; 4-nitrophenol (4-NP) was obtained from Shanghai Lingfeng Chemical Reagent Co., Ltd.; and all the reagents were used without further purification. Deionized water (DIW, 18.2 MΩ) was used for all solution preparations.

Methods

Synthesis of the PANI Nanofiber

In a typical procedure, 0.5 g anline monomer and 0.6 g APS were added into a 50-mL beaker that contained 1.0 M 10 mL HCl solution. Then, the mixed solution was vigorously stirred for 30 seconds; afterward, the deep green solution reacted for 2 hours without stirring under room temperature. Then, the as-prepared Cl^--doped PANI was washed with deionized water three times and the filtered product was redispersed in the excess 1.0 M $NH_3 \cdot H_2O$ solution for doping. The as-prepared NH_4^+-doped PANI was first washed by deionized water and then alcohol for three times, respectively. The precipitate was dried in 50°C for 36 hours.

Synthesis of the PANI/Au Hybrid Structure

In a typical procedure, 0.6 g TA was added into a mixed solution containing 0.2 g as-prepared PANI nanofibers, maintaining the reaction without stirring for 1 hour. Then, the solution was filtered and washed with water and alcohol several times and redispersed in deionized water. Then, 5.0 mL $HAuCl_4$ solution was drop-wise added into the beaker and the mixed solution reacted for 24 hours under slight stirring. Finally, the aforementioned deeply black solution was filtered and washed several times with water and alcohol, and the precipitate was dried at 50°C for 48 hours.

Catalytic Activity Measurements

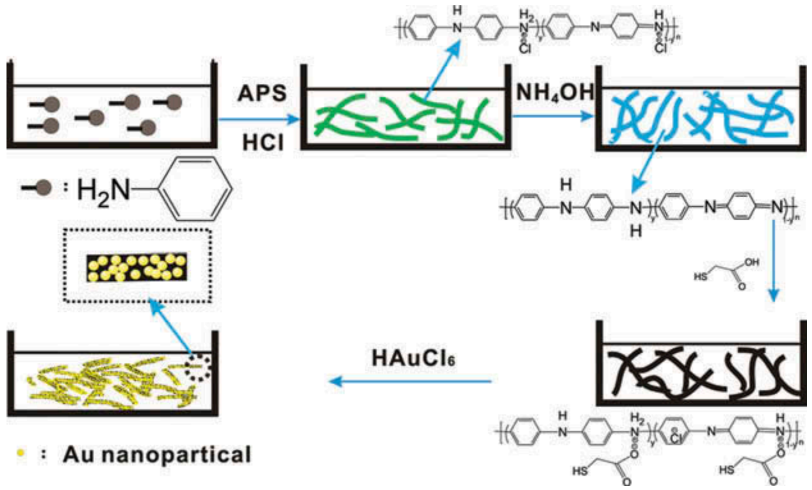
The 4-NP (4-nitro phenol) and MB (methylene blue) were employed as the organic dyes to test the catalytic degradation activity. In a typical process, 25 mL 4-NP and MB were blended with 25 mL deionized water, and then 20 mL (0.083mol/L) $NaBH_4$ solution was added into the organic solution as assist-catalyst. The 4-mL solution was removed for UV measurements to obtain the initial data. The 15 mg and 5 mg catalysts were respectively added into the samples, and a 4-mL solution was removed and filtered to eliminate the catalyst for UV-Vis measurements when the time interval was set. Meanwhile, to eliminate the absorptive effect of PANI fiber, pure PANI nanofibers were chosen as the control for the aforementioned catalyst degradation measurement.

Insrumentation

Field emission scanning electron microscopy (FE-SEM) images of the synthesized PANI and PANI/Au hybrid structures were obtained using a JSM-6700F field-emission scanning electron microscope (JEOL, Japan). Transmission electron microscope (TEM) characterization was performed on a JEOL JEM-2100 electron microscope operating at 200 KV. X-ray diffractometer using a Cu K_α radiation source at 35KV, with a scan rate of 0.02° 2θ s^{-1} in the 2θ range of 10–80°. UV-Vis absorption spectra were acquired with the use of a Lambda 900 UV-Vis spectrophotometer (Perkin Elmer, USA). Fourier transform infrared (FTIR) spectra were recorded on a Nicolet 5700 FTIR spectrometer in transmittance mode at a resolution of 4 cm^{-1} and 32 scans.

Results and Discussion

Here, in order to achieve this hybrid nanostructure, the process includes two steps (Scheme 1): (a) The obtainment of TA-doped PANI nanofibers: PANI nanofibers were obtained from the aniline through the oxidation-catalyze of APS in the HCl solution, which eliminated the doping effect of Cl^- in the excess



Scheme 1 Schematic illustration of the achievement of PANI/Au hybrid nanostructure.

$NH_3.H_2O$ environment. Then, TA was added to dope the PANI polymer to achieve the TA-doped PANI fibers through the electric force between the N atom and the -OH group on TA. (b) The achievement of the PANI/Au hybrid nanostructure: $HAuCl_4$ was employed as the oxidant for this polymerization. The PANI/Au hybrid nanostructure was obtained by the reduction effect of PANI and the combination between –SH and AuNPs. Meanwhile, with the assistance of TA, the dispersion of AuNPs can be easily controlled.

From the SEM and TEM images of the obtained powder from Fig. 1(a) and Fig. 1(c), PANI nanofibers with a diameter size of approximately 50 nm were successfully synthesized. Compared to Fig. 1(a), the SEM image of PANI/Au hybrid structure had no significant difference in morphology (diameter and length) compared with PANI nanofibers (Fig 1b). As shown in a magnified TEM image (Fig 1d), there are numerous "black dots" on the surface of the PANI that reveal actual crystallized AuNPs with an average size of 5 nm (16). It indicates that when aniline is under oxidative polymerization, $AuCl_4^-$ can be reduced to Au nucleates deposited on the PAIN nanofibers. From Fig. 1(d), the HRTEM image of the PANI /Au nanofibers reveals distinct lattice fringes with d spacings of 0.23 nm, which corresponds to the (111) lattice planes of face-centered cubic (fcc) gold (5–7).

To further confirm the presence of the PANI and analyze the crystal phase structures, the pure PANI and PANI/Au hybrid structure were characterized by XRD. XRD patterns of the PANI nanofibers and PANI/Au hybrid nanostructures are shown in Fig. 2. As shown in Fig. 2, the XRD pattern of pure PANI is amorphous and has a broad band at 22–32°, which is ascribed to the periodicity parallel to the polymer chains of PANI (3). As shown in the XRD pattern of PANI/Au hybrid nanostructures, all the diffraction peaks located at 38.1°, 44.3°, 64.5°, and 77.58° can be considered an Au crystal (JCPDS: 65-2870), which correspond to the (111), (200), (220), and (311) planes of fcc gold, respectively (7, 8, 12–16). Compared to the two patterns in Fig. 2, it is concluded that large numbers of AuNPs were synthesized and covered on the surface of PANI nanofibers, which caused the XRD weak signals of PANI peaks, due to the interference.

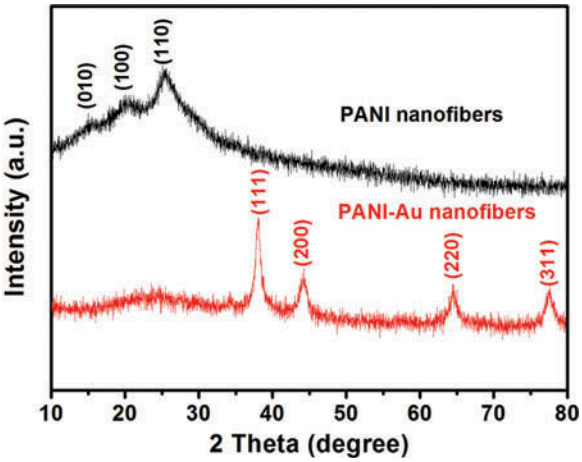


Fig. 2. XRD patterns of the pure PANI nanofibers and PANI/Au hybrid structure.

As shown in Fig. 3, for pure PANI (Fig. 3a), the peaks in the range of 2900–3500 cm^{-1} wavenumbers are ascribed to stretching vibrations of the leucoemeraldine component. The peak at 1588 cm^{-1} and 1498 cm^{-1} are related to the C=C stretching vibration of benzenoid and quinoid rings, respectively. The peak at 1296 cm^{-1} was ascribed to the C-N stretching vibration with aromatic conjugation (17). The peak at 1139 cm^{-1} belongs to the characteristic $A{=}NH^+$ -B (where A and B denote quinoid ring and benzene ring, respectively) (18, 19). In addition, from the Fig. 3(a), we found the intact peak intensity of PANI/Au was stronger than that of pure PANI, which meant that the structure of PANI in the composite material had a higher conjugation length (6).

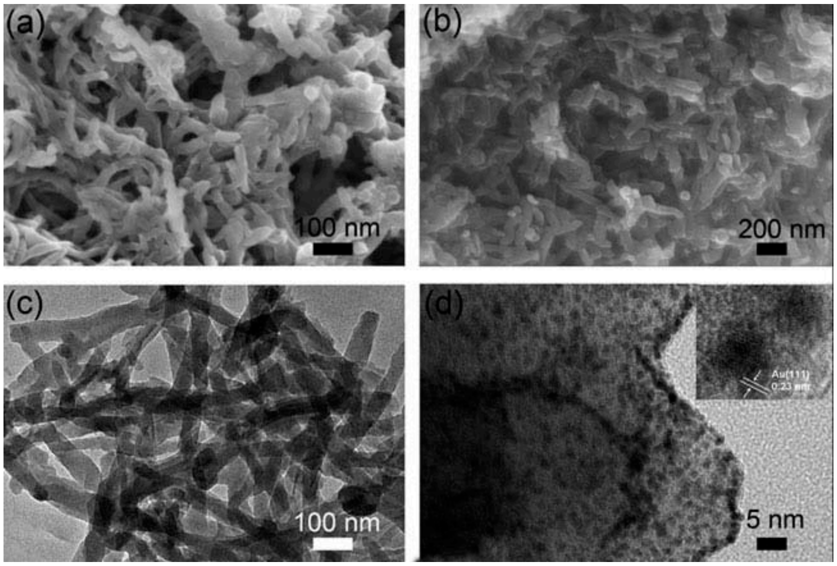


Fig. 1. FE-SEM and TEM images of the PANI (a, c) and PANI /Au (b, d) nanofibers. The inset is the corresponding image of HRTEM images.

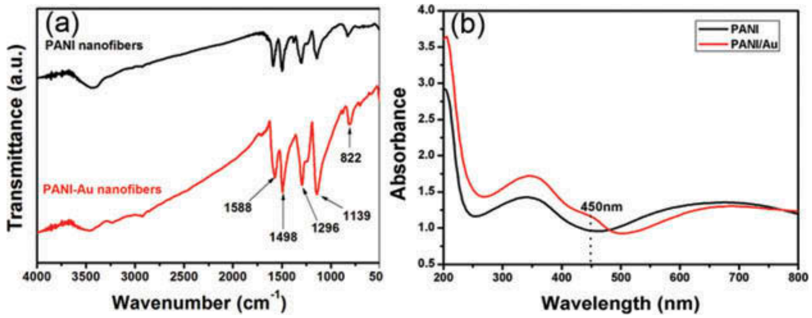


Fig. 3. (a) FT-IR spectra of pure PANI and PANI/Au hybrid nanostructure and (b) UV-vis spectra of pure PANI and PANI/Au hybrid nanostructure.

UV-vis absorption spectra of pure PANI and PANI/Au hybrid nanostructure are shown in Fig. 3(b), the characteristic bands of pure PANI appear at 340 nm and 681 nm, which are attributed to the π - π^* transition of benzenoid rings and polaron- π^* transition, respectively (20, 21). In comparison with the UV-vis spectrum of the PANI nanofibers (Fig. 3b), a distinct peak emerged around 450 nm, which is ascribed to the SPR peak of AuNPs. The hypochromatic shift in the UV-Vis spectrum is related to chemical changes in the environment. Thus, the observed shift here would be in accordance with the reduction of Au ions through PANI nanofibers, followed by structural changes in the PANI polymer chains. In addition, compared with the pure PANI, a red shift of the absorption band of PANI/Au hybrid mainly occurs at 681 nm, which is linked to this band as a function of structural and oxidative changes (14). For this reason, Au metal ions can be successfully reduced to AuNPs by PANI and well-dispersed on the surface of PANI fibers with the assistance of TA (22). After the growth of AuNPs on PANI nanofibers, the binding energies of the N 1s changed, indicating the redox reaction between PANI and HAuCl$_4$. In the redox reactions, the benzenoid amines (-NH-) were oxidized to quinonoid imine (=N-), whereas the AuCl$_4^-$ were reduced to AuNPs (22).

In order to explore the catalyst activity of the PANI/Au hybrid nanostructure toward organic dye and synthesized polymer, the reduction of 4-NP and MB in the presence of NaBH$_4$ was demonstrated. In the absence of catalysts, the mixtures of 4NP and NaBH$_4$ showed an adsorption band at $\lambda_{\max} = 400$ nm, corresponding to the 4NP ions in alkaline conditions. The 4-NP and MB will not be degraded only from the effect of NaBH$_4$ as can be evidenced from previous literature (23). As a type of catalyst with high efficiency, AuNPs can stimulate the electrons of NaBH$_4$ transmitted to 4-NP or MB when the size is less than 5 nm. When the catalyst is added, the reduction of 4-NP or MB occurs immediately, accompanying the vanishing of the color. Time-dependent adsorption spectra of this reaction mixture show the disappearance of the peak at 400 nm that was accompanied by a gradual development of a new peak at 300 nm corresponding to the formation of 4AP (Fig. 4a). It can be seen that the reduction of 4NP to 4AP can finished within a few minutes with gold catalysts of small size. As shown in Fig. 4(c), the typical UV-Vis absorption spectra of the reaction progress between catalyst and organic dyes at approximately 3 min are demonstrated. Fig. 4(a) and 4(c) show obvious absorption of 4-NP and MB that nearly reduces to zero with the existence of the PANI/Au hybrid nanostructure during the degradation reaction. Through theoretic calculation, the degradation ratios of 4-NP and MB almost approach 99.0% and 99.6% in 3 min, respectively. A controlled comparative experiment of the degradation of 4-NP and MB was conducted to understand the role of AuNPs in enhancing the electron-holes separation. Fig. 4(b) and 4(d) show the absorbance of the organic dyes decreases minimally when PANI nanofibers were used as the catalyst, which was ascribed to the absorption of PANI nanofibers. Meanwhile, comparing Fig 4(a) with 4(b), during the reduction progress of 4-NP, the intensity of p-aminophenol (300 cm^{-1}) was gradually increased, which might certify the degradation reaction occurred (9). The aforementioned characterizations and results indicate high efficiency of the synthesized PANI/Au hybrid nanostructure and the reason may lie in the stimulation effects of AuNP on the electrons of NaBH$_4$ transmitted to 4-NP or MB (24).

Conclusions

In summary, a facile and simple approach to obtain PANI/Au hybrid nanostructure was explored. Through the assistance of TA, AuNPs were evenly grown onto the surfaces of the as-prepared PANI nanofibers to achieve a PANI/Au hybrid nanostructure. FE-SEM, TEM, XRD, FTIR, and UV-Vis were employed to investigate the morphology and crystalline structure of the novel nanostructure. The prepared PANI/Au showed excellent degradation efficiency for the reduction of 4-NP and MB by NaBH$_4$ and the complete vanishment of dyes can be reached in several minutes.

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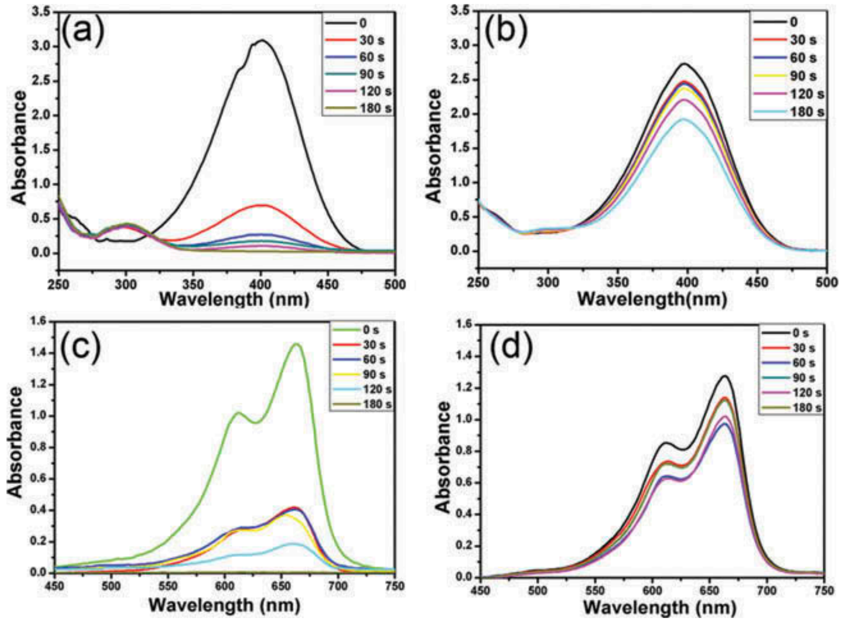

Fig. 4. (a, b) PANI/u an pure PANI in the degradation of 4-NP and (c, d) PANI/u an pure PANI in the degradation of MB.

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