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Synthesis of Au decorated V_2O_5 microflowers with enhanced sensing properties towards amines

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ABSTRACT

This study developed an innovative two-step method to synthesize hybrid gold decorated nanosheet-assembled V_2O_5 microflowers (Au/V_2O_5), through in-situ reduction of Au nanoparticles on nanosheet-assembled V_2O_3 microflowers at room temperature in aqueous solution and further thermal oxidization as V_2O_5. Various characterization techniques were employed, such as SEM, TEM, XRD, EDX, XPS, and BET, to reveal that Au nanoparticles (diameter of ~10 nm) evenly attached on the surface of nanosheet-assembled V_2O_5 nanoparticles (diameters of ~1–2 μm). This method shows several advantages in generating such nanocomposites: low cost, highly efficient, room-temperature and easy operation, as well as no need for extra reducing agents and surfactants. The gas sensing properties of the Au/V_2O_5 composites were investigated toward toxic 1-butylamine, an important marker compound in food and medical industries, showing that the Au modification can effectively enhance the sensing performance: high response (100 ppm of 1-butylamine), high selectivity, and low working temperature (~240 °C), compared to bare V_2O_5 microflowers. The effect of the molar ratio of Au to V_2O_5 on nanostructure and sensing property was also investigated. The findings may bring a new insight into the fabrication of noble metal(s) modified V_2O_5 composites with high sensing performance.

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1. Introduction

Semiconducting (chemiresistance) gas sensors have gained special attention in fundamental science and practice for sensing harmful gases owing to their low manufacturing cost, easy processing, and reliable performance [1, 2]. Various conventional metal oxides, such as vanadium oxide (V_2O_5) [3], CuO [4], zinc oxide (ZnO) [5–8], tin oxide (SnO_2) [9, 10], iron oxide (Fe_2O_3) [11–13], nickel oxide (NiO) [14], tungsten oxide (WO_3) [15, 16] and indium oxide (In_2O_3) [17], exhibit good sensing performance. Even some of them have been used for diagnosis of disease [18]. On the other hand, the performance is dependent upon the surface properties of the materials due to the sensing mechanism [19]. Thus, the metal oxide particles in nanoscale with various morphologies are of significant interest.

V_2O_5, an important *n*-type semiconductor, has been intensively studied as sensing materials recently due to high stability, low cost, and high resistance to corrosion [20, 21]. The sensing mechanism of metal oxide semiconductors is based on the change in resistance during the adsorption and desorption process between the target gas molecules and the surface of sensors. For an *n*-type semiconductor, metal oxides adsorb ionized oxygen species which can trap electrons from the conduction band of metal oxide nanoparticles and increase the resistance when exposed to air. When target gases are introduced, the adsorbed oxygen and oxygen vacancies can react with the gas molecules which are chemically adsorbed at the active sites on the surface of sensing materials. In this process, the trapped electrons are released and transferred to the conduction band of semiconductors, resulting in the decrease of the resistance. Therefore, the surface area plays an important role in the sensing process, since the surface reactions are directly dependent on the oxygen vacancies with active centers and defects. Recently, the sensors based on V_2O_5 have been widely reported. For example, Schneider et al. prepared a V_2O_5 thin film (thickness of 200–600 nm) by a reactive sputtering method. These thin films exhibit good responses toward hydrogen, methane and propane [22]. Dhayal Raj et al. reported self-assembled V_2O_5 nanorods prepared by a solvothermal method, which presented good sensitivity toward ethanol

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[23]. Raible et al. synthesized V_2O_5 nanofibers by a hydrothermal method. The nanofibers exhibit excellent response toward 1-butylamine [24].

However, pure V_2O_5 sensors typically suffer from relatively low sensitivity and selectivity, largely impeding their broader applications [25]. To enhance the sensing performance, many efforts have been made in this area, such as size and morphology control [3, 19, 24–29], metal oxide additives [30–32], and noble metal sensitizers [33]. These ways have been proved to improve the sensing performance to various extents, especially for modification of noble metal, which is proposed to be an effective way to improve the functional properties of metal oxides because of spillover effect and tunable band structure by combination of noble metal and metal oxides nanoparticles [34]. The most popular way to prepare noble metals-metal oxide nanocomposites is usually to add reducing agent solution into the suspension containing noble metal ions and metal oxide nanoparticles [5, 34]. However, doping noble metals (Au, Ag, Pt, and Pd) by an *in-situ* reduction method is of a challenge for V_2O_5 particles. The main reason is due to the higher redox potential of V_2O_5 than noble metals ($E^0_{V_2O_5/VO^{2+}} = 0.957$ V, $E^0_{VO^{2+}/V^{3+}} = 0.337$ V, $E^0_{Ag^+/Ag} = 0.779$ V, $E^0_{AuCl_4^-/Au} = 1.001$ V) [35], which means V_2O_5 will be preferentially reduced and the reducing reaction on noble metal will not happen. Therefore, the methods for synthesis of such composite are concentrated on pulsed laser deposition (PLD) [36], emulsion-electrospinning [33] or chemical vaper deposition (CVD) method [37], which are expensive and complicated. Specifically, Liang et al. reported an Au-decorated VOx thin film prepared by ion sputtering Au metal on VOx thin film (produced by DC-magnetron sputtering of V metal), followed by rapid thermal annealing in O_2 from 470 to 500 °C [38]. Furthermore, hollow Au/V_2O_5 nanotubes were synthesized by emulsion electrospinning (polystyrene as the template) and a post-calcination treatment [33]. However, the nanotube structures are formed by assembled V_2O_5 nanoparticles due to the removal of the template and organic solvent by calcination, rather than formed by reeling of V_2O_5 nanosheets. Thus, this causes the fact that the Au nanoparticles inlaid among the V_2O_5 nanoparticles, instead of attached on the surface of nanotubes. Therefore, the method for simple and low-cost preparation of noble metal decorated V_2O_5 nanocomposites is still required to further study.

On the other hand, detecting organic amines by chemiresistor gradually attracts considerable attention due to their toxicity and widespread application in food industries and medical diagnosis [39]. Compared with the current sensing techniques (e.g., isotachophoresis and high-pressure liquid chromatography (HPLC)) [40], chemiresistor shows several advantages, such as fast response, low cost, high stability and easy operation. Therefore, some sensors based on semiconductor metal oxide have been reported. For instance, Shah et al. demonstrates that V_2O_5/In_2O_3 core-shell nanorods are highly sensitive to *n*-propylamine with the sensitivity of ~14, but suffering from long response and recovery time [30]. Recently, silver vanadate nanoparticles ($Ag_{0.35}V_2O_5$ and $Ag_2V_4O_{11}$ nanobelts) have been used for sensing organic amines. Despite some success, limitations, such as low response and high working temperature, still exist [41, 42].

Herein, to overcome this problem, we demonstrate a novel and simple method to prepare the Au-decorated V_2O_5 particles for the first time. That is, noble metal ions are added to a V_2O_3 suspension at room temperature. The noble metal ions can be reduced by V_2O_3 particles in aqueous solution, and noble metal particles can be directly attached on the V_2O_3 particles, formed nanocomposites. In the end, noble metal-doped V_2O_5 can be obtained by calcinating the precursors in the air at 400 °C. This method shows several merits, such as low cost, room-temperature and easy operation, as well as no need for addictive. The products were characterized by several advanced techniques, including transmission electron microscopy (TEM), high-resolution TEM (HRTEM), scanning electron microscopy (SEM), X-ray diffraction (XRD), X-ray photoelectron spectroscopy (XPS), as well as N_2 sorption isothermals. The effect of the different amounts of Au decoration on the sensing properties of V_2O_5 particles toward 1-butylamine and other gases were evaluated. Finally, the possible mechanisms for achieving high sensing performance of such nanostructures were discussed.

2. Experimental

2.1. Synthesis of nanosheet-assembled V_2O_3 microflowers

The nanosheet-assembled V_2O_3 microflowers were prepared according to our previous work [3]. Specifically, V_2O_5 (0.14 g) and various amounts of $NaHCO_3$ (1.25 M) were added in 16 ml of EG with vigorous stirring at 60 °C for several hours until the color of the suspension changed from brownish yellow to light yellow. Then, 20 ml of such obtained suspension was added into a 50 ml Teflon container. After the container was sealed in an autoclave, it was heated at 260 °C for 24 h. After cooled down to room temperature naturally, the resulting black precipitate was collected by centrifugation, washed thoroughly with deionized water and ethanol several times and finally dried at 70 °C overnight before further use. Finally, the precipitate was sintered at 400 in the atmosphere of air for 2 h.

2.2. Synthesis of Au nanoparticles-decorated V_2O_5 microflowers

0.03 g of V_2O_3 microflowers was well-sonicated in 8 ml of deionized water for 30 min. Varying quantities of a 0.01 M solution of $HAuCl_4 \cdot 3H_2O$ were added into the V_2O_3 suspension at room temperature under magnetic stirring to achieve Au/V_2O_3 nanocomposites with four different Au loading. The molar ratios of Au to V are 0.5%, 1.5%, 2.5%, 3.5%, and 5%, and the Au content characterized by EDS is shown in Table 1. The blackish cotton-like precipitates quickly formed and were deposited to the bottom. The precipitates were thoroughly washed with DI water and ethanol several times until the supernatant was clear, then collected by centrifuge and dried in an oven at 70 °C for 4 h. Finally, the precipitate was sintered at 400 °C in the atmosphere of air for 2 h. After cooling down to room temperature, the product was collected for further use.

2.3. Characterization

The phase composition and purity of the synthesized particles were examined using Phillips X'pert Multipurpose X-ray Diffraction System (MPD) equipped with graphite mono-chromatized Cu-Kα radiation (λ = 1.54 Å) in the 2θ range of 10–80°. To further identify the elemental composition, XPS analysis was performed with a Physical Electronics PHI 5000 Versa probe spectrometer with Al Kα radiation (1486 eV).

Table 1
The molar ratios of Au to V characterized by EDS.

Molar ratios of Au/V		0.42%		1.47%		2.45%		3.46%		4.97%	
Element	Series	Wt%	At.%	Wt%	At.%	Wt%	At.%	Wt%	At.%	Wt%	At.%
Carbon	K-series	10.51	20.77	10.30	20.48	8.43	17.17	10.94	21.87	9.42	19.31
Vanadium	K-series	14.46	6.72	18.01	8.43	14.66	7.03	24.09	11.33	20.10	9.70
Oxygen	K-series	11.24	16.66	14.00	20.88	11.66	17.82	18.94	28.39	15.59	23.98
Aluminum	K-series	63.55	55.82	56.67	50.09	63.86	57.81	42.81	38.02	51.04	46.53
Gold	M-series	0.23	0.03	1.02	0.12	1.39	0.17	3.22	0.39	3.86	0.48

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