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Preparation and humidity sensing properties of Fe-doped mesoporous silica SBA-15

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ABSTRACT

Fe-doped mesoporous silica SBA-15 has been studied as a humidity sensing material. Comparing with pure SBA-15, Fe-doped SBA-15 shows improved humidity sensing properties within the relative humidity (RH) range of 11–95%. The impedance of our product changes by more than four orders of magnitude over the whole humidity range with the response time and recovery time are about 20 and 50 s, respectively. High selectivity and good stability are also observed based on our product. These results make Fe-doped SBA-15 a good candidate in fabricating humidity sensor.

1. Introduction

Increasing demands for ever more sensitive chemical sensors for air-quality detection, environmental monitoring, inflammable-gas inspection, healthcare, industrial production, defense and security, and other applications have led to an upsurge of research devoted to the development of new sensing materials [1–5]. Many scientific and technological efforts to develop sensing materials are focused on improving their overall properties. The new generation sensing materials should exhibit a wide operation temperature range, high sensitivity, rapid speed, good accuracy, reproducibility, durability, small hysteresis loop, easy processing and low cost. Recently, mesoporous materials, based on their structural characteristics (ordered pore distributions, high pore volumes and high surface areas [6–9]) combined with the possibility to process them in various shapes (calibrated spherical powders, thin films, membranes, and monoliths [10–13]), have attracted much research interest in developing its potential applications in optoelectronics [14–16], catalysis [17–20], and sensors [21–26]. Till now, many mesoporous materials have been synthesized and applied in sensing materials [21–26]. The most important property for the sensing applications of mesoporous materials is represented by the accessibility from the external environment of the mesophase, which has a large interfacial area [27].

Herein, we present a simple and effective route for the synthesis of a highly efficient humidity sensing material of Fe-doped SBA-15. SBA-15 has been chosen as the substrate material for its large surface areas and very uniform mesoporous structures [28–29]. Fe has been chosen as the dopant for its excellent sensing properties [30–32]. Compared with the pure SBA-15, Fe-doped SBA-15 presents much better linearity of the impedance versus relative humidity (RH) curve in the whole range of 11–95% RH, and it also presents quick response, high selectivity and good stability. In addition, a possible mechanism is provided to explain the humidity sensitive properties.

2. Experimental

2.1. Preparation of materials

Hydrochloric acid (HCl) and tetraethyl orthosilicate (TEOS) of analytical grade were purchased from Tianjin Chemical Co. (China). $EO_{20}PO_{70}EO_{20}$ (Pluronic P123) was purchased from Aldrich. The deionized water with a resistivity of 18.0 MΩ cm^{-1} was used in all experiments.

Mesoporous silica SBA-15 was synthesized as reported by Zhao et al. [33–34] using triblock copolymer P123. In a typical synthesis procedure, 4 g of P123 was dissolved in 30 g of water and 120 g

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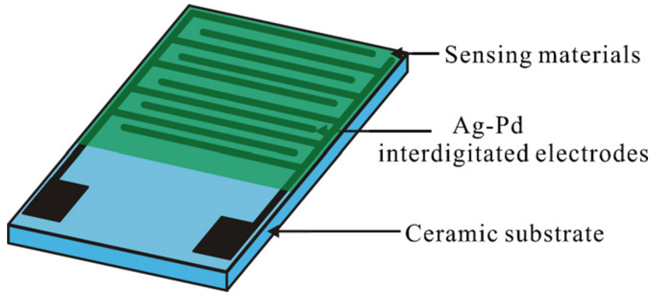

Fig. 1. A schematic image of the humidity sensor.

of 2 M HCl solution by stirring. Then 8.50 g of TEOS was added, and the resulting mixture was stirred for 5 min, and then kept at 40 °C for 20 h without stirring. It was followed by aging for 2 days at 100 °C under static condition. Then the solid products were filtrated and washed repeatedly with deionized water. After drying at room temperature (25 °C) overnight, the product was sintered in air at 550 °C for 5 h to remove the organic template and obtain SBA-15.

The sensing materials of Fe-doped SBA-15 were prepared by mixing different weight ratios of $Fe(NO_3)_3$ and SBA-15, and grounding in an agate mortar for 1 h. The products were designated as Fe-SBA-15(*X*), where *X* was the content of $Fe(NO_3)_3$ in 1 g of SBA-15. In our experiment, the value of *X* was 0.1, 0.3, 0.5, 0.8 and 1.0 for five Fe-SBA-15 samples, respectively.

2.2. Measurement

X-ray diffraction (XRD) data were obtained with a Siemens D5005 diffractometer (40 kV, 30 mA) using Cu-Kα radiation with a wavelength of $\lambda = 0.15418$ nm, diffraction patterns were collected under ambient conditions in the 2θ range of 0.6–4.0° and 4–80°. Infrared (IR) spectroscopy was performed on a Bruker IFS 66 v/S infrared spectrometer in the range 400–4000 cm^{-1} using KBr pellets in vacuo (<0.4 Pa) at 25 °C. The nitrogen isotherms at the temperature of liquid nitrogen were measured using a Micromeritics ASAP 2020 M system. The samples were outgassed for 10 h at 300 °C before the measurements. The specific surface area, S_{BET}, was determined from the linear part of the BET plot. The average pore size was the peak value on the pore size distribution (PSD), which was calculated from the adsorption branch using the BJH method. Transmission electron microscopy (TEM) experiments were performed on a JEM-3010F electron microscope (JEOL, Japan) with an acceleration voltage of 300 kV.

To study the humidity sensing properties of pure and Fe-doped SBA-15, each sample was mixed and ground with deionized water in a weight ratio of 100:25 to form a paste. The paste was screen-printed on a ceramic substrate (6 mm × 3 mm, 0.5 mm in thick) with five pairs of Ag–Pd interdigitated electrodes (electrodes width and distance: 0.15 mm) to form a film with the thickness about 200 μm, and then the film was dried in air at 60 °C for 5 h. Finally,

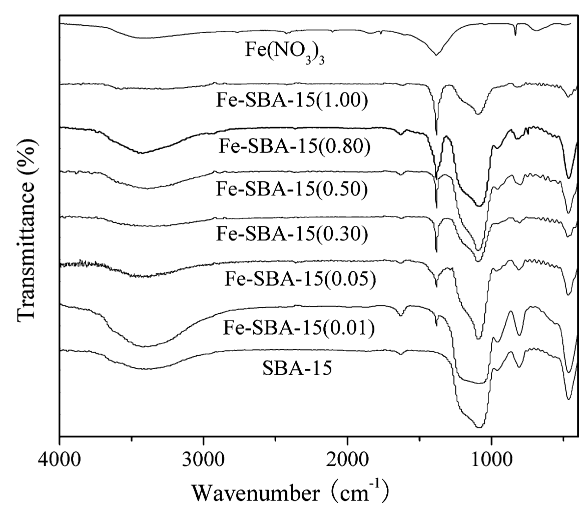

Fig. 3. IR spectra of SBA-15, Fe-SBA-15(*X*) (*X* = 0.1, 0.3, 0.5, 0.8 and 1.0, respectively), and $Fe(NO_3)_3$.

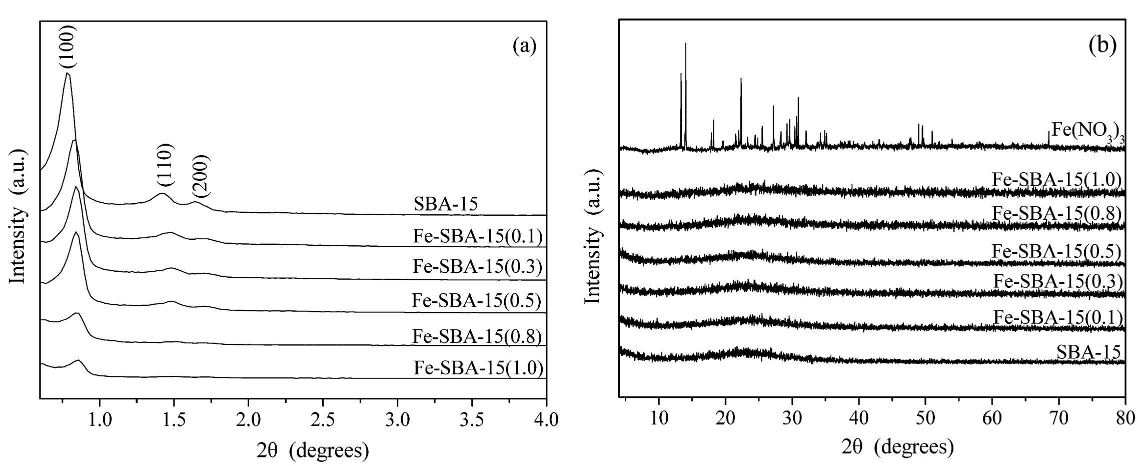

Fig. 2. (a) Low-angle XRD patterns of SBA-15 and Fe-SBA-15(*X*) (*X* = 0.1, 0.3, 0.5, 0.8 and 1.0, respectively) and (b) wide-angle XRD patterns of SBA-15, Fe-SBA-15(*X*) (*X* = 0.1, 0.3, 0.5, 0.8 and 1.0, respectively), and $Fe(NO_3)_3$.