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Synthesis, Characterization and Sensing Behavior of WO_3 Nanocrystalline Powder for Toluene Vapor

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Tungsten oxide (WO_3) nanocrystalline powder was successfully synthesized by direct precipitation technique starting from sodium tungstate dehydrate ($Na_2WO_4{\cdot}2H_2O$) as precursor at 80 °C for 48 h. The morphological characteristics, distribution, and crystallite size of WO_3 nanocrystalline powder were analysed by transmission electron microscopy, X-ray diffraction, and the Fourier transform infrared spectroscopy. According to transmission electron microscopy results, the mean particle size of WO_3 nanocrystalline powder was approximately determined as 41.7 nm. The gas sensing performance of WO_3 nanocrystalline powder for saturated toluene vapor was measured by two probe resistivity unit in a closed vessel at room temperature. In addition, WO_3 nanocrystalline powder was able to respond the lowest concentration of 500 ppm. The results showed that the sample exhibited high sensitivity (93%) and good response reproducibility ($0.93 + 5.5\%$) for toluene vapor.

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PACS/topics: tungsten oxide, direct precipitation technique, toluene

1. Introduction

Among the volatile organic compounds (VOCs), toluene is one of the most dangerous air pollutants because of its carcinogenicity property [1]. Therefore, improving the detection materials towards toluene is of great significance for human health and environmental protection [2]. Nanostructured materials and nanocrystalline metal oxides have attracted increasing attention in various areas of material engineering due to magnetic, mechanical, chemical, and physical properties. In addition, metal oxides have been widely used as gas sensing materials for improving the performance of gas sensors [3, 4]. Among these, tungsten oxide (WO_3) which is a wide band gap n-type semiconductor material is a potential candidate and has attracted considerable attention due to high sensitivity for reducing and oxidizing gases [5, 6].

This paper deals with the synthesized WO_3 nanocrystalline powder by direct precipitation technique without surfactant and the sensing properties of the obtained nanomaterial toward different concentrations of toluene vapor.

2. Experimental

WO_3 nanocrystalline powder was synthesized by direct precipitation technique starting from 0.5 M $Na_2WO_4{\cdot}2H_2O$ in HCl solution of pH 1 and then the solution was magnetically stirred for 48 h in oil bath in a water-cooled condenser at 80 °C reaction temperature. After the obtained precipitate was filtered and washed several times with deionized water and absolute ethanol, the final product was dried at 60 °C for 24 h and calcined at 500 °C for 4 h.

For gas sensing test, the synthesized sample pellet which was pre-prepared by keeping it under 7 t pressure for 3 min, was exposed to the toluene vapors in a glass closed test chamber. The gas sensing measurement was performed using two-probe method with a Thurlby electrometer [7] and the gas response R (%) is defined as $(R_0-R_g)/R_0 = (\Delta R/R) \times 100$, where R_0 and R_g are the sample resistances of the pellet with air and the presence of toluene vapor, respectively.

3. Result and discussion

3.1. Characterization

Figure 1a shows the X-ray diffraction (XRD) pattern of WO_3 nanocrystalline powder prepared by direct precipitation technique. The synthesized WO_3 is in a monoclinic phase. It can be clearly seen that the diffraction peaks at 2 theta (23.12, 23.59, and 24.24), which belong to characteristic of WO_3 monoclinic phase, are well distinct and sharp [8]. In Fig. 1b, the Fourier transform infrared (FTIR) spectrum displayed the broad bands between 592 and 946 cm^{-1}, which corresponded to the stretching of short W = O bonds while the peak at 815 cm^{-1} was ascribed to the O–W–O stretching modes [9].

The morphology of WO_3 nanocrystalline powder was observed by transmission electron microscopy (TEM). As seen from Fig. 2, the particle size of WO_3 has approximately determined as 41.7 nm with the Uthscsa Image Tool 3 analysis program through 90 particles.

3.2. Gas sensing properties

We have studied the gas sensing properties of WO_3 nanocrystalline powder to detect toluene gas with different concentrations. The gas sensing properties are shown in Fig. 3. The response of WO_3 nanocrystalline powder

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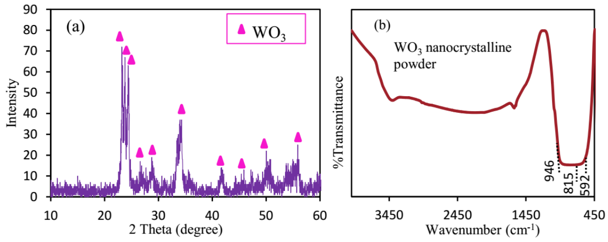

Fig. 1. (a) X-ray diffraction pattern and (b) FTIR spectrum of WO_3 nanocrystalline powder.

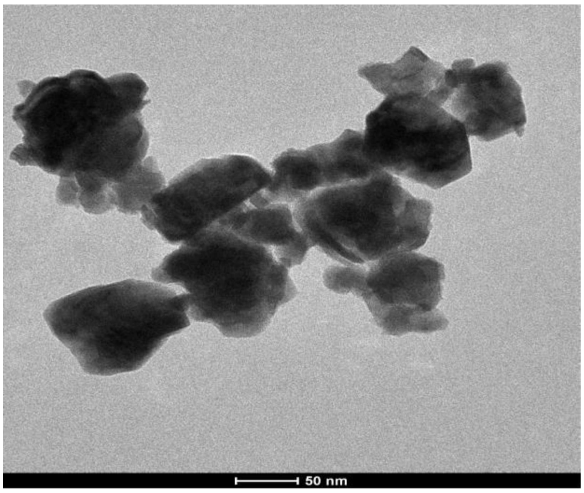

Fig. 2. TEM image of WO_3 nanocrystalline powder (scale bar: 50 nm).

of saturated toluene at room temperature is 93%. The gas response towards various concentrations of toluene in the range of 500–3000 ppm and the response is about 37, 49, and 63%, respectively (Fig. 3a). Figure 3b shows that the sample exhibited high sensitivity (93%) and good response stability of $0.93 + 5.5\%$ for toluene vapor. Also, WO_3 nanocrystalline powders have good reproducibility with high sensing performance after three on-off cycles which are repetitive exposure of the sample pellet to saturated toluene vapor and air (Fig. 3b).

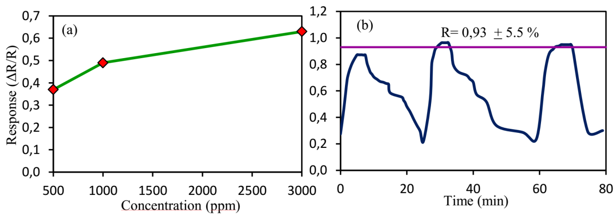

Fig. 3. Gas sensing properties of WO_3 nanocrystalline powder. (a) Response to toluene towards different gas concentration at 25 °C. (b) Response reproducibility on exposure to saturated toluene vapor at 25 °C.

4. Conclusion

We have successfully prepared WO_3 nanocrystalline powder by direct precipitation technique at low temperature without surfactant. TEM analysis of nanopowders showed that the average size of WO_3 nanocrystalline powder was approximately determined as 41.7 nm. As gas-sensing material, WO_3 nanocrystalline powder exhibits more excellent sensing performances towards toluene at room temperature.

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