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Abstract

Optical Interference Analysis of ZIF-8 Films for Chemical Vapor Detection †

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Abstract: Metal–organic frameworks (MOFs) are materials that feature a large surface area with permanent porosity, which is an attractive property for chemical and gas sensing, making them a good candidate for sensor fabrication. In this paper, we present a sensor that employs zeolitic-imidazolate-based MOFs (ZIF-8) to detect ethanol vapors via refractive index-dependent optical interference.

Keywords: nanomaterials; gas sensor; MOFs; ZIF-8; optical detection

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1. Introduction

Metal–organic frameworks (MOFs) are being regarded as an advanced material to improve some current technologies, among which are gas storage, molecule separation and chemical catalysis [1]. Their intrinsic porous characteristics have proven to be effective for chemical and gas sensing through the molecular absorption of gases into their porous structure, which translates into a change in the effective refractive index (RI) of the MOF film [2]. To this end, Zeolitic Imidazolate Framework-8 (ZIF-8) has been used to design thin films on substrates via layer-by-layer deposition to optically detect ethanol vapors at room temperature by monitoring the reflectivity spectra shift.

2. Materials and Methods

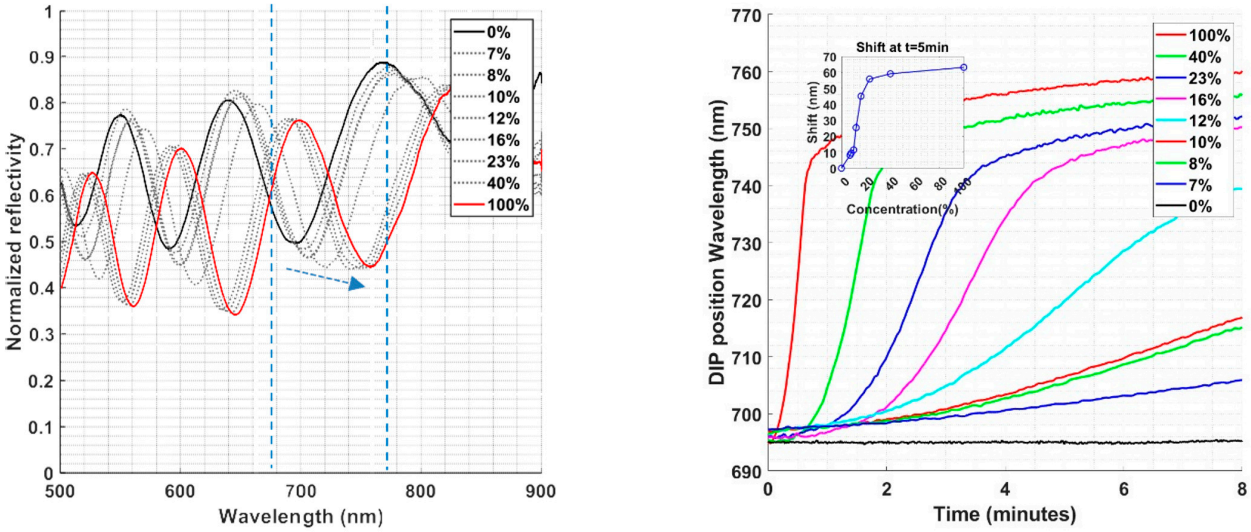
To synthesize ZIF-8, a 1:1 volume mixture solution of 2-methylimidazole (50 mM) and $Zn(NO_3)_2{\cdot}6H_2O$ (25 mM) was prepared. Each growth cycle was performed by immersing cleaned 1×1.5 cm^2 silicon substrates into the freshly made solution for 40 min at room temperature. The samples were then rinsed with pure methanol and dried with a nitrogen flow. The characterization of ZIF-8 film thickness was carried out with an optical Dektak XT profilometer by measuring a 2.5 mm long line at the substrate/ZIF8 border that was obtained by etching part of the layer in a dilute nitric acid solution (68% nitric acid: H_2O, 1:1000 (v/v)) for 5 s. The obtained results indicate that the layer's growth is in accordance with the reported deposition of 100 nm per 30 min cycle [1].

The reflectivity spectra were acquired with an Ocean Optics SD2000 spectrometer to characterize the interference fringes between ZIF-8 and the substrate, which, in the presence of ethanol vapor, experience a red shift due to an RI change. In this regard, vapor testing was performed by placing the samples inside a home-built aluminum chamber, in which 1 µL of different ethanol/water solutions were added without contacting the samples. White light was focused on a fiber optical cable and was then collimated and divided with a beam splitter. One of the beams was focused on the sample through a

quartz window using a 4× objective lens. The reflected spectra were always normalized to a reference spectrum in a ZIF-8-free area.

3. Discussion

Room-temperature gas sensing measurements were taken at different concentrations of ethanol/water solutions with samples containing various ZIF-8 film thicknesses. The results for a 10-cycle ZIF-8 layer (~1600 nm) are shown below. Figure 1a shows the interference fringes of the reflectivity spectra, in which the red shift caused by increasing the ethanol concentration can also be observed. The monitored dynamic shift of the spectrum's dip localized between blue lines under the first 8 min of ethanol exposure can be observed in Figure 1b, which shifts the fringes by up to 60 nm for concentrations over 40%, which is similar to the reported data [1] and larger than recorded other optical techniques [3]. Overall, ZIF-8 appears to be an advanced sensing material for room-temperature ethanol sensors. Our current focus is on studying the sensitivity of ZIF-8 to various volatile compounds and discussing its detection limits, considering its practical applications.



(**a**) (**b**)

Figure 1. (**a**) Interference fringes' red shift (arrow) for increasing ethanol/water concentrations (volume percentage in water). (**b**) Comparison of the monitored dynamic interference peak shift in an 8 min time frame. The figure inset shows the interference peak shift at t = 5 min for the different concentrations.

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