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Characteristic study on volatile organic compounds optical fiber sensor with zeolite thin film-coated spherical end

Binqing Wu, Chunliu Zhao, Juan Kang *, Dongning Wang

Institute of Optoeletronic Technology, China Jiliang University, Hangzhou, Zhejiang 310018, China

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ABSTRACT

In this paper, characteristic of volatile organic compounds (VOCs) optical fiber sensor with zeolite thin film-coated spherical end were investigated detailedly. The zeolite film and spherical end constituted an arc-shaped inline Fabry-Perot (F-P) cavity, and VOCs were measured by monitoring the wavelength shift of F-P interference which induced by the VOCs molecule adsorption of the zeolite film. The responses of the optical fiber sensor for monitoring isopropanol and formaldehyde were observed and especially observing the response of the optical fiber sensor in the mixed VOCs state. Experimental results show that the sensitivities of the optical fiber sensor for monitoring isopropanol and formaldehyde are 281.9 pm/ppm and 4.99 pm/ppm, respectively. The optical fiber sensor is more suitable for isopropanol measurement than formaldehyde. In the mixed VOCs state, the characteristic of the optical fiber sensor for isopropanol measurement is slightly changed when the air chamber is mixed with low concentration of formaldehyde, but the optical fiber sensor is still effective for isopropanol measurement.

1. Introduction

With the increasing request for better living environment, detection of toxic and flammable volatile organic compounds (VOCs) has received immense attention in recent years [1]. If people tough VOCs for a long time, the industrial emissions from wide-range chemical compounds can lead to causing some sick such as unconsciousness, dizziness, etc. even at low concentrations [2]. The most common conventional methods for VOCs measurement are sophisticated, high cost and can't be used in the extreme states. Hence, it is necessary to fabricate new sensing methods with a high sensitivity and efficiency for VOCs measurement. Because the optical fiber sensors generally offer many advantages such as high sensitivity, small volume and real-time monitoring, a lot of optical fiber chemical sensing devices have been reported and widely applied. Meanwhile, optical fiber sensor can be used in special industrial environment because of its characteristic. In the last few years, many optical fiber sensors for VOCs measurement were proposed. For example, L. Niu et al. put forward a sensor based on Rayleigh scattering effect in simplified hollow-core photonic crystal fiber to monitor VOCs [3] and photonic crystal fiber loop mirror-based chemical vapor sensor [4]. Besides, optical fibers over coated by thin film which can interact with the VOC molecules to achieve VOCs detection have been attracting increasing interests, such as polymer coated on fiber gratings [5–7], incorporating Nile red and polyvinylpyrrolidone (PVP) coated on single-mode fiber [8], surface plasmon resonance based on graphene [9] and so on [10–12].

Zeolite is crystalline aluminosilicate materials possessing a unique combination of chemical and optical properties that is ideal for developing various optical chemical sensors [13]. The zeolite can selectively adsorb molecules by means of size exclusion or shape selectivity which is depending on the crystal structure, framework Si/Al ratio, and type of extraframework cations [14]. Because of crystalline nature, zeolite also has excellent thermal, chemical, and mechanical stability to function in harsh states. Therefore, zeolite with multidimensional function as effective sample concentrator and optical probe has the potential for developing a variety of optical chemical sensors. So far, there are some reports about detecting VOCs with zeolite film coated on different structures, such as long period fiber [15], Fabry-Perot (F-P) interference [16] and spherical end fiber [17]. Among these structures, spherical end fiber sensor has higher sensitivity. Although zeolite film-coated spherical end fiber sensor has been proposed, the characteristic study of this sensor is not enough. In the existing reports, the zeolite film coated spherical end fiber sensor only for one kind VOC, and the optical fiber sensor response of different VOCs has not studied detailedly yet. Only understanding the influence of different VOCs for the optical fiber sensor and solving this problem, accurate measurements can be achieved in the real use.

* Corresponding author.
E-mail addresses: kangjuan@cjlu.edu.cn, 1099407934@qq.com (J. Kang).

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In this paper, characteristic of volatile organic compounds optical fiber sensor with zeolite thin film-coated spherical end were investigated detailedly. We observed the responses of the optical fiber sensor for monitoring isopropanol and formaldehyde as well as the optical fiber sensor in the mixed VOCs state. The sensitivities of the optical fiber sensor for monitoring isopropanol and formaldehyde are 281.9 pm/ppm and 4.99 pm/ppm, respectively. The OSA with a 0.02 nm measurement precision was used in our experiments, so the precisions for detecting isopropanol and formaldehyde could reach about 0.071 ppm in the concentration range from 0 to 70 ppm and 4.01 ppm in the concentration range from 0 to 1000 ppm, respectively. The sensitivity of the optical fiber sensor for isopropanol measurement is 56 times bigger than formaldehyde. The optical fiber sensor is more suitable for isopropanol measurement than formaldehyde. Furthermore, the characteristic of the optical fiber sensor for isopropanol measurement is slightly changed when the air chamber is mixed with low concentration of formaldehyde, but the optical fiber sensor is still effective for isopropanol measurement.

2. Experimental setup and sensing principle

2.1. Experimental setup

Fig. 1 shows the experimental setup for measuring the characteristic of the VOC optical fiber sensor with the zeolite thin film-coated spherical end, which includes a broadband light source (BBS) with 200 nm spectral bandwidth, an optical spectrum analyzer (OSA) with a wavelength resolution of 0.02 nm, a 3 dB optical fiber coupler and a VOC concentration controlling and detecting system. The sensing head as shown in the insertion part of Fig. 1, was placed into the air chamber, whose length, width and height is 70 cm,40 cm and 30 cm, respectively. The concentration of VOCs in air chamber was controlled by a pump and was detected and recorded by a gas detector (GasAlert-Micro5, BW Technologies).

The spherical end of the used VOC optical fiber sensor is fabricated by the electrical arc discharge on the single mode fiber (SMF) end. Then zeolite thin film is coated on the spherical end. The zeolite thin film preparation is based on the hydrothermal synthesis method. The structure directing agent of hydro-thermal synthesis is tetrapropylammonium ion (TPA+), and the synthesis solution is mixed by 60 ml deionized (DI) water, 11.3 ml TPAOH solution (tetrapropyl ammonium hydroxide), and 20.4 ml TEOS (tetraethyl orthosilicate). Firstly, the mixture was stirred at 50 °C for 3 h, then hydro-thermal synthesis was processed in the reactor

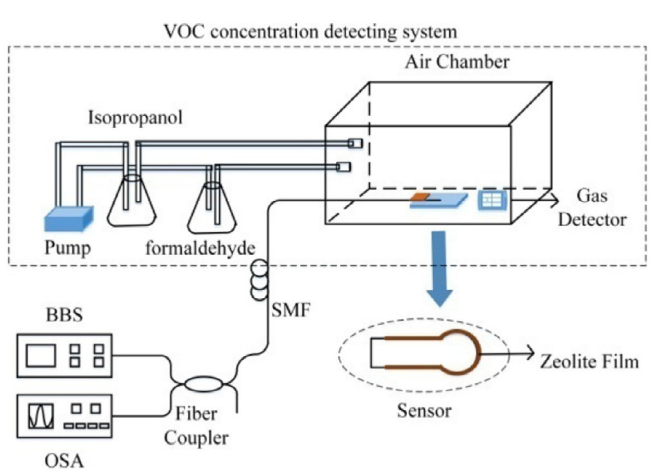

Fig. 1. Experimental setup for the VOC optical fiber sensor based on zeolite thin film-coated spherical end.

at 180 °C for 6 h. At last, the zeolite-coated fiber end was dried in air at 500 °C for 2 h to remove the TPA + SDA residues. The stirred time of mixture and the time of hydro-thermal synthesis in the reactor at 180 °C can influence the synthesis of zeolite film, which is related to the absorption of the zeolite film for VOCs. Fig. 2 shows the scanning electron microscope (SEM) images of the used VOC optical fiber sensor with zeolite thin film-coated spherical end. The zeolite film firmly covered on the spherical end. As shown in Fig. 2(a) and (b), the diameter of zeolite thin film-coated spherical end of the VOC fiber sensor is ~192 μm. The thickness of the zeolite film is ~25 μm as shown in Fig. 2(c) and (d).

2.2. Sensing principle

Fig. 3 shows the structure of sensing head. Input light is launched into a single mode fiber (SMF) and reached the sensing head by 3 dB coupler. The light is reflected partly at the spherical end, and part of light is reflected at the zeolite thin film, two reflected light is produced Fabry-Perot (F-P) interference. As the refractive index of the zeolite thin film is varied with the VOC concentration and the thickness of the zeolite film is constant when the zeolite film absorbing VOC molecules, the wavelength shift of F-P interference is caused by the refractive index changes. Therefore, the measurement of VOC concentration can be achieved by measuring the wavelength shift of F-P interference. The main principle is summarized as following. The reflected output intensity of light is I_1 is given as [18]

$$I_1 = \frac{2R_1(1-\cos\varphi)}{1+R_1R_2-2R_1\cos\varphi}I_0 \tag{1}$$

where $\varphi = 4\pi nL/\lambda$ is the phase difference of the F-P interference, L is the thickness of zeolite thin film, n is the refractive index of zeolite thin film, λ is the wavelength, I_0 is the intensity of input light, the reflectance of spherical end and zeolite thin film is R_1, R_2, respectively. The resonant wavelengths satisfies with $4\pi nL/\lambda = 2k\pi$, where k is any integer. Thus, the resonant wavelength can be described as

$$\lambda = 2nL/k \tag{2}$$

When the zeolite thin film absorbs the VOC molecules, the refractive index of zeolite thin film n changes while the thickness of the zeolite thin film L remains the same. So the relationship between the wavelength shift $\triangle\lambda$ and the refractive index of the zeolite thin film $\triangle n$ can be expressed as

$$\Delta\lambda = 2\Delta nL/k \tag{3}$$

According to the Eq. (4), the refractive index of the zeolite thin film changes with the VOC concentration increasing and the thickness of the zeolite film is constant, so the wavelength of F-P interference shifts when the refractive index changes. Thus, we can achieve the detection of VOC by monitoring the wavelength shift of F-P interference.

Fig. 4 shows the simulation results of the reflect output intensity of the sensor. When the zeolite thin film absorbs the VOC molecules, the refractive index of zeolite thin film $\triangle n$ changes, which leading to the wavelength shift of the F-P interference. The reflectance of R_1, R_2 are 0.006 and 0.005 in the simulation, respectively. The thickness of the zeolite thin film L is 25 μm. The initial interference spectrum is the black curve whose spectrum width is about 25 nm. When the refractive index of zeolite thin film $\triangle n$ only changes 0.002, the wavelength of the F-P interference shifts about 2.5 nm. And the wavelength shifts about 19 nm if the refractive index of zeolite thin film $\triangle n$ changes 0.016. Thus, we can achieve the detection of VOC by monitoring the wavelength shift of the F-P interference.