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Abstract. A novel approach using microstructured optical fiber and Raman spectroscopy for identifying acetone is reported. This technique combines the advantage of small sampling volume of microstructured optical fiber and the specificity of Raman spectroscopy.

1. Introduction

Acetone is toxic and explosive gas. Inhalation of acetone gas will do obviously harm to the mucous membrane and living in the circumstance with acetone toxic symptoms will appear [1]. Besides, Acetone is an exhaled volatile organic compound that has been shown to act as a biomarker for metabolic conditions in the bloodstream [2,3]. Accordingly, persistent efforts have been directed so far to develop the detection of acetone by many analytical methods, such as gas chromatography [4], electrochemistry detection [5], fluorescence quenching method [6,7], mass spectrometry [8], etc. However, disadvantages such as poor selectivity, high operating temperature, long response times [5, 9] and sample contamination [8] are associated with these methods.

A novel approach for identifying acetone is reported, using microstructured optical fiber (MOF) and Raman spectroscopy in this paper. MOF with small core dimensions result in a significant overlap of the guided light with any liquid or gas that is loaded into the holes in the fiber, which in turn creates a response signal along the entire length of the fiber that is subsequently waveguided by the core to a detector [10], what’s more enable the development of small sample volume, flexible and cost-effective sensing architectures [11]. Raman spectroscopy is a powerful analytical technique for detecting a wide range of chemicals in any state (solid, liquid, or gas phase) and is widely used in laboratories for chemical identification [12]. In this work, MOFs were used as an active dip sensor platform for Raman sensing of acetone in water solutions using the solvent’s Raman signature as an internal calibration standard.

2. Experimental section

The experimental setup used in these experiments is shown in Fig.1 which is the same as Ref. [13].
Continuous wave light from a 488nm Ar+ laser was reflected off a long-pass Raman filter and coupled into the silica MOF (made by Institute for Photonics and Advanced Sensing, The University of Adelaide, shown in the insert of figure 1) using a 40x microscope objective. The length of MOF is 16 cm resulting in a total sampling volume of 60nL (the diameter of the air void is about 6.3 μm). The other end of the fiber was dipped into the sample, capillary forces drew the acetone solution into the air voids along the length of the fiber. Backscattered Raman signal from the fiber was collected through the long pass filter and recorded by a fiber-coupled cooled-CCD spectrometer.

Acetone solutions were prepared by mixing pure acetone with milliQ water. Figure 2 is the Raman spectrum of MOF filled with 50% (volume ratio) acetone in water. The resulting spectrum for acetone and water are easily distinguishable. The strong Raman peak at 2856cm⁻¹ is the C-H stretching peak of acetone which can be used to determine the concentration of acetone by its relative intensity. The broad Raman peak centered at 3371cm⁻¹ is H-O stretching peak of water which can be used as an internal calibration signal.

Raman spectroscopy of MOF with different filling length (50% acetone) can be studied, as shown in figure 3(a). Both signatures increase in intensity as the fiber fills up with acetone solution by capillary force action. As an indication of the total intensity of the Raman scattering for each molecule, the area under the spectral curve is integrated and the time evolution of Raman intensity of acetone and water is
shown in figure 3 (b). The integrated intensities of the acetone and water Raman peaks increase in parallel as the fiber fills up.

Figure 3. (a) Raman spectroscopy of MOF with different filling length (50%acetone) (b) Curves of Raman peaks of acetone and water with filling time

The ratio of the Raman peak intensities of acetone and water is calculated throughout the measurement and the value is plotted in figure 4 against the known sample concentration. The observed linear relationship between the Raman intensities ratio and the acetone concentration allows the use of this internal calibration technique to measure the concentration of acetone in water that does not depend on input laser power fluctuations, coupling instabilities and other changes in the fiber environment [13]. When the concentration of acetone is 1%, the Raman peak of acetone at 2856cm⁻¹ is nearly invisible. However, the detection limit can be lower down by Surface Enhanced Raman Spectrum(SERS) which is a useful technique resulting in strongly increased Raman signals from molecules which have been attached to nanometer sized metallic structures [14].

Figure 4. The relationship between acetone concentration and Raman ratio
3. Conclusions

In this paper acetone was detected by using MOF and Raman spectroscopy. This is based on an unmodified MOF as a Raman sensing platform, making use of the relatively large power fractions of excitation light available in this geometry to interact with analyte molecules along long lengths of the fiber. By using the Raman signature of water as an internal calibration standard, quantification of the acetone concentration is possible.

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