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Heterodyne Interferometry for Detection of Toluene using PDMS as a Sensing Film Sulaiman Khan¹, Stéphane Le Calvé^{2,3}, David Newport^{*1}

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KEY WORDS

VOCs, BTEX, Mach-Zehnder Interferometer, Optical sensor, film-mediated optical sensor.

ABSTRACT

Monitoring of air quality in indoor spaces is important for healthy living. On average a person spends about 90% time in indoor environments where exposure to various indoor air pollutants is inevitable [1]. Indoor air can contain volatile organic compounds (VOCs) among others pollutants like air-borne particles, microorganisms, household odors and gases. Some of VOCs i.e. benzene, toluene, ethylbenzene and xylene (BTEX) are harmful and long-term exposure may pose a serious threat to human health. Among BTEX, benzene is identified as carcinogenic [2,3]. There are stringent regulations about the exposure of BTEX. The European Union regulated the maximum exposure limit of benzene down to $5\mu g/m^3$ (1.6 ppb) for indoor spaces [4,5]. The monitoring of BTEX at sub-ppb level requires a ultra-sensitive technique. Interferometry base sensing can be a viable option for detecting BTEX.

Interferometry based sensing techniques are ultra-sensitive and have been employed for pressure, temperature and concentration measurements in microchannels [6]. Its application has been extended to gas detection by employing a BTEX sensitive film. In previous work, the VOCs detection using interferometry limited to ppm level. Martinex *et al.* used a Pohl interferometry setup for VOCs detection with a sensitivity of 1500 ppm [7]. Xiangping *et al.* applied interferometric configurations i.e. Faby-Perot (FP) and Sagnac interferometer (SI) [8]. A sensitivity of 9.02 x 10^{-4} nm/ppm and 1.17 x 10^{-3} nm/ppm was achieved using SI and FP interferometer respectively. Karthik *et al.* used FP interferometer integrated with μ GC and detected toluene down to 25 ppb [9]. Kacik *et al.* developed optical fiber sensor by using a micro cavity in PDMS and detected toluene with a sensitivity of 0.15 nm/g-m⁻³ [10].

In this study we investigated Mach-Zehnder interferometer configured with heterodyning for detection of air-borne toluene. It is sensitive technique capable to retrieve nanometric level variations (40nm) [11] and has the potential to measure air-borne BTEX at sub-ppb level.

The optical setup is designed based on Mach-Zehnder interferometer with heterodyning. A light from a coherent He-Ne laser ($\lambda = 633$ nm, 35mW, JDS Uniphase) source is guided by a single mode (SM) optical fiber. The light beam is split into two beams by 2x2 fiber coupler (BS1). One beam acts as a test beam while the other acts as a reference beam. An inverted optical microscope (Olympus





IX41) is configured with the test beam through a fiber from the BS1 fiber coupler. The condenser lens is replaced by a collimating lens positioned with the help of in-house machined optical cage system. The collimating lens allows a planar light wavefront to transverse the sample. The phase is same all across the plane and a proportional variation occurs while traversing the sample. Microscope objective collects the wavefront passing the sample which then interferes with the reference beam at the cubical beam splitter (BS). The setup is shown in the Fig.1 (A). The reference arm acts as a wavefront shaping optical arrangement which can be used to control the number and shape of fringes on the camera. The magnification of the setup is a function of focal length of objective and lens L_3 . By changing lens L_3 with a different focal length lens, the system can be optimized and the system magnification can be adjusted to suit the detector pixel size. The interferograms are captured by high speed CMOS camera. LabVIEW program is developed to capture and analyze the interferograms.



PC maintains the polarization of light beam

Figure 1 (A) Heterodyne interferometer configuration. Light beam is split into test and sensing beam by beam splitter and recombine to get the fringe patterns. AOMs are connected to a radio frequency driver and introduce frequency shift. (B) Micro gas chamber contain PDMS film. The chamber is 25 mm x 25mm x 25mm. Top and bottom are covered with glass. The gas flow is allowed from the side walls.

A polydimethylsiloxane (PDMS) is used as a toluene sensing film, which changes its volume and refractive index when expose to toluene [12]. PDMS are silicone-based elastomer and are widely used in microfluidics applications due to its features like optical transparency, chemical inertness, biocompatibility, low cost, flexibility, reversible deformation in mechanical stress and tunable permeability [13,14]. Although the swelling of PDMS is not desired for many application however it can be exploited for the actuation mechanism in sensing devices [15]. PDMS is shaped into a rectangular slab with thickness of 100 μ m and 500 μ m and enclosed in a small chamber. The relative height change in PDMS is used for estimation of phase change so the surface roughness is not a critical parameter. The gas chamber has size 25mm x 25mm x 25mm. Top and bottom are covered with glass to allow the light beam to pass through the PDMS film. The gas is flowing through the sides and the light beam is passing through PDMS from top to bottom as shown in Fig.1 (B). Highly precise mass flow controller (accuracy < 0.5%) are used to generate the desired dosage of toluene. The concentration of toluene down to ppb can be generated with precision of 10 %.

Heterodyning is realized with a pair of fiber coupled down-shifted Acoustic-Optic Modulator (AOM) in each arm of the interferometer as shown in Fig.1(A). AOMs employ sound waves to diffract the light beam and shift its frequency. The AOMs introduce frequency shift and provide a linear phase shift for temporal phase extraction. It is a phase extraction process in which a high frequency signal (carrier signal) is mixed with another constant high frequency signal. The purpose is to extract the low frequency message embedded in the carrier signal [16]. When the mixing is performed at frequencies in the visible range then it is called heterodyne interferometry. The time varying intensity signal generated from the appropriately sampled by the digital camera is mathematically given by

$$I_{x,y} = a_{x,y} + b_{x,y} \cos(2 \Phi' f_c t + \varphi_{x,y})$$
(1)





$$a_{x,y} = I_R(x,y) + I_S(x,y),$$

$$b_{x,y} = 2\sqrt{(I_R(x,y)I_S(x,y))}$$

$$\Phi' = 2\pi f_c t$$

 $I_R(x,y)$ and $I_s(x,y)$ are light frequency in reference and sample arms respectively. $\Phi^{\}$ is the known phase shift. f_c is the temporal carrier signal. $\varphi_{(x,y)}$ is the unknown phase. AOMs are operated in MHz range i.e. (70-100 MHz). The known phase shift, $\Phi^{\}$ is in the range of 50Hz-150Hz. Five cycle phase extraction method is employed by using 21 phase-shifted Interferogram image frames to obtain the phase-image. The algorithm is improved by increasing the number of signals and Hanning window function. The wrapped phase is unwrapped by a using phase-unwrapping algorithm [17]. The molecules absorbed can be estimated from the volume change. The variation in height, Δh is calculated by using the phase information as given by

$$\Delta h = \frac{\Delta OPL - \Delta n h}{(n-1)}$$

$$\Delta OPL = \lambda(\varphi_f - \varphi_o)$$
(2)

 Δh is the change in PDMS height due to VOCs absorption, ΔOPL and Δn are the change in the optical path length and refractive index respectively. The initial height and refractive index before the VOCs absorption are given by h and n respectively. The phase change before and after gas exposure are given by φ_o and φ_f respectively.

A heterodyne interferometer using a Mach-Zehnder configuration is designed for detection of airborne toluene. The design is based on film-mediated sensing technique in which the response of intermediate sensing film is monitored using interferometry. PDMS is used as a sensing film, which changes its volume and refractive index when absorbed BTEX molecules. PDMS acts as a phase object and any variation in its state causes proportional phase shifts in the transmitted light beam. The Mach-Zehnder interferometer is integrated with a microscope and acoustic-optic modulators. The information of the absorbed gas concentration into the film are encoded in the fringe patterns and are extracted by a temporal phase extraction heterodyne method. The concentration of absorbed gas into PDMS are estimated from height change by calibration.

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