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General

Carbon dioxide luminescent sensor based on a CMOS image array

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ABSTRACT

In this work, we describe an optical carbon dioxide (CO_2) sensor based on luminescent quenching of hydroxy-pyrenetrisulfonic acid (HPTS). A low-cost CMOS image array was used as detector, while cross-polarization was used to filter the excitation light and reduce background interference. The sensor exhibited excellent performance, with high sensitivity over a wide dynamic range of 0–100% CO_2. As proof-of-concept demonstration, the sensor was field-tested with automobile exhaust and benchmarked with a commercial sensor as a reference. By using an image sensor typically found in consumer products, a compact, low-cost luminescence-based CO_2 sensor was successfully demonstrated with performance comparable to that of the commercial sensors.

1. Introduction

Abnormal levels of carbon dioxide (CO_2) can have serious health effects [1–3], while prolonged exposure to high levels of CO_2 (>2%) have been reported in various occupational settings such as firefighting and mining [4]. These levels can result in adverse respiratory effects as well as inhibition of cell functions even in healthy adults [5–8]. Carbon dioxide detectors using the Non-Dispersive Infrared (NDIR) principle have been demonstrated showing increased sensitivity; however it can require expensive, bulky equipment which deters its implementation in occupational settings [9]. Smaller, portable versions of these sensors have been developed, but they display increased response as high as 30 s per measurement and have a 5% CO_2 concentration detection limit. Electrochemical methods have been demonstrated as a low-cost alternative, but the electrodes degrade over time [10]. Detection methods need to be realized for commercial environments and field use which are stable over time, have increased detection limits and lower response times.

Optical detection methods are popular due to reduced response time and non-consumption of analyte; however, they often require expensive instrumentation such as spectrophotometers and trained personnel. In this work we used a CMOS image array to miniaturize the sensor and eliminate expensive components. Both we [11] and others [17] used this approach to miniaturize optical sensors. In our case, we have demonstrated a portable optical oxygen sensor using a low-cost off-the-shelf CMOS image sensor as a detector and a cross-polarization scheme for signal isolation. Our oxygen sensor was based on the oxygen sensitive metalloporphyrin luminophore platinum octaethylporphine (PtOEP) and oxygen insensitive Rhodamine B (RhB). The CMOS image sensor permitted selective detection of the red emission from PtOEP and RhB as well as simultaneous monitoring of their emissions. The oxygen sensor exhibited high sensitivity and good stability, comparable with commercial oxygen gas sensors [11].

In this work, we used the same general approach to develop a portable, optical CO_2 sensor. The sensor again was based on a low-cost commercially-available CMOS image array and the fluorescent quenching of a hydroxy-pyrenetrisulfonic acid (HPTS) (Fig. 1a). HPTS has been used in sensor applications for the detection of gaseous and dissolved CO_2 [12,13], and displays excitation and emission peaks in the visible range (λ_{ex} = 460 nm, λ_{em} = 520 nm). This makes HTPS especially suitable to our optical detection approach. We used a stacked arrangement with cross-polarization to filter out excitation light and increase sensitivity of the CMOS array (Fig. 1b) [14]. Raw data were taken from the image sensor and analyzed in the green channel to measure HPTS emission intensity vs. CO_2 concentration. For further evaluation of the utility in real-world situations, we tested our sensor with automobile exhaust and compared performance with a commercial sensor. This is the first demonstration of a HPTS-based CO_2 sensor in the field.

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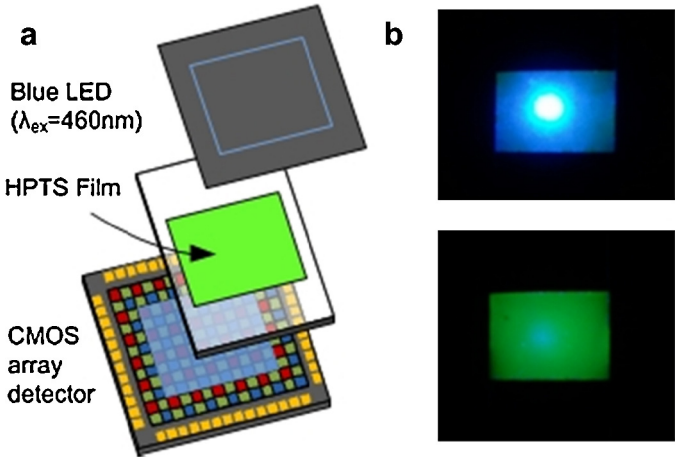

Fig. 1. (a) Arrangement of the light source (LED), detector (CMOS), polarizers, CO_2 sensitive HPTS film in the portable CO_2 sensor. (b) Illustration of polarization filtering which permits isolation of optical signal (fluorescein emission centered at 520 nm).

2. Experimental methods

2.1. Setup

This sensor is based on the CO_2 sensitive HPTS encapsulated in gas-permeable ethyl cellulose (EC) matrix. The light source used for excitation was a blue LED (R20BLU-F-0160) with a peak emission wavelength of λ_{ex} = 460 nm. Blue light is passed through polarizer 1 (NT45667, Edmund Optics), becoming linearly polarized and exciting the HPTS film. The green emission from HPTS has a central peak of λ_{ex} = 520 nm and also becomes linearly polarized as it passes through polarizer 2 on its way to the sensor array. Polarizer 2 is placed orthogonal to polarizer 1 therefore filtering out blue excitation light and increasing sensitivity of the sensor.

We constructed a gas detection setup consisting of a chamber able to introduce CO_2 and N_2 gases to our sensor. Two flow meters (6A01, Omega) were used to control flow rate through the system and into the main detection chamber. Previous findings showed HPTS film is sensitivity to humidity, ultimately causing a change in emission intensity. To maintain constant humidity, a humidity sensor (Sensiron SHT11) was placed inside the measurement chamber and monitored during experiments. An oxygen humidifier commonly used in hospitals was filled with distilled water and placed in-line with the nitrogen gas allowing us to accurately control humidity concentration inside the chamber by varying flow rate of nitrogen directly from the tank and the nitrogen that we passed through the humidifier.

To reduce measurement errors and chamber fill time, we constructed a pre-mixing gas chamber from PVC and a small heat-sink fan also shown in Fig. 2. Nitrogen, humidity controlled nitrogen, and CO_2 were connected via separate inputs into the mixing chamber. Using flow rate valves, the inlets were independently controlled to allow mixing before exiting the pre-mix chamber to the main measurement chamber. The chamber was constructed to be just large enough to contain all of our measuring devices including the commercial CO_2 sensor (K33-BLG), O_2 sensor (Nuvair, Pro O_2), and humidity sensor (Sensiron SHT11), as well as our CMOS camera detection setup. We also included an outlet proportional to the inlet so as to not pressurize the chamber and provide an even flow of test gases over the detection film.

2.2. Gas sensitive film

Gas sensitive films were fabricated using the ion-pairing method [15], by combining HPTS with 1 mL tetraoctylammonium hydroxide and 2.5 mL of methanol. A mass of 5 g of EC was combined with 10 mL of ethanol and 40 mL of toluene to form the encapsulation matrix [16,17]. Films were fabricated with varying thicknesses and HPTS concentration to optimize fluorescence for our setup. A spectrophotometer was used to measure peak emission intensity and optimize film thickness to reduce sensor response time. Commercial CO_2 sensors suffer from increased response time (>30 s), which led to the choice of a thinner film to increase diffusion of the gas into the sensor. The films were spun coat at 1000 rpm for 30 s on 2 × 3 in. glass slides resulting in an even distribution of detection film.

2.3. Characterization

Without further filtering, the CMOS detector's color discriminating capability is not sufficient by itself to adequately detect HPTS emission. Since we used a stacked arrangement for our sensor setup, the blue LED with a central emission wavelength of 460 nm will saturate the CMOS array resulting in high noise, thereby reducing sensitivity. Optical signal isolation based on cross polarization has proven to be a simple approach to filter out excitation light regardless of wavelength.

The sensor was placed in this sealed chamber along with a commercial CO_2 detector (K-33 BLG) for calibration. For these experiments, nitrogen and CO_2 were premixed before entering the main sensor chamber. Input flow rate was varied to adjust concentration. In order to verify sensor specificity, oxygen concentration was varied from 0% to 5% by adjusting flowrate into the main chamber. The oxygen concentration was calibrated with a commercial oxygen sensor (Nuvair, Pro O_2). Humidity was held constant while changing oxygen concentration in the chamber, taking images at each concentration point for subsequent green channel intensity measurement.

The CMOS image array (OmniVision, OV9810) was used as the detector for this work. The sensor array size was 3488 × 2616 pixels or 9 megapixels, each pixel having an active area of 1.75 μm × 1.75 μm. At each pre-determined concentration, raw images were taken using the supplied development software. All automatic CMOS functions such as gamma correction, white balance, and exposure time were manually set. Images were then analyzed using ImageJ (ver. 1.43u), by obtaining varying intensity in the green emission channel. The background signal was obtained by taking images of a bare glass wafer, then subtracting this value from the average intensity to give us only the HPTS emission intensity value.

3. Results and discussion

We first explored suitability of HPTS for luminescent CO_2 sensing. The thin film with HPTS exhibits a central absorption wavelength at 462 nm and a central emission wavelength at 525 nm when encapsulated in ethyl cellulose [18]. From previous work, HPTS fluorescence intensity is known to be sensitive to CO_2 concentration. Fig. 3 shows that there is a large overlap between the blue emission from the LED (460 nm) and the absorption wavelength from HPTS.

CMOS detectors typically have integrated filters where certain pixels respond only to certain colors. Our CMOS detector (Omnivision OV810) blue pixels display a peak response at 460 ± 30 nm, while the green and red pixels peak at 540 ± 40 and 600 ± 50 nm, respectively. Since the HPTS film exhibits visible green emission, our CMOS detector's color discriminating capability, coupled with the cross-polarized filters to reduce the excitation light, will be sufficient in quantifying the film response. The green channel response is much reduced at approximately 600 nm, which filters much

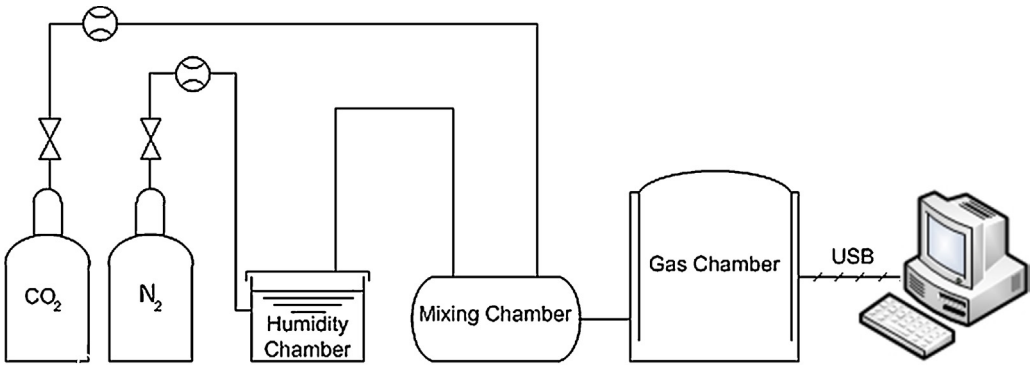

Fig. 2. Nitrogen and CO_2 flow were varied using control valves and meters to vary CO_2 concentration. Nitrogen was flowed through distilled water to maintain humidity in the main chamber for sensor measurements.

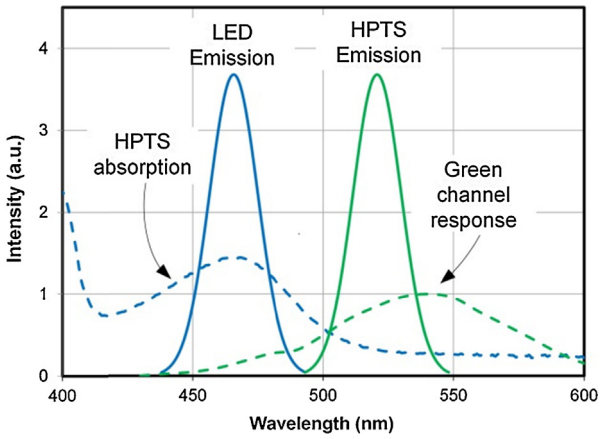

Fig. 3. The HPTS absorption and emission spectra overlaid with the blue LED emission and the green channel response of the CMOS detector.

of the background light interference. This capability allows us to also monitor the excitation light in the blue channel without any interference from the green emission. Moreover, since HPTS emission does not overlap significantly with the blue LED excitation wavelength, we can monitor the HPTS emission with minimal interference from the excitation source.

Emission intensities of HPTS for all three color channels of the CMOS array under constant humidity of 70% and varying concentrations of CO_2 and N_2 are shown in Fig. 4a. The green channel displays a significant decrease in emission intensity, indicating the quenching effect by CO_2 while the red and blue channels remain relatively stable. To compensate all three channel emission intensities, background intensity was subtracted from each channel individually. To obtain background intensity, the HPTS film was replaced with a clean glass slide and images were taken at 0% and 5% CO_2. Intensity values obtained from the images were then averaged together and subtracted from each color channel intensity. The glass slide used for this measurement was the same used to spin coat the HPTS film.

The HPTS films displayed high sensitivity and good linearity. To characterize the film, we used the Stern–Volmer analysis, which is the standard approach to characterize luminescence-based quenching. The Stern–Volmer equation is given as

$$\frac{I_0}{I} = 1 + K_{SV}[CO_2] \quad (1)$$

where I_0 is the emission intensity with no CO_2 present, I is the emission intensity with CO_2, and K_{SV} is the Stern–Volmer constant. By plotting this relationship, the x term gives us the sensor sensitivity. From our data, our sensor exhibits sensitivity of 0.089 [1/%], which is comparable to the previously-reported commercial NDIR sensors that are widely accepted for field use. Fig. 4b illustrates a representative Stern–Volmer plot of our sensor from 0% to 5% CO_2. The correlation coefficient of 0.94 indicates a highly linear relationship between the emission intensities from 0% to 5% CO_2, and shows comparable sensitivity to commercial NDIR sensors which displayed 20 ppm CO_2 sensitivity [9,13].

Response time and repeatability are important characteristics when referencing sensor performance. Emission intensity was recorded while CO_2 concentration was cycled from 0% to 100% by flooding the chamber with nitrogen. The sensor shows good repeatability (Fig. 5) and stability over multiple detection cycles which is essential for eventual commercial use. Response time

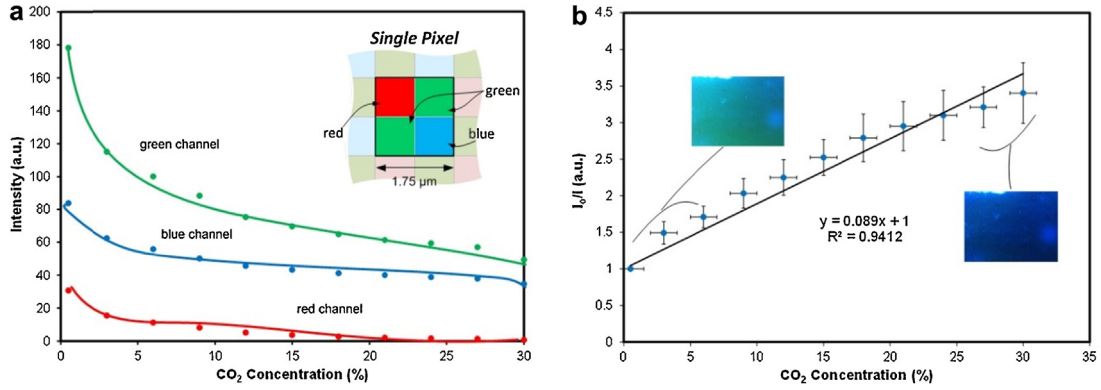

Fig. 4. (a) Emission intensity of HPTS as a function of CO_2 concentration. (b) Stern–Volmer plot of the CO_2 sensor. Horizontal error bars represent 3% detection limit of the commercial NDIR sensor, while the vertical bars represent the error from our developed sensor for multiple experimental trials. The inset images illustrate emission of HPTS at low and high CO_2 concentrations.

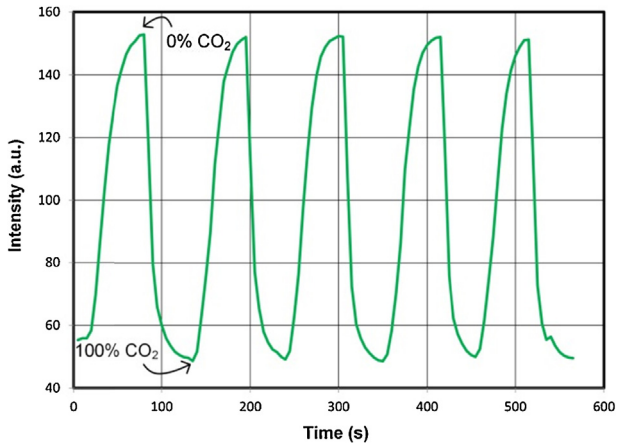

Fig. 5. Time response of the CO_2 sensor exposed to alternating streams of CO_2 and N_2 gases. 100% CO_2 concentration shows a fully quenched emission vs. 0% CO_2 which allows full emission of the film. The response time T is 21.62 s.

(time to increase intensity from 10% to 90%) was recorded at 21 s which is better than that of commercial NDIR sensors that are 30 s for a 0–2000 ppm detection range [19].

HPTS film intensity was found to decrease over time depending on the type of encapsulation matrix chosen. Sol–gel films doped with HPTS showed a 15% decrease in film emission over a 6 week period under normal conditions [16]. The films used for these experiments, encapsulated in ethyl cellulose (EC) exhibited similar behavior. Fig. 6a shows film emission from an excitation source of 380 nm taken over an 11 week period. The resulting emission shows a 10× increase in the 430 nm range, while simultaneously showing an 87% reduction in 520 nm emission. Measurements were also taken using a 460 nm excitation source. Fig. 6b shows a similar reduction in sensitivity over an 11 week period, rendering the film unusable. A more detailed view of the intensity deterioration is shown in Fig. 6c, where emission intensity of 460 nm (blue) is increased over time, while emission intensity of 520 nm (green) is reduced to near zero over a period of 80 days. Though EC and sol–gels have been readily established as good matrices for chemical sensing [20], HPTS has been shown to leak through the polymeric layer, resulting in a reduction of emission intensity. To better improve the sensitivity of the detector, the encapsulation matrix must be carefully chosen to prevent this phenomenon. Fluorescence studies suggest that matrices consisting primarily of polyurethane or polystyrene appear to limit leakage between HPTS and the polymeric layer [21].

Gas sensors used in environmental monitoring encounter harsh conditions such as temperature, humidity, pressure, and multiple gas exposure which can have adverse effects on sensor performance. Commercial NDIR sensors typically used for CO_2 detection have been reported to give inconsistent readings under varying conditions of humidity and oxygen [19,22]. For example, NDIR sensors for HVAC applications have a ±3 ppm per %RH sensitivity. In order to test the CO_2 CMOS sensor against these conditions, humidity and oxygen concentration were varied inside the measurement chamber. Humidity was varied in the 1–70% RH range, while keeping CO_2 at atmospheric conditions (Fig. 7a). Film emission intensity was monitored closely during this experiment showing an emission intensity variation with standard deviation $\sigma = 1.8$, which constitutes a minimal change in the presence of varying humidity. This

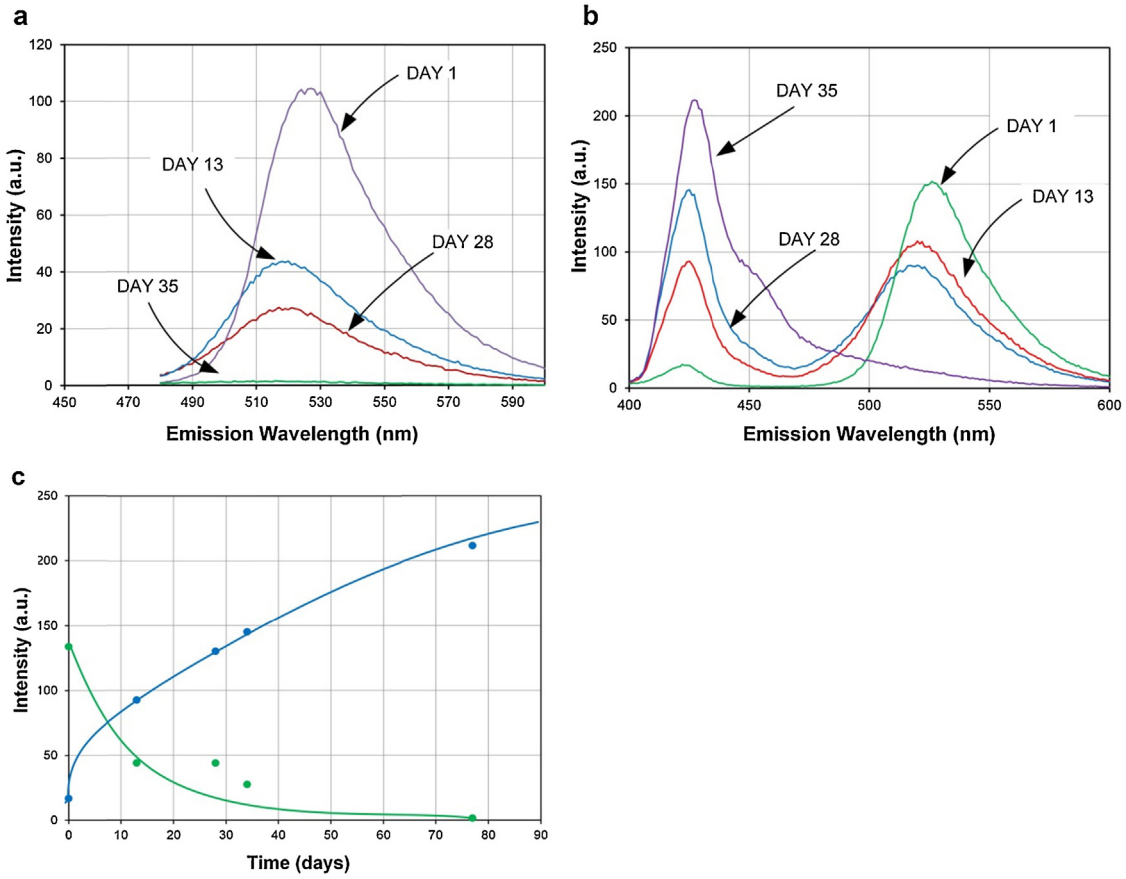

Fig. 6. (a) 520 nm emission intensity caused by 460 nm excitation over a 4 week period. (b) 520 and 465 nm emission intensity caused by 380 nm excitation over an 11 week period with the arrows showing trend. (c) 520 nm emission intensity (green) and 460 nm emission intensity (blue) shown over an 11 week period with 380 nm excitation.

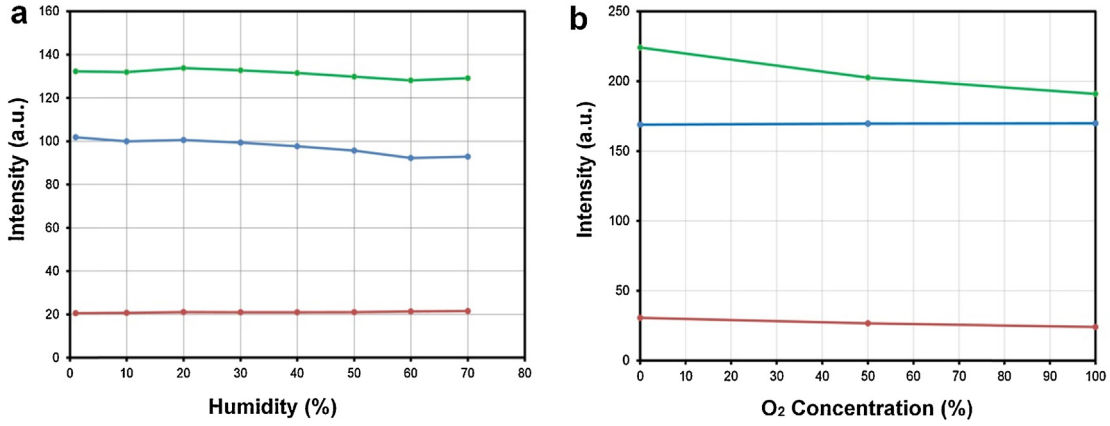

Fig. 7. (a) Emission intensity of each RGB channel under varying humidity concentration at 21% O_2. (b) Sensor response of each RGB channel during varying O_2.

result suggests that the sensor does not exhibit significant sensitivity to variations in humidity. This implies that the sensors could be used in a wide range of environmental climates, from firefighters in the mountains of Colorado to occupational workers at sea level, which is an important characteristic for a point-of-use sensor. Temperature changes greater than 20 °C have shown an effect on the setup; however specific calibration was not performed. Temperature was held constant during all background and experimental measurements. While keeping humidity constant, oxygen concentration was varied from 0% to 100% (Fig. 7b). The sensor showed good stability and low deviation in the changing O_2 environment, which will allow us to use this method for multi gas detection. HPTS exhibits small sensitivity to O_2 which has not been reported in previous work. Further investigation should be done when commercializing a sensor of this type.

The experiments reported thus far were performed in a controlled laboratory environment. Next, as proof-of-concept, we took the sensor to the field and demonstrated measurements in automobile exhaust. A commercial NDIR sensor (K33-BLG, CO_2 Meter.com), was used for comparison. A vacuum pump was connected to the exhaust of an automobile running in idle filled with 89 octane fuel in order to pull the sampling gas past the sensors. A particle filter with a 0.45 μm porosity was placed in line before the measurement chamber to filter the exhaust and prevent fouling of the commercial and experimental sensors. Typical automobile exhaust consists of up to 15% CO_2, which is a sufficient concentration for detection with our sensor [3,23,24]. Fig. 8 illustrates the standard curve that compares performance of our optical sensor with the commercial NDIR sensor. Our sensor performed well over the entire range, exhibiting linear although bimodal curve. In the lower range, 0–2% CO_2, the slope of the standard curve was ~1.1, indicating performance comparable to that of the commercial sensor. This range is typical for most detectors currently on the market, since indoor air quality is the most common application and concentrations over 2000 ppm can have detrimental health effects. In the higher range of CO_2 concentrations, >2%, the optical sensor deviated from the commercial detector, exhibiting a decreased slope of the standard curve of ~0.3. The response remained consistently linear, however, with a very high coefficient of determination $R^2 = 0.99$. One of the reasons of this apparent decreased response of our sensor is that after the post-field-test examination of the setup revealed combustion particulates [3,23,24] inside the sampling tube and the testing chamber. These particulates will affect the optical detection setup by blocking signal and causing lower intensity readout. Another reason is that also post-test condensation was discovered on the input of the sampling filter and the sampling line, most likely due to the combustion process. This condensation eventually degraded filter performance and the overall gas flow through the field test chamber, affecting CO_2 analysis. In the future, better filtering of input air will be necessary to compensate for these particulates and condensation in the sampling lines. Nevertheless, our field test results are encouraging and suggest that further development of this method into a viable field sensor maybe possible.

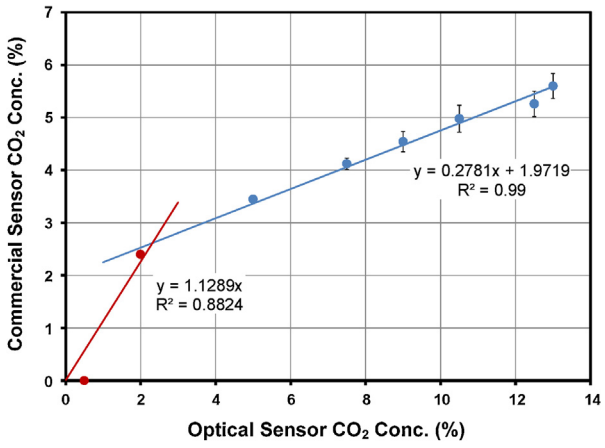

Fig. 8. Result of tailpipe test comparing optical sensor vs. commercial sensor.

4. Conclusions

A portable optical CO_2 sensor was demonstrated using a cross polarization scheme to isolate the emission signal and a low cost CMOS image array for signal detection. The sensor was characterized using a commercial NDIR sensor and displayed sensitivity and linearity comparable to small commercial sensors as well as larger benchtop systems. The sensor exhibited a significantly faster response time than commercial NDIR sensors, while achieving similar sensitivity and detection limits. The sensor also showed good stability in the presence of varying O_2 concentrations as well as changes in humidity, demonstrating the viability for outdoor use in the field. Overall, the approach demonstrates a low-cost portable optical CO_2 sensor with increased time response which can be integrated with other gas detection films and be used for multiple gas detection. The components used can be miniaturized to provide increased streamline integration and portability for field use. In the future, an optical reference such as fluorescein ($\lambda = 520$) could be added to ensure accurate measurements and provide background compensation. By taking advantage of a reference with the same emission wavelength as fluorescein, we can compensate

from exterior illumination sources causing emission light not from fluorescence quenching.

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