Gaseous contaminant detector in compressed air systems using photoacoustic spectroscopy

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Abstract. A photoacoustic (PA) setup is presented, based on diode laser excitation in the infrared (5 mW, 1728 nm). The sensor being developed is designed to detect spurious amounts of gaseous contaminants like oil vapour in a highly perturbing environment (high flow, high pressure) via excitation of molecular vibrations of C-H bonds. An acoustic oscillator is driven at resonance to amplify the photoacoustic signal. The measurement cell is designed for optimum quality factor (Q) and setup constant $\left[C_n(\omega)\right]$.

1. Introduction
Laser photoacoustic (PA) spectroscopy is a technique for detection of gaseous contaminants, having attractive features like small cell size, stability, ease of optical alignment, and inexpensive microphones. High sensitivities in gas detection have been reported using infrared lasers such as CO and CO\textsubscript{2} lasers, to detect contaminants in the ppb (parts per billion) range, due to their high output power and tunability to strong fundamental vibration transitions [1].

There exist two types of photoacoustic cells, resonant and non resonant. Though non resonant cells are smaller than resonant cells, a resonant cell is used in the setup due to the following advantages. The photoacoustic signal can be elevated with the resonance of the PA cell, and has a higher signal to noise ratio (SNR) compared to the non resonant cell. Sample gas can be flowed continuously through the cell as long as the inlet and outlet of the gas is at a place where the standing acoustic wave has a node [2].

An important requirement in many industries is clean compressed air which satisfies ISO 8573 with specifications of class 1 demanding an equivalent of a maximum residual hydro-carbon contamination of less than 0.005 mg/m\textsuperscript{3}[3]. Today air quality is ensured by scheduled maintenance of filter units and laboratory analysis. This project aims at developing a platform for detection of oil contaminants in compressed air, the main requirements of such a sensor being sensitivity, applicability to a strongly perturbed system, reliability, and robustness. The availability of diode lasers has sped up the development of compact gas detection systems. This work presents the implementation of a diode infrared laser in a compact setup for the detection of gaseous hydrocarbons.
2. Photoacoustic spectroscopy

In this technique, a modulated light source preferably a laser passes through a cell containing a sample gas. The absorbed energy of the laser heats the gas resulting in an expansion. This in turn produces an acoustic and thermal wave which is detected with sensitive microphones. The amplitude of the photoacoustic signal (S) for a cell with high Q is given by

\[
S = C_n(\omega_n)\alpha W_L
\]

Where \( C_n(\omega_n) = (\sigma - 1)LF_nQ_n/V_{cell}\omega_n \)

Where \( C_n(\omega_n) \) is called the setup constant, \( \sigma \) is the adiabatic index of the gas, \( L \) is the length of cell, \( F_n \) is the normalized overlap integral of the resonant eigenmode with the laser field, \( Q_n \) is the acoustic quality factor of the cell, \( \alpha \) is the absorption coefficient of the gas, \( W_L \) is the power of the laser, \( V_{cell} \) is the volume of the photoacoustic cell, \( \omega_n \) is the resonant frequency. The cell constant is not only dependent on the frequency, dimensions of the resonator, and quality factor but also the spatial overlap of the laser beam with the standing acoustic wave [1].

3. Experimental setup

Depending on the carbon hydrogen bonds there are four groups of hydrocarbons: paraffin’s, olefins, cyclo paraffin’s and aromatics. As can be observed in Figure (1) strong C-H absorption bands can be observed for oils at a wavelength region of 2300 – 2500 nm and higher. Compact, cheap laser sources with sufficient power in this region are readily not available. The availability of a cheaper, compact, high power laser at 1728 nm has resulted in the selection of this wavelength.

![Infrared spectra of oil](image)

Figure 1: Infrared spectra of oil [4]

The schematic of the experimental setup is shown in Figure 2. The optical source used in the setup is a 5 mW, 1728 nm room temperature DFB laser from Nanoplus. The divergence of the laser diode is corrected by using an aspheric lens. The cell is of resonant type operating with its first longitudinal mode at around 2.45 kHz. An electret microphone (Knowles EK-23028-C36) is mounted flush in the wall of the resonator at the anti-node of the acoustic wave.

The laser is modulated with a sinusoidal waveform generated by the lock-in amplifier in order to generate a photoacoustic signal. The cell was used with gaseous hydrocarbon contamination like n-
heptane due to ease of handling and high vapour pressure. A calibrated commercial instrument Innova 1412 was used as a reference to monitor the contamination level of n-heptane. The microphone signal was amplified and band pass filtered using the Kemo Bench master 8.41 filter/amplifier unit and recorded on a personal computer through a Stanford research SE830 lock-in amplifier with a time constant $\zeta = 3$ seconds. The intension of using longer averaging time was to reduce the noise of the measurements (noise reduces with the square root of the averaging time). The cell was evacuated to a level of $10^{-2}$ bar before filling a new level of contamination. As seen in figure (3) a test rig has been built to generate flow rates of up to 4500 Nl/min, pressures of up to 10 bar and a controlled level of contamination of oil, and n-heptane. This high pressure is reduced to atmospheric pressure before entering the PA cell. A heater is used in the test rig in order to clean the rig of any residual contamination present, by flushing the test rig with heated air.

Figure 2: Experimental setup

Figure 3: Test rig

Figure 4: Signal/noise ratio vs. buffer/resonator diameter ratio [5]
4. Results

The laser used in the experiments operates at a wavelength of 1728 nm, where several C-H absorption bands are observed. These absorption lines are free from interference of water vapour [4]. The design of the cell is a critical parameter in determining the sensitivity of the setup. Though it is expected that no signal is generated when no contaminant gas molecules are present, background noise that is coherent with the photoacoustic signal is present due to heating at the windows by the laser light, this being the limiting factor for sensitivity. Numerical modelling shows that an optimization of the cell geometry can increase the signal to noise ratio. As seen in Figure 4 above the signal to noise ratio increases by increasing the diameter of the buffer inserted on either side of the resonator.

The resonator is made from aluminum with a length of 63 mm and inner diameter of 15 mm. Buffers which acts as acoustic (\(\lambda/4\)) notch filters with a diameter of 39 mm and lengths of 31.5 mm are added on either side of the resonator to help reduce window noise. The cell is designed for a quality factor (\(Q\)) of 125 and a setup constant (\(C_n(\omega_n)\)) of 1662 Pa cm/W.

The concentration of n-heptane was varied and the magnitude of the PA signal was measured by the lock in amplifier as observed in Figure 5. The solid line shows a linear fit of the data.

![Figure 5: Plot of PA signal (mV) vs. Contamination level of n-heptane (mg/m³)](image)

5. Future work and conclusions

The experiments demonstrate the use of the photoacoustic setup as a compact detector for the monitoring of gaseous hydrocarbon contaminants. The laser wavelength is selected to achieve a good balance between strong absorption lines, size and cost. The mechanical noise due to a chopper used in some setups to modulate the laser source is avoided by instead using the modulation of the injection current to generate a photoacoustic signal. In the above setup, the modulation frequency is set to the resonance frequency of the cell, this resonance frequency fluctuates due to variations of temperature and gas composition. Thus it is necessary to perform scans of the modulation frequency across a small range around the resonance frequency. The background signal due to the absorbance of the windows, and the cell walls by the laser light also poses the limiting value of sensitivity. Future developments to improve sensitivity include use of optical microphones (Sensitivity 5V/Pa) to avoid electromagnetic interference and the use of a 300 mW, 1728 nm semiconductor laser with the intention of increasing the sensitivity.
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