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The optimisation of a paired emitter-detector diode optical pH sensing device

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ABSTRACT

With recent improvements in wireless sensor network hardware there has been a concurrent push to develop sensors that are suitable in terms of price and performance. In this paper a low-cost gas sensor is detailed, and significant improvements in sensor characteristics have been achieved compared to previously published results. A chemical sensor is presented based on the use of low-cost LEDs as both the light source and photodetector, coupled with a sensor slide coated with a pH sensitive colorimetric dye to create a simple gas sensor. Similar setups have been successfully used to detect both acetic acid and ammonia. The goal of this work was to optimise the system performance by integration of the sensing technique into a purposely deigned flowcell platform that holds the colorimetric slide and optical detector in position. The reproducibility of the sensor has been improved through this arrangement and careful control of deposited film thickness. The enhanced reproducibility between sensors opens the potential of calibration-free measurement, in that calibration of one sensor can be used to model the characteristics of all sensors in a particular batch.

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1. Introduction

Over the last two decades chemical sensing has consistently remained an important research topic owing to a diverse range of applications in various areas such as clinical assays [1–3], food quality monitoring [4,5], process monitoring [6], and environmental sensing [7,8].

In order for sensing devices to be practical, key parameters such as sensitivity, reliability, reproducibility, sensing range, automation, cost, size and ease of use must be carefully considered [9]. The use of paired LEDs, in which one LED serves as a light source (forward biased), while the other serves as a photodetector (reverse biased) is an approach that can be used to make very low-cost, low-power chemical sensors. In these so-called PEDD (paired emitter–detector diode) devices, the light coupled between the emitter and the detector passes through a chemical coating wherein the color changes due to chemical effects resulting in a chemical sensor [10,11]. PEDDs are low-cost, small in size, consume a small amount of power and have a simple principle of operation. Most importantly, this LED sensing technique shows good sensitivity and excellent signal to noise ratios with target gas detection in the low ppm to ppb region being reported [10–12].

In search of sensitive, reproducible, reliable and low-cost sensors ideal for sensor net applications [13], we present here the optimisation of a colorimetric gas sensor which is coupled with a PEDD optical measurement device, with a significantly enhanced performance over previous sensor configurations [11,14]. These improvements have been achieved through the alteration of the fabrication method for sensor film preparation, and the development of a flowcell housing to hold both the sensor film and the optical detection setup securely in position. The development of the flowcell with a mechanical flow control allows for a greater level of control over our samples, and as a result more effective experimentation.

2. Experimental

2.1. Film preparation

The chemical formulation used is based on a pH indicator dye (bromophenol blue) a phase transfer salt (tetraoctylammonium bromide) and a polymer (ethyl cellulose) dissolved in 1-butanol as developed by Lau et al. [17]. The formulation undergoes a colour change from blue in its alkaline state towards yellow in the presence of acidic species. Previously, films have been drop cast directly onto the LED surface [10,11], but this results in uneven coatings and sensors that were not very reproducible. In this paper a novel flowcell housing has been designed to hold and secure the position of the sensor slide coated with the chemo-sensitive film. Designing the platform to accommodate a sensor slide allows the deposition of the coating onto a PET substrate, which can be done in a more reproducible fashion than direct coating of the LED surface. The films were prepared by casting a 50 mm \times 40 mm film onto a 1.8 mm thick clear flat PET sheet. The films prepared

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Fig. 1. Top view is a horizontal cross-section of the sensor flowcell, bottom is an exploded view of the flowcell and the sensor components.

were measured to be $5-6\,\mu m$ thick using a Wyko NT1100 Optical Profiling system. $10\,mm \times 10\,mm$ squares were cut from this film, from its most uniform midsection for experimentation.

2.2. Flowcell fabrication

The flowcell was CNC milled from PTFE (Teflon), chosen for its high temperature stability and more importantly its high chemical resistance [16]. The housing accepted two LEDs, a rubber gas seal, four locking screws, the coating slide and two M5 pneumatic gas fittings (Fig. 1) which acted as the gas inlet and outlet. Two LEDs were aligned on the same axis facing each other, on opposite sides of the coated slide and acted as the optical detection system. The emitter LED pulses light on one side of the slide and the light intensity reaching the detector LED is modulated by the colour change of the colorimetric film indicating the presence and concentration of our target species (acetic acid). As part of this study the paired emitter–detector diode setup was redesigned in a flowcell configuration for a number of reasons.

• In many previously published paired emitter-detector diode setups there have been a number of system or performance

variables being very loosely controlled or often not being controlled at all. It was decided that a redesign of the sensor housing would be used to more effectively control these experimental variables, which were affecting system readings and inhibiting good repeatability. These variables included distance between LEDs (the distance was optimized to achieve maximum light coupling through use of an adjustable test rig), alignment of LEDs, position of coating slide, etc.

- The flowcell design was also chosen to allow for more effective experimentation, where close control of the gases coming in contact with the colorimetric pH indicator slide could be guaranteed.
- The flowcell design also allows for the system to be easily coupled with a grab sampler. Running the device with the grab sampler ensures that there will be a gas flow inside the device at all times, not like in previous setups where the device relied on plumes of contaminant blowing in contact with the sensor of their own accord. (Furthermore, this also opened up potential for the sensor to be developed as a standalone sampling and sensing system).
- The flowcell design importantly accommodates the use of a coated slide. Immobilisation of the dye coating on a transparent substrate facilitates the use of more reproducible deposition techniques to create the colorimetric coatings.

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