Journal of Engineering Research & Education
Vol. 3, 2006 (106-112)

Detection of Landfill Odour Using Quartz Crystal Microbalance (QCM)

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Received : 14 January 2006 / Accepted : 22 November 2006

ABSTRACT

Odour is considered as the main environmental pollution agent in Malaysia. Even though the standard permissible level for odour in Malaysia has not been set yet as for air and water, malodour is considered as one of the main polluting agents, since the people has started to be concerned about the malodour. Conventionally, the odour was detected by the olfactometer, which used human as its panel for smell. But, the human panels are unable to produce a consistance result, especially for the continuous monitoring. In this study, the Quartz Crystal Microbalance (QCM) was used as an odour detector. The QCM is basically a mass sensitive device with the ability to measure a very small change on a quartz crystal resonator in real time. This system basically comprises of an array of sensors as well as data acquisition and components analysis. A Principle Component Analysis (PCA) is used to define three distinct regions, according to the time of the samples being taken and hence allow the landfill odour differentiation.

Keywords: Odour, Quartz Crystal Microbalance (QCM), Principle Component Analysis (PCA)

INTRODUCTION

The development of the odour detection equipment is still a major problem because of a lack of technical solution. Although the odour is recorded utilizing language expression, it is still difficult for us to associate actual odour with the expression and regenerate it again by only using that expression. It is important to establish the electronic method for odour recording.

In the 1964, the study by King demonstrated that quartz crystal could be used as absorption detectors (mass sensitive devices) by coating the crystals with liquid Gas Chromatography (GC) stationary phases [1]. Nowadays, there's a growth interest on the use of quartz crystal as gas sensor for identification of odour, fragrance and aromas [2].

Following are some of the previous study of which show successful examples of the application of the sensor system based on the quartz crystal microbalance. Chay and Shih had detected organic vapour by using multichannel piezoelectric quartz crystal. By applying an artificial neural network, they recognized organic vapour and demonstrated the distinction between these organic vapour [3].

Ulmer *et al.* [4], used a polymer coated quartz microbalance as one of the sensors to identify odour and flavour. They optimized the quantitative analysis of known gases mixture. Cui *et al.* [5], made use of quartz crystal microbalance with deposited polypyrole for odour mapping. Better odour discrimination was achieved using this system. Moreover, odorants could be clearly distinguished by their particular position.

The Quartz Crystal Microbalance (QCM) sensor is an example for a very sensitive detector of mass change [6]. Quartz crystal is an earth mineral that is used as the basic materials of the sensors. Figure 1, show the experimental set up used to analyze the odour produced from the landfill area.

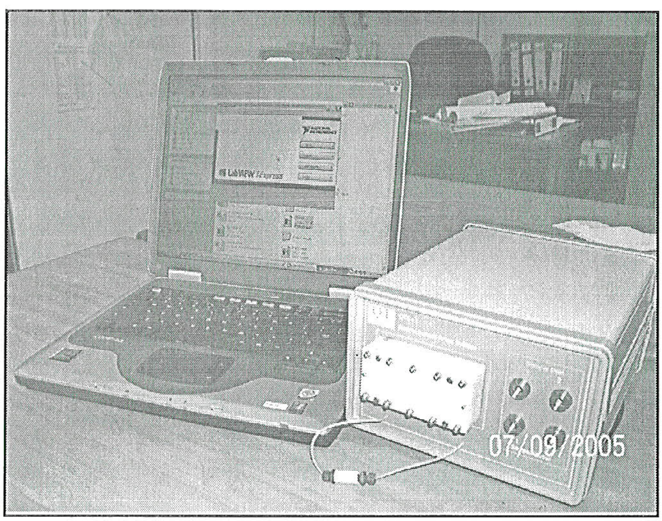

Figure 1. Experimental set up for odour detection.

Many studies had shown the application of the QCM sensors for odour detection. Among those are gas mixture analysis [6], solvent vapour detection [3], detection of organic vapour in the environment [7, 8], determination of single odour substances in various types of botanical species such as fruit and flower [9], detection of odour emission from composting facility [10], etc.

The QCM sensors were developed by using lipid blended with PVC as sensing material. Upon excitation by application of a suitable A.C voltage across the 2 electrodes, the device can be made to oscillate at a characteristic resonant frequency. As the mass being adsorbed or placed onto a quartz crystal surface. Then the oscillate frequency will start to change and it can be shown by Sauerebrey Equation [11]:

$$\Delta f = -2.3 \times 10^{6} f_o^2 (m/A)$$

where,

Δf = sensor frequency change (Hz)
f_o = oscillation frequency of quartz crystal (MHz)
m = mass change of the adsorbed analytes (g)
A = area of the crystal surface

The method for determining mass and measuring the change in the oscillation frequency of a quartz crystal is extremely sensitive, since this type of crystal has a sensitivity of about 10^{-9}gram [12].

Analytes that are present in the surrounding space (i.e. in sample bags) of the QCM sensors will interact with the sensitive coating materials. The adsorption or absorption of the analytes by the coating materials result in a mass change on the sensor surface. Consequently, the mass change on the sensor surface is converted to the frequency changes.

The QCM are rugged, low power, easily miniaturized and capable of direct chemical sensing in liquids. Moreover, QCMs can be adapted for many different uses by developing coatings that response to different target molecules, adding to their versatility.

MATERIAL AND METHODS

Sensor Preparation

The quartz crystal was an AT-cut piezoelectric with fundamental frequency of 10MHz, 8.0 mm diameter, with gold electrodes of 4.0mm in diameter on both sides (Quartz Technology Ltd. UK)

In this experiment, 8 quartz crystals were used as sensors. Each quartz crystal was coated with Polyvinyl Chloride (PVC) blended lipid on the sensing membrane. 10mg of PVC was blended with 100mL of liquid and 5mL of tetrahydrofuron. The lipid membrane used in this study as shown in Table 1 below.

Table 1 Lipid membrane used in this experiment.

Sensor	Lipid Used
1	Decyl alcohol (DA)
2	Oleic acid (OA)
3	Dioctyl phosphate (DOP)
4	DOP: TOMA = 5:5 (D:T = 5:5)
5	DOP: TOMA = 3:7 (D:T = 3:7)
6	Trioctyl methyl ammonium chloride (TOMA)
7	Oleyamine (OAm)
8	DOP:TOMA= 9:1 (D:T= 9:1)

5mL of this solution was coated on both sides of the quartz crystal by spin coating method at 1500rpm. The coated crystals were then dried for a few minutes. The frequency of the quartz crystals sensor due to coating process was measured by using home-build data acquisition system. The frequency shifts of the coated quartz crystal vary from 1000Hz to 8000Hz, depending on the lipid coating materials.

Odour Sampling

Air samples from the landfill area were collected for the analysis by QCM. A battery power mini piston air sampling pump (Cole-Parmer) was used to collect air samples in Tedler Bags. The air samples taken at the landfill area was in the morning and evening for a period of five days. The sample of fresh air was also collected for comparison.

Odour Measurement

Figure 2 below shows the experimental set up for the odour measurement.

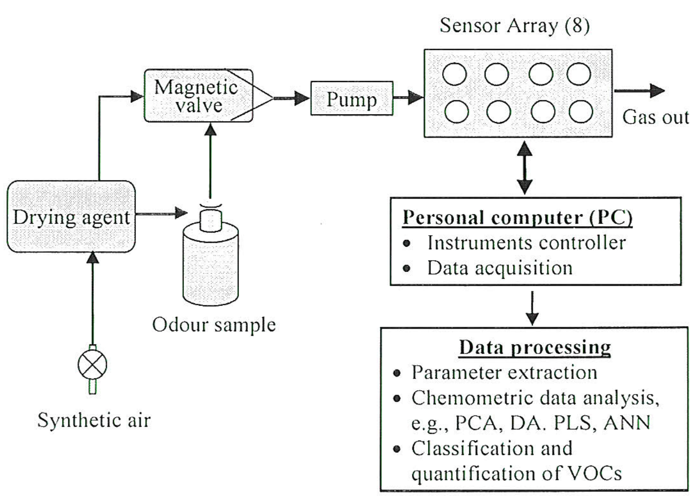

Figure 2. Experimental set up for odour detection.

The measurement procedures were as follow. First, the valve was switch opened and the pump was turned on. The cell was then supplied with the compressed air without the odourant from the air samples. Then, the routing valve was activated to supply the sample vapour and the resonant frequency shift was measured. Finally, the routing valve was deactivated and the cell was purged for the cleaning purposes. The flowrate of the gas is set at 230mL/min. The frequency shift was measured for 30s when the odour sample was supplied to the flow cell. The cleaning process time was set for the duration of 150s.

RESULT AND DISCUSSION

The baseline frequency shift reading of all sensors were recorded for five days, during the data acquisition measurement in August 2005. From the figure 3 below, it shows that the sensors were stable, except for sensor DOP: TOMA, which was drifted to lower vales as five days go by.

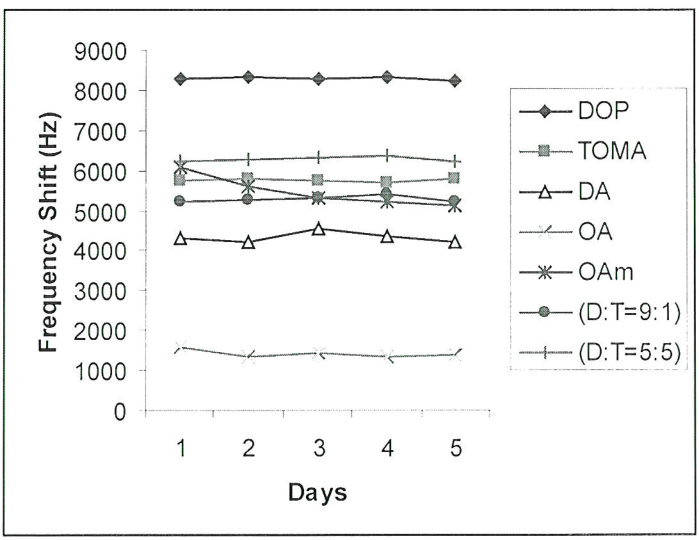

Figure 3. Baseline frequency shift during data acquisition (August 2005).

The PCA now was used to access clustering within the data. PCA is a linear method that has been proven to be efficient to distinguish the response of QCM.

The result of the PCA is shown in Figure 4. The result of the PCA shows that all sensors are corelated, as nearly 90% of the variances of the data are contained within the first principle.

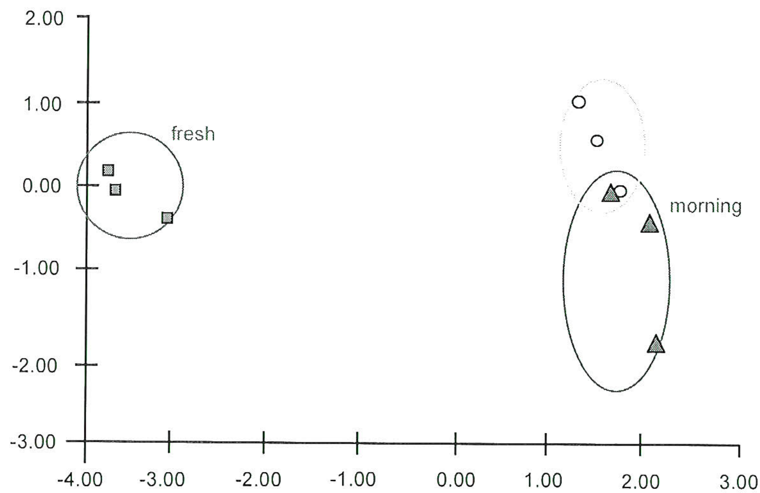

Figure 4. The scores plot of the principle component analysis.

CONCLUSION

Although a great deal of work still needs to be done. The preliminary results are very promising. QCM can recognize and quantify the odours released by the landfill site. The technique of using one electronic nose as a measuring device for the chemical background noise is totally new and possibly a solution to the problems usually encountered when working in ambient atmosphere with such devices. Aging and replacement of sensors need to be investigated and considered while developing the sensor for odour monitoring.

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