Implementation of a quartz microbalance (QMB) sensor array - based instrument and olfactometer for monitoring the performance of an odour biofilter

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Abstract

An olfactometer and a self developed instrument based on a quartz microbalance (QMB) sensor array were implemented under field conditions for monitoring the performance of an odour biofilter. In an industrial-scale biofilter (measuring 19.0 m x 6.8 m x 1.5 m) the filter material was made up of wood chops. The waste gas originating from a composting facility was biodegraded in the biofilter with a surface-loading rate of 9.5 $m^3m^{-2}h^{-1}$. The monitoring instrument consists of an integrated preconcentration unit, a micro-controller, and six QMB sensors with different sensitive coating materials and their supporting parts. Using the principal component analysis (PCA), a clear difference between odour emission after and before the biofiltration process was shown. Chemical analysis using gas chromatography indicated that at least 26 odorous compounds were identified from the waste gas. Based on the results from the olfactometer, it was demonstrated that the biofilter has reduced 92% of the odour intensity.

Keywords

Biofilter, odour, olfactometer, quartz microbalance, sensor array

INTRODUCTION

Biofiltration, a waste gas biodegradation process well-known world wide, is a cheap and simple method to reduce the pollutant content of off-gases from industrial and biological processes. There has been extensive research on biofiltration with regard to improvement of this environmentally friendly waste gas emission control technique including studies on microbiological aspect [Jones et al., 1997; Juteau et al., 1999], technical characteristics [Rihn et al., 1997; Mohseni and Allen, 2000; Pagella and De Faveri, 2000], performance [Sorial et al., 1997; Wani et al., 1998; Jorio et al., 2000], modelling [Zarook et al., 1996, 1997; Alonso et al., 1999] and on the economical aspects [Gao et al., 2001].

Along with the research and development in the field of sensor technology, modernisation of the biofiltration monitoring technique has also been taking place. At present, the use of the olfactometer together with the human nose and gas chromatography are not the only ways to monitor the performance of a biofilter. Odour sensors are a sound alternative for this monitoring task, as it is clear the human nose cannot be exploited for online monitoring. Besides the health implication of the odorous compounds for humans, application of an olfactometer may cause olfactory fatigue to the panel members. With regard to gas chromatography, not only is it an expensive monitoring method [Gardner and Bartlett, 1999; Bailey and Persaud, 2000], it also requires considerable operational skills.

In this study, an instrument based on the QMB sensor array together with an olfactometer was implemented to monitor the performance of an odour biofilter. The olfactometer was not intended

to be a part of the future online monitoring system, but was used merely as a reference in order to calibrate the QMB sensor array - based instrument. Here, the objectives of the experiment were:

- 1. To implement a QMB sensor array based instrument and an olfactometer to monitor the odour biofiltration process
- 2. To define the relationship between the sensor signals and the odour units from the olfactometer
- 3. To conduct a statistical analysis (PCA) to determine the different odour patterns before and after the biofiltration process.

METHODS

In an industrial-scale biofilter (measuring 19.0 m length x 6.8 m width x 1.5 m height), the filter material was made up of wood chips. The waste gas originating from a composting facility in Bonn, Germany (capacity 12,000 tons year⁻¹) was biodegraded in the biofilter with a surface-loading rate of 9.5 m³ m⁻² h⁻¹. The performance of a biofilter is measured by comparing the pollutant content of the inlet flue gas to the outlet flue gas. In the same way, the performance of an odour biofilter is determined by measuring the concentration of the odour compounds of the waste gas before biofiltration and those of the clean gas after biofiltration. The performance can be expressed, for example, as removal efficiency, i.e., the fraction of the odour concentration removed by the biofilter and expressed as a percentage:

Removal efficiency =
$$\left[\frac{C_{in} - C_{out}}{C_{in}}\right] x100$$

where : C_{in} = odour concentration of the waste gas before biofiltration [ppmv; g m⁻³] C_{out} = odour concentration of the clean gas after biofiltration [ppmv; g m⁻³]

In a similar way, in this study the performance of the odour biofilter was assessed by applying olfactometer for measuring the odour unit [OU] of the waste gas before biofiltration and the clean gas leaving the biofilter as follows:

Performance of odour biofilter,
$$\eta = \left[\frac{OU_{before} - OU_{after}}{OU_{before}}\right] x 100$$

where: $\eta = \text{odour biofilter performance [%]}$ $OU_{before} = \text{odour unit of waste gas before biofiltration [OU]}$ $OU_{after} = \text{odour unit of clean gas after biofiltration [OU]}$

To determine the performance of the biofilter, 2 sampling points were required, i.e. one point before and another after the biofiltration process. The samples from the waste gas before the biofiltration process were taken from a pipe connecting the humidifier and the biofilter, while those from the clean gas after the biofiltration process were taken from the top of the biofilter (Figure 1). A special trapezoid-shaped metal structure was developed to facilitate waste gas sampling. Both sample flow rates (before and after the biofiltration process) were sucked using a pump (*KNF Neuberger*) at the same rate, i.e. ca. 700 ml min⁻¹.

A cooling unit (*M&C Products GmbH*) was placed just behind the sampling point in order to reduce the water content of the sampled waste gas. Using this cooler, the waste gas sample temperature can be kept at about 5 °C. This was necessary since the waste gas was always water-saturated due to the humidifying treatment prior to biofiltration. The water condensed in the cooling unit was then set free. A filter (0.2 μ m; *Gelman, Acro 50 NPT*) was placed behind the cooling unit to prevent coarse material entering the measuring chamber. The gas sample was then channelled to the measuring chamber of the sensor array (*HKR Sensorsysteme GmbH*). The sensor system consists basically of an array of six QMB sensors with different sensitive coating materials and an integrated a micro-controller. A personal computer was used to control the sensor system and for data acquisition.

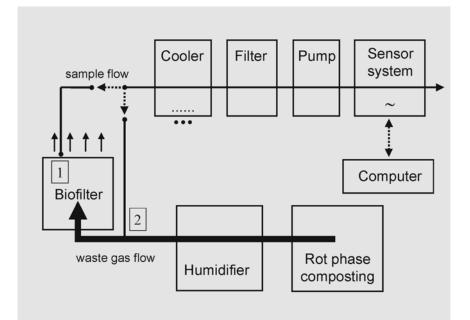


Figure 1. Sampling points (1 and 2) and instrument set-up

A thermal desorber preconcentration unit developed at the Department of Agricultural Engineering, University of Bonn [Nieß, 2002) consisting basically of 35 mg Tenax GR was integrated in the sensor system to pre-concentrate the odour components and to improve the sensitivity and detection limit of the sensor system. The use of this unit was based on the previous findings that odour detection using only sensors results in a low signal, whereas implementation of the thermal desorber unit showed considerably better results [Yuwono et al., 2002, 2003]. In operating conditions, the unit was heated up to 250 °C within 60 sec by using an inside heating coil in order to release the pre-concentrated odour substances. The measurement cycle lasted 540 sec and comprised three phases, i.e. 300 sec for sample enrichment, 210 sec for sample heating up and peak measurement, and 30 sec for cooling down.

Figure 2 shows the separation of the analyte (i.e. only odour molecules) from the sample flow. The water content of the sample was reduced by means of a cooler, while a filter was used to detain the dust in the sample. Then a thermal desorber preconcentration unit was implemented to enable collection of the analyte from the sample. The rest of the sample, i.e. non-odorous substances such as CO_2 and methane, were then released.

For the olfactometer test, gas samples were taken using Tedlar bags. Two groups of olfactometer samples were taken, i.e. before and after the biofiltration process. For each sample group, 3 Tedlar bags were used, but only 2 were used for analysis. Analysis using an olfactometer (TO-7, *Ecoma GmbH*) was carried out on sampling day. The sniffing panel consisted of staff from the Department of Agricultural Engineering, University of Bonn. The chemical analyses were carried out by a research project partner (*HKR Sensorsysteme GmbH*, München) using gas chromatography mass spectrometry (GC-MS).

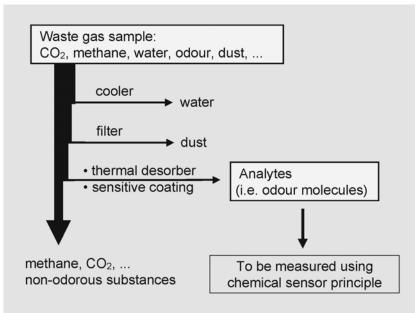


Figure 2. Separating analytes (i.e. odour molecules) from the gas sample

RESULTS AND DISCUSSION

Response of the QMB sensor array to the presence of odorous substances before and after biofiltration is presented in Figure 3. There is a clear difference in the average frequency change before and after the biofiltration process. The average response of the QMB sensor array after the biofiltration process is only about 40% of the responses before the biofiltration process. However, this does not mean that the odour intensity after the biofiltration process (outlet) is 40% of the previous one in the biofilter inlet: The test using the olfactometer showed that the odour intensity after biofiltration remains just about 8% (Table 1). The sensor response (average sensor frequency change) cannot be directly correlated to the odour intensity reduction.

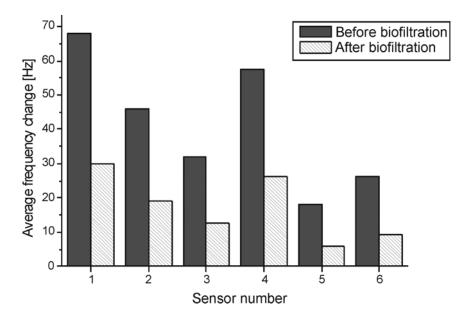


Figure 3. Average frequency change of QMB sensor array as response to the biofiltration process

The odour intensity is defined here as ten times the logarithmic value of associated odour concentration [Oberthür, 1990]. The odour intensity before biofiltration process was ca. 46 dB, whereas after biofiltration process was ca. 35 dB. These values are ten times logarithmic values of 43000 OU and 3350 OU, respectively. Zhang et al. (2002) demonstrated the similar fact that when a strong source odour was diluted, its intensity decreased linearly with the dilution level on a log-scale.

If a linear relationship between OU and the sensor frequency change (see Table 1) is drawn, it indicates that the average OU before the biofiltration process is about 1024 times the average sensor frequency change. This means that a 1-Hz sensor frequency change indicates a change of 1024 OU, whereas after the biofiltration process, 1 Hz represents only a ca. 176-OU change. It can be concluded from these measurements that this relationship is poor, since the difference between both numbers is extremely large.

	Before biofiltration		After biofiltration	
-	Probe 1	Probe 2	Probe 3	Probe 4
Odour unit	49000	37000	2600	4100
[OU]	Avg. = 43000		Avg. = 3350	
Odour intensity	46.9 ± 2	45.7 ± 2.2	34.1 ± 1.7	36.1 ± 1.4
[dB] –	Avg. ≅ 46.3		Avg. ≅ 35.1	
Avg. sensor frequency	40	44	13	25
change [Hz]	Avg. = 42		Avg. = 19	
[OU/Hz]	1024		176	

Table 1.	Olfactometer results as c	ompared to the average	ge sensor frequency change
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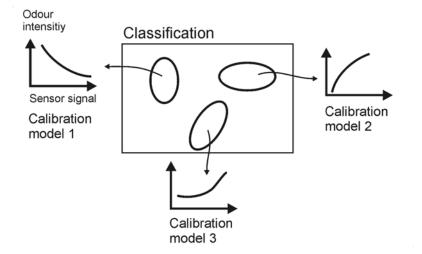


Figure 4. Proposed scheme of odour monitoring with chemical sensors: classification followed by calibration (adapted from Boeker et al., 2003)

Two important steps are necessary to improve this relationship, i.e. classification and calibration (Figure 4). Classification on the gas mixtures should be carried out since biogenic odour sources

varies. In this case, there are two odour mixture classes, i.e. before and after biofiltration. Afterwards, a number of calibrations for different gas mixtures are required to set up a more reliable relationship, so that the difference between both numbers becomes as small as possible. A series of tests on various odour emission sources are required to verify the relationship. It is also necessary, in the near future, to conduct a series of extensive experiments in order to develop an exact correlation between the sensor response and reduction of odour intensity that was measured using an olfactometer. The relationship can also be extended to various odour emissions in the environment. Such a correlation can be then used as a guide for measuring odour intensity.

An analysis using PCA (principal component analysis) demonstrated a clear odour separation between before and after the biofiltration process (Figure 5). During the biofiltration process, odorous substances were consumed by microorganisms. The products of this biodegradation process are mainly carbon dioxide (CO₂), water vapour (H₂O) and heat. The biodegradation changes most of the odour substances into non-odorous substances. Therefore, as indicated in Figure 6, a substantial change took place during this process.

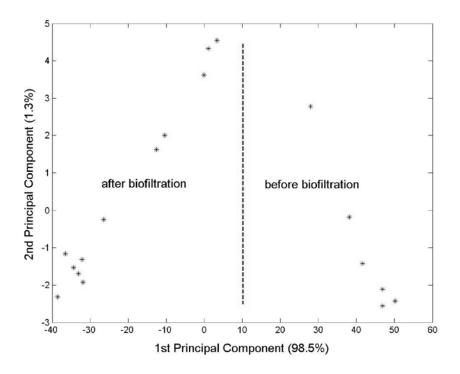
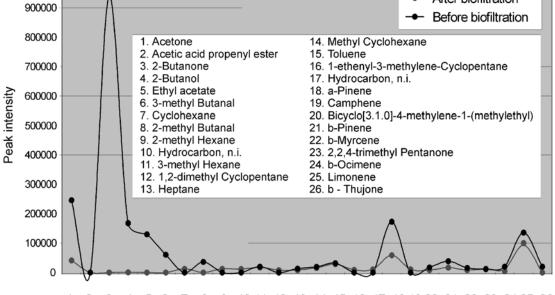


Figure 5. Principal component analysis (PCA) showing a clear odour separation before and after the biofiltration process

Based on Table 1, the odour biofilter performance can be calculated as follows:

$$\eta = \left(\frac{43000 - 3350}{43000}\right) x 100 = 92\%$$

which means that the biofilter has reduced 92% of the odour intensity. It can be considered as a good biofilter performance. This finding is in accordance with the result of chemical analysis using gas chromatography - mass spectrometry (GC-MS), as depicted in Figure 6. The peak intensity of a number of substances such as 2-butanone, 2-butanol, ethyl acetate, acetone and a-Pinene were reduced remarkably after biofiltration process.



1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18 19 20 21 22 23 24 25 26

Figure 6. Chemical analysis showing the peak intensity changes of substances in the sample gases after and before biofiltration process

CONCLUSIONS

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The following conclusions can be drawn from this experiment:

- 1. A QMB sensor array based instrument and an olfactometer were applied to monitor the performance of an odour biofilter. The odorous waste gas originating from an industrial composting facility was detected by the QMB sensor array and its presence expressed as a sensor frequency change. At the same time, the odour intensity of the waste gas was measured by an olfactometer and expressed in odour unit [OU]. The same procedure was carried out for the clean gas sample leaving the biofilter. Based on the results from the olfactometer and GC-MS, it was found that the odour biodegradation process through the biofilter had been good.
- 2. The preliminary results indicate that the relationship between OU and sensor frequency change is still poor. A series of extensive test on various odour emissions including classification and calibration is, therefore, required to set up this relationship if the sensor array is in future to be used as an instrument for direct odour emissions monitoring.
- 3. Statistical analysis showed that there was a clear odour pattern separation between before and after the biofiltration process.

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