

# Compost Pile Monitoring Using Different Approaches: GC–MS, E-nose and Dynamic Olfactometry

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**Abstract** The evaluation of odour emissions associated to the composting process is complex because these emissions depend on several factors such as the raw material to be composted, the different stages of the composting process, meteorological conditions, and others. For this reason, the aim of this paper is to compare complementary approaches to monitor odours. The odour source selected for this study is green waste compost at different maturity stages. The study site is a composting facility located in the south of Belgium. The compared approaches were: a portable e-nose developed by the Environmental Monitoring Research team (Arlon, Belgium) to monitor odorous emissions from the composting piles; chemical analyses performed in the laboratory using a GC–MS (manufactured by Thermo) to analyse volatile organic compounds which were collected by active sampling on Tenax TA<sup>®</sup> sorbent simultaneously to the in situ e-nose measurements and olfactometric measurements to determine the odour concentration ( $\text{ou}_E/\text{m}^3$ ) using the Odile olfactometer (Odotech). The portable e-nose was also used in the laboratory with compost odour samples collected in bags. The large numbers of data sets obtained were explored by statistical methods such as principal components analysis. The results obtained highlight the

advantages of monitoring the composting process with these three approaches. Each approach gives different information about the composting process and the emissions generated. While the e-nose is capable of identifying some chemical family emissions and some activities such as turning steps, the GC–MS identifies each chemical compound emitted and dynamic olfactometry quantifies the odour concentration ( $\text{ou}_E/\text{m}^3$ ) in relationship with these emissions.

**Keywords** Composting · Dynamic olfactometry · E-nose · VOCs · PCA · GC–MS

## Introduction

In recent years, odours annoyance from different industrial sources has become a serious environmental concern, especially in the case of odour emissions from municipal solid waste (MSW) plants. The principal reason for the increase in odour complaints is that industrial plants are situated nearer to urban and residential areas [1]. The complexity of emissions and the subjectivity of the odour perception can partially explain the difficulties involved in determining regulation and control. Although odour legislation in the form of acts or regulations has been enacted in North American, Asian, Australasian and European jurisdictions [2], the current legislation does not cover certain aspects related to the control and regulation of odour emissions. For instance, the various techniques usually used to measure off-odours are not yet standardised. Currently, there is only one European olfactometry standard to measure the concentration of odour expressed in  $\text{ou}_E/\text{m}^3$ . Hence, there is a great need to improve the usual techniques or to develop new ones to help lawmakers.

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While composting is one of the most efficient ways to treat different kinds of organic wastes, it is always associated with off-odours and volatile organic compound (VOCs) emissions. Despite being present at trace level concentrations, most volatile compounds are malodorous and have very low odour thresholds, potentially resulting in odour impacts on nearby populations [3]. In addition to the potential for odours annoyance, volatile compound emissions from MSW can also have health impacts due to their toxic nature, and can also cause the corrosion of process equipment [4, 5].

Odours can be produced during the different stages of the process: reception, conveying, turning over, composting, and curing or storage. However, the major emissions of odours and VOCs occur from the reception step and during the turning over of the compost piles for the aerobic biological treatment [6]. Incomplete or insufficient aeration can produce sulphur compounds of intense odour, whereas incomplete aerobic degradation processes result in the emission of alcohols, ketones, esters and organic acids [7, 8]. The chemical composition of odour emissions depends on the waste materials, the level of decomposition and the type of handling [9]. The composting process can be developed using different raw materials such as municipal solid waste, poultry waste, wastewater sludge and green waste.

A previous paper [10] revealed the importance of odour emissions from landfill sites and composting facilities using different methods such as chemical analyses, dynamic olfactometry, sniffing teams and electronic noses. By using these methods in a complementary manner and exploiting the strong points of each, it is possible to tackle the whole investigation of sites such as landfills, examine the emissions of the volatiles that cause the odour and assess the odour annoyance in the environment to verify compliance with an exposure limit.

The European standard EN 13725 [11] for determination of odour concentration by dynamic olfactometry is a sensorial technique that allows determining the odour concentration ( $\text{ou}_E/\text{m}^3$ ) of an air sample. A selection of panellists sniffs the sample in various dilution levels in odourless air and indicates if they smell the odour of the diluted sample or not. This method is used in an air-controlled laboratory a few hours after sampling in the field. Nowadays, this method has different applications such as the sensorial characterisation of the aroma of food. Many authors use this standard method to quantify odours emitted by the composting process [12, 13]. However, the physiological differences in the sense of smell among people often lead to subjective and highly uncertain results.

The e-nose is able to monitor gas emissions in real time in the field and to link them to the odour concentration expressed in odour units [14]. However, some limitations

have to be considered, for instance humidity sensitivity, drift, and high detection limits. While analytical techniques allow identifying and quantifying the chemical compounds emitted from these gas emissions, the chemical composition of the gas mixture does not always represent the odour perception.

All these measurement techniques are complementary and have drawbacks and advantages. According to [15], the e-nose offers clear advantages with regard to chemical analysis in terms of its rapidity of execution. In comparison with panel tests, the e-nose also presents other advantages aside from its rapidity such as lower costs, repeatability of the results and continuous monitoring. The e-nose permits analysing air samples with a low odour level, and even with contents that are hazardous to health. Once the fingerprints of the odorous sources are learned by the device, the e-nose is, in principle, able to predict the class of an unknown sample and subsequently identify its source. As for the identification of odours, the quantification of odours by the e-nose requires developing mathematical models. Sensory techniques allow the sensorial component of odours to be evaluated both qualitatively and quantitatively using the human nose as a detector. Unlike analytical analyses, sensorial techniques present lower accuracy and repeatability due to their subjective nature and their results must be carefully interpreted [16].

Chemical analyses such as gas chromatography-mass spectrometry (GC-MS) provide information on the chemical composition of the emissions in terms of chemical concentration. However, the relationship between the chemical profile and the odour is not always well established [17]. Moreover, this method is time consuming and costly, particularly when used for routine analysis purposes.

The main objective of this paper is to compare these three approaches usually used to monitor odours and to highlight their utility in monitoring odour emissions as well as the processing of green compost piles. It is also necessary to validate each of these methods in relation to the others for some typical cases of odour emissions.

## Materials and Methods

### Field Campaign and Data Collection

The study was carried out on a composting facility located in the south of Belgium. The waste treatment plant consists of a municipal solid waste reception unit, an area dedicated to the composting of piles of green waste, a landfill area and a garbage collection point where customers can get rid of their waste, such as textile, paper and cardboard and plastic bottles. The odour source selected for this study is

green waste compost at different stages of maturity. Typically, the facility has six to eight piles at different stages of maturity. The final pile size is about 2.5 m high and 50 m in length. Aeration is achieved by turning the piles about twice a week and the composting process is carried out under natural aeration where the air flow out the pile is not monitored. The odours released by the compost vary with time and type of handling. Green waste composting is a slow process and the composition of the compost piles varies so slowly that the odour emissions will depend on how they are managed. For this reason, VOCs and odour emissions are more influenced by turning activity and parameters such as temperature or humidity.

The measurement campaign was carried out over 2 months from 17 May 2012–20 July 2011. Three different batches of green waste were randomly monitored during this period. In the field, real time e-nose measurements were performed in both the morning and in the afternoon under different meteorological conditions. The measurements were made during 30 min in order to achieve the perfect stabilisation of the sensor signals. At the same time, VOC adsorption was realised on Tenax TA<sup>®</sup> sorbent. Moreover, samples of the emissions released by the green compost piles were simultaneously collected in two 60 l-Tedlar bags. A sealed barrel maintained under negative pressure by a vacuum pump was used to collect the odour in a bag. The aspiration generated by this vacuum pump was determinant to collect the samples since the natural aeration of the pile was not enough to fill the bag with odorous air. One of the samples was analysed by olfactometry and the other with the e-nose in the laboratory within a maximum of 30 h after sampling.

The aim of using the same e-nose in the lab and in the field was to compare the results of the e-nose obtained by online measurements in the field with the results obtained after sampling in bags followed by e-nose measurement in the lab with odourless-odour cycles. For the laboratory e-nose measurement, odourless samples were collected in the field about 500 m up wind of the compost piles where the operator was unable to smell any odours.

Table 1 shows the scheduled activities carried out during the measurement campaign. The different activities performed the same day are specified. For each measurement day, at least two different approaches were used to compare the different information under the same conditions of temperature, humidity, maturity time of compost, etc.

In the field, bags with one sample were collected from the compost piles. This sample was analysed by dynamic olfactometry. When GC–MS analysis was possible, the odour sample was collected on Tenax TA<sup>®</sup> sorbent at the same time as the odour samples were collected in Tedlar bags. Simultaneously, the e-nose analysed the emissions of

the compost piles. The odourless sample was collected in the field immediately after the odour samples were collected.

### Sensorial Analysis

In this study, we used the dynamic olfactometry sensorial method to determine the odour concentration of an odorous air sample, expressed in European odour units per cubic metre ( $\text{ou}_E/\text{m}^3$ ) according to the standard EN 13725:2003. The analysis was performed using an Odile olfactometer (Odotech, Canada) at the Olfactometric Laboratory of the Environmental Sciences and Management Department, Campus d'Arlon, University of Liège. The laboratory is maintained at a temperature below 25 °C and is “odour free”.

A panel of six members judges the samples of gas odours. A decreasing step sequence in geometric series of factor 1.58 and the triangular choice are used. A no odour response is allowed. Only a “with certainty” odour response is considered to be correct. Dynamic olfactometry is used to obtain the European odour concentration of samples. The odour concentration represents the number of dilutions with neutral air necessary to bring the concentration of the sample to its odour perception threshold concentration. It is assumed that the results obtained by dynamic olfactometry have a confidence level from half to double the value of the odour perception threshold concentration. The analysis is carried out after the odorous gas is sampled in the field.

### Electronic Nose System

The odour emissions of the compost piles were monitored using a portable e-nose developed by the ULg team. The e-nose consists of a battery-powered sensor array and a PC board with a small keyboard and a display. The array contains six commercial metal oxide sensors (Figaro<sup>®</sup>) (TGS822, TGS2620, TGS2180, TGS842, TGS2610, and TGS880). Each of these sensors has a specific application from the manufacturer: the TGS822 sensor is sensitive to organic solvents, the TGS880 sensor to alcohols, the TGS842 sensor to natural gas and methane, the TGS2610 sensor to propane and butane, and the TGS2620 sensor to hydrogen, alcohols and organic solvents. Although TGS2180's response to compost emission is low, this sensor is very sensitive to water vapour and it is considered only for humidity correction but not to develop an odour classification model.

The variation of the conductance of the sensors is recorded. The array is placed inside a thermostatic chamber and linked to a pump with a constant flow rate of 200 ml/min. The chamber temperature is kept at 60 °C by a heating

**Table 1** Activities schedule during measurement campaign

DAY	GC-MS	DINAMYC OLFACTOMETRY	E-NOSE	
			E-NOSE (Field)	E-NOSE (Laboratory)
17/05/2011				
19/05/2011				
24/05/2011				
25/05/2011				
27/05/2011				
06/06/2011				
14/06/2011				
16/06/2011				
20/06/2011				
21/06/2011				
23/06/2011				
27/06/2011				
29/06/2011				
05/07/2011				
06/07/2011				
07/07/2011				
12/07/2011				
13/07/2011				
19/07/2011				
20/07/2011				

resistor and natural cooling, thanks to a suitable control system. Relative humidity of the sensor chamber is also recorded. Specific software controls the hardware and allows the acquisition of the sensor signals. The raw electrical conductance of the sensors is recorded every 15 s in the local memory and it can be monitored with a little laptop in real time. The data are then downloaded in an external computer for off-line processing by multivariate tools using Statistica and Matlab software. The features considered for the data processing are the raw sensor electrical conductances (S), normalised by the square root of the sum of all the sensor conductance values squared, without any reference to base line.

#### Physicochemical Measurements

The VOCs were collected by active sampling on Tenax TA<sup>®</sup> sorbent for 30 min with a flow rate of 150 ml/min using a calibrated air pump (Gilian<sup>®</sup> Gilair 3SC) with a low flow module, and a Gilibrator to calibrate the flow. The VOCs are desorbed and analysed in the laboratory using a thermal desorber (TD, Unity, Markes International) linked to a GC–MS (Thermo). During desorption, a split ratio of 5 was applied on the sorbent tube. The carrier gas was helium. A constant flow rate is set. Its value is evaluated at 1.5 ml/min.

The chromatographic column is a CP-sil 8 CB MS (5 % phenyl and 95 % dimethylpolysiloxane) with a length of 50 m, an internal diameter of 0.25 mm and a film thickness

of 0.4 µm. The running conditions for the GC were: initial temperature 38 °C, initial time 7 min, rate 5 °C/min and final temperature 200 °C. The MS was used in the scan mode. The mass range was from 33 to 300 amu.

The compounds were identified by comparing the mass spectra obtained to the reference mass spectra of the NIST database (National Institute of Standards and Technology, United States) using XCallibur software. AMDIS software is also used to confirm the identification. Moreover, the retention times of the analysed compounds were compared to the retention times of the previously identified compounds. External calibration was used to quantify the identified compounds. The concentrations of the substances were evaluated by external calibration at several calibration points (four different concentrations in the range of expected concentration). Standard samples were prepared in methanol solution and injected onto the cartridges by the “spiking” method using the Markes International<sup>TM</sup> calibration solution loading rig. External calibration was carried out using toluene following the ISO 16000-6 standard and the NIOSH guidelines for VOC screening (Method 2549, VOC screening using multibed thermal desorption sorbent tubes, GC–MS, 1996). Hence, only semi-quantitative analyses were performed.

#### Statistical Analysis

The data sets were analysed with software using statistical methods (Statistica<sup>®</sup> and Matlab<sup>®</sup>). Pattern recognition

techniques were used. Principle components analysis (PCA) is a linear unsupervised technique that is very useful for analysing, classifying, and reducing the dimensionality of numerical datasets in multivariate problems. PCA is often used for visually inspecting the evolution of observations over short time periods [18].

Principle components analysis allowed highlighting correlations between the sensors, the chemical families and the odour concentration ( $\text{ou}_E/\text{m}^3$ ).

## Results and Discussion

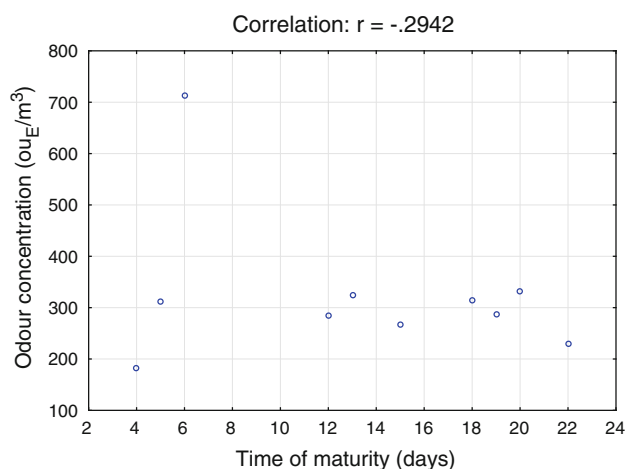
### Univariate Correlation Between the Composting Maturity Time and the Chemical-Olfactometry Variables

#### *Evolution of Odour Concentration with Time of Maturity*

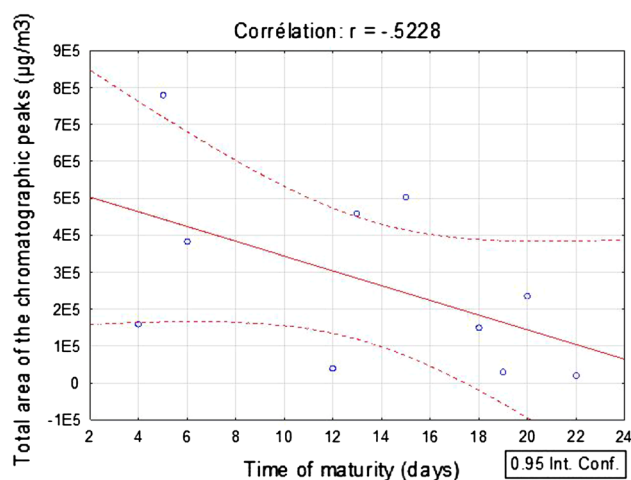
Measurements taken over a total of 22 days were used to investigate the relationship between the e-nose data, the chemical analysis and the odour concentration during the maturity time of the composting piles.

The results (Fig. 1) show that the odour concentration ( $\text{ou}_E/\text{m}^3$ ) tends to increase during the first 6 days of maturity time to a value of  $714 \text{ ou}_E/\text{m}^3$ , after which an expected decrease in odours is observed. The initial odour concentration may be due to higher biological degradation in the first days of the composting process which generates volatile emissions. Odours are mainly caused by VOCs such as alcohols, esters, ketones, aldehydes, acid, terpenes and sulphur compounds. This is in line with [19], who showed that odour concentration increases in the first stage of a composting cycle over 7 weeks.

A rather low odour concentration range for the compost emissions was observed. This may be due to the



**Fig. 1** Evolution of odour concentration ( $\text{ou}_E/\text{m}^3$ ) with maturity time (days)



**Fig. 2** Evolution of total VOC concentration ( $\mu\text{g}/\text{m}^3$ ) with maturity time (days)

composition of the green compost. Vegetable remains contain high amounts of cellulosic material which degrade slowly by microbiological activity. Moreover, green waste releases lower emissions than household waste. In addition, it can be also an indicator of a good composting process. For these reasons, the less VOCs emitted, the lower is the odour concentration.

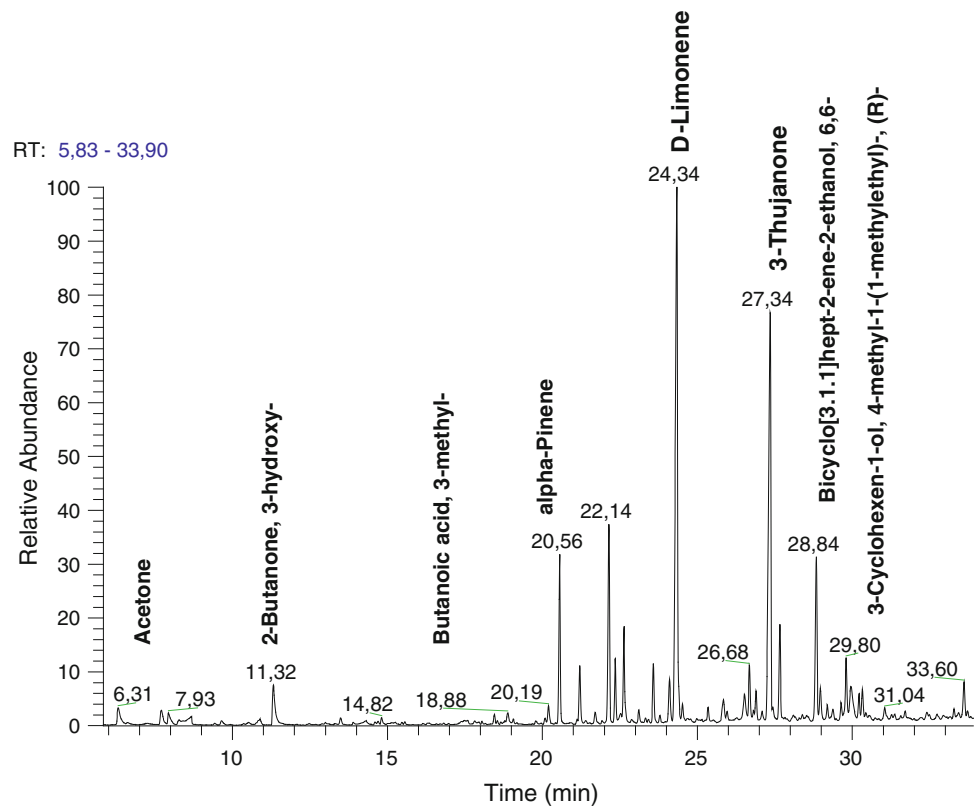
The odour concentration and total VOC emissions showed different trends. Although both decreased with time of maturity, the odour concentration increased until day 6 of the composting process, while the total VOC emissions decreased with composting time. The evolution of total VOC concentration with maturity time can be observed in Fig. 2. Although a decrease was observed for the odour concentration and total VOC emissions, the data sets are not well correlated. For this reason, we studied the evolution of each chemical family with maturity time in order to understand the differences in behaviour. The study of each chemical family allowed determining that the concentration of some families as well as the odour concentration increased the first days of composting.

#### *Evolution of Chemical Families with Time of Maturity*

The chemical families studied were terpene, ketone, aldehyde, organic acid, alcohol, ester, hydrocarbon and sulphur compounds. The results of the GC–MS analysis allowed detecting the highest concentration (as  $\mu\text{g}/\text{m}^3$ ) of some VOCs at the start of the process. In [20], a total reduction of 98.6 % in the VOC concentration was calculated from the start to the end of the composting process.

Figure 3 shows a representative chromatogram obtained during the monitoring process on day 13 of composting maturity. As can be observed in the figure, some representative compounds of the composting process such as

**Fig. 3** Chromatographic profile obtained during the monitoring of 13th composting process



**Table 2** Percentage of identified chemical families for simple collected on 13th day of composting process

Chemical families	Concentration (mg/m <sup>3</sup> )	Relative abundance (%)
Terpene	215	46.65
Ester	137	29.77
Hydrocarbon	29.5	6.45
Alcohol	26.3	5.75
Ketone	21.3	4.65
Aldehyde	9.9	2.15
Acid	3.8	0.86
Sulphur compounds	1.9	0.42
Others	15.3	3.30
Total	460	100

Limonene and Pinene are typical compounds emitted during the composting process. Table 2 shows the percentages of the chemical families identified in the sample collected on day 13. Terpene and ester compounds are the most abundant chemical families. Terpenes are produced by the degradation of lignin contained in wood-derived products as well as by fruits and plants (orange, mint). Although alcohol, acid and sulphur compounds are less abundant, these chemical families generate strong odour emissions even in low concentrations. The most common compounds

found in the samples were: acetic acid, acetone, limonene, benzene derivatives, hexane, pinenes, toluene, naphthalene, undecane and xylene. These VOC compounds, which are associated with the composting process, are widely cited in the literature [21, 22] and [23]. However, this chemical composition does not represent the global odour of solid waste, which is the result of the reception of a complex volatile mixture. A comparison between chemical composition of emissions generated on day 6 when the odour was high and after day 12 when the odour was lower is performed. Total VOCs on days 13 and 15, as well as Terpene, Ketone and Ester compounds were more abundant than on day 6. However, the acid compound emission was strongly higher on day 6. Actually, the acid concentration has decreased from 222.4 mg/m<sup>3</sup> on day 6 to 3.9 mg/m<sup>3</sup> on day 13. This observation could explain that the organic acids were the major compounds responsible for the odorous emission generated during this composting.

Regarding the evolution of some chemical families during the composting process, the concentration of terpene and alcohol decreased with time of maturity as well as the total concentration (μg/m<sup>3</sup>). On the other hand, the concentration of ketone, acid and ester compounds decreased with time of maturity, but increased on day 5, and day 5 and 6 of maturity time, respectively. These compounds exhibited the same behaviour as the odour concentration. The hydrocarbon compounds did not show a clear trend with time of maturity.

**Table 3** Correlation coefficient between the conductance of e-nose sensors with odour concentration, total COV concentration, each chemical family concentration and with composting time

	Sensors					
	TGS 822	TGS 842	TGS 880	TGS 2180	TGS 2610	TGS 2620
Odour concentration	<u>0.11</u>	<u>0.2</u>	0.04	0.02	0.05	<u>0.16</u>
Total COV	0.6	0.5	0.6	0.06	0.5	0.6
Terpene	<b>0.7</b>	0.5	<b>0.7</b>	0.1	0.6	<b>0.7</b>
Ketone	<b>0.7</b>	0.4	<b>0.7</b>	0.02	0.6	0.6
Aldehyde	−0.06	0.08	0.08	−0.1	0.03	−0.1
Acid	0.5	<b>0.7</b>	0.5	0.2	0.6	0.5
Alcohol	<b>0.8</b>	<b>0.7</b>	<b>0.8</b>	0.1	<b>0.8</b>	<b>0.9</b>
Hydrocarbon	0.3	0.4	0.3	−0.1	0.3	0.4
Esters	<b>0.8</b>	0.5	<b>0.8</b>	0.07	<b>0.7</b>	<b>0.8</b>
Time of maturity	−0.3	−0.3	−0.3	−0.2 <sup>a</sup>	−0.3	−0.2

Correlation coefficient (linear regression)

Underlined values indicate correlation coefficient above 0.1 for the odour concentration

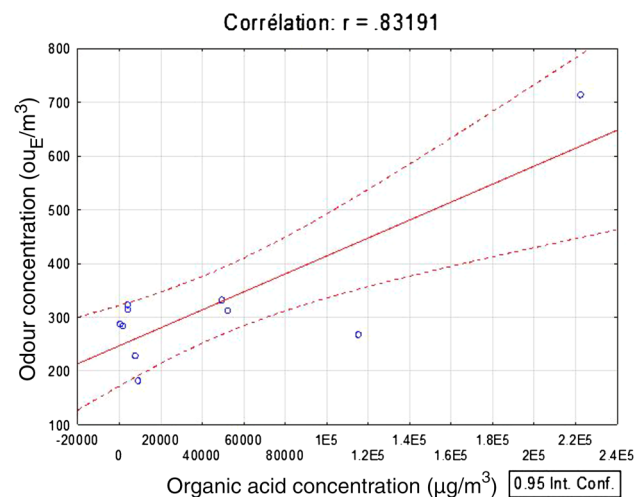
Bold values indicate correlation coefficient above 0.6

 $P < 0.05$ <sup>a</sup> With high dispersion

The aldehyde compounds increased during the composting process. This increase could be due to interferences with the sorbent material used for the sampling (Tenax TA<sup>®</sup>). According to [24], despite recent advances in gas chromatography, several limitations remain. In particular, this analytical technique is still limited by: (1) the broad concentration range of the compounds present in odorous emissions and the difficulties inherent in the preparation of multi-component gaseous standards; (2) the potential decomposition of thermally unstable volatiles compounds due to high operating temperatures in both thermal desorption units and injectors; and (3) water interferences.

#### Relationship Between Chemical Families and Odour Concentration

The odours produced depend on the presence of organic volatile compounds in the exhaust air [25]. A comparative study on the relationship between the concentration of chemical families and odour concentration was performed. The results showed a good correlation ( $r^2 = 0.83$ ) between the concentration of acid compounds and the odour concentration detected with dynamic olfactometry (Fig. 4). Moreover, acid compounds are one of the most odorant chemical families. Like the odour concentration, the major emission of acid compounds is associated to the first stage of the composting process when the fresh material is more easily degraded by microorganisms. In spite of the good correlation obtained, more intensive monitoring during the first 8 days of the composting process must be carried out to obtain acid compound concentration values when the

**Fig. 4** Relationship between odour concentration ( $\text{ou}_E/\text{m}^3$ ) and organic acid concentration ( $\mu\text{g}/\text{m}^3$ )

odour concentration shows values between 400 and 600  $\text{ou}_E/\text{m}^3$ . The results did not show a high correlation with the other families or with the total chemical concentration. These results may explain the fact that some VOCs in such low concentrations, which cannot be detected by GC–MS or by e-nose, produce very high odour emissions. For this reason, the simultaneous monitoring with e-nose, dynamic olfactometry and GC–MS allows us to obtain complementary information in order to perform a more complete study about the odour emissions in real time and the VOCs that produce these emissions.

### Univariate Correlation Between E-nose with Odour Concentration, Maturity Time and Chemical Families

The univariate correlation between the e-nose sensors and the odour concentration as determined by olfactometry, the maturity time of the composting piles and the chemical families obtained by GC–MS were studied and is shown in Table 3. The results obtained in this section show a positive correlation between the conductance of the six sensors and odour concentration, except for the TGS 2180, which is more sensitive to humidity than the other chemicals. This sensor was used to correct the humidity, but not to develop the odour classification model or determine the chemical families. A positive correlation was also observed between the sensors and the chemical family concentrations (except for the aldehyde family, see previous explanation in section “Evolution of chemical families with time of maturity”).

The linear correlation of each sensor with odour concentration was quite low, with the highest being  $r^2 = 0.2$ . The correlation with VOCs was about 0.5–0.6, slightly higher values than for the odour. Although all the sensors have a similar linear coefficient for the total concentration of hydrocarbon compounds, each sensor has a different coefficient for each chemical family.

The bold values show the best correlation coefficient between each sensor and each chemical family. The results indicate that the sensors were more sensitive to alcohol and ester compounds.

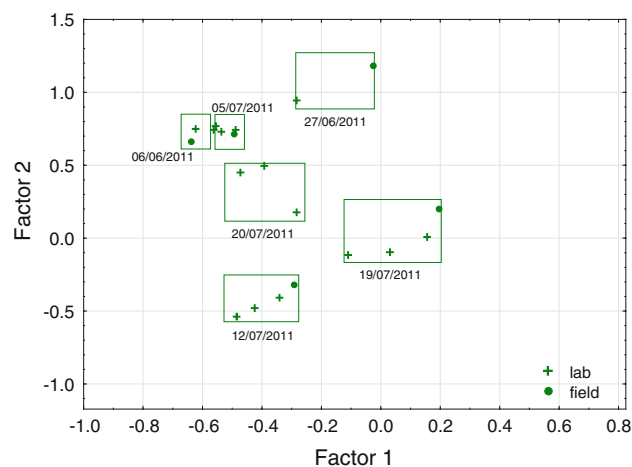
Finally, a negative coefficient was observed for composting time. Although this coefficient was low (about 0.3), it can be concluded that each sensor reacts as a function of maturity time. Due to the decrease in emissions according to maturity time, the sensors are able to respond to this timer.

Hence, odour concentration did not linearly correlate well to maturity time and chemical composition, with the exception of the acid compounds. The further processing of the data sets would probably be useful to develop an approach.

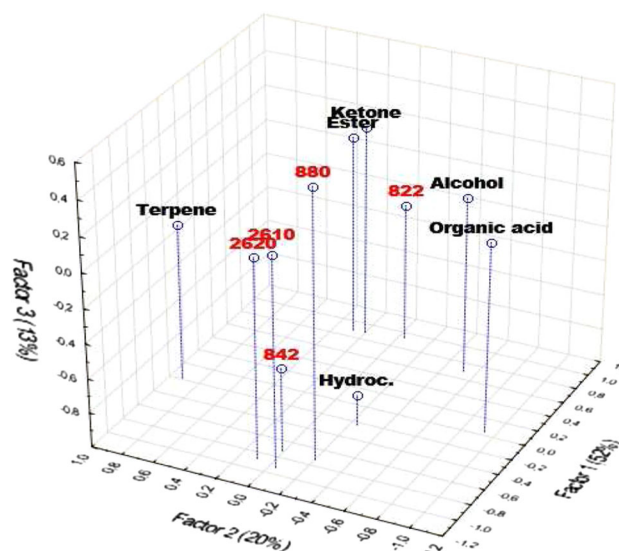
### Multivariate Analysis: The E-nose Data Set

The previous results indicate the appropriateness of performing multivariate analysis (principal components analysis, PCA) to test the efficiency of the e-nose in monitoring the compost pile emissions.

Firstly, it is of interest to note (Fig. 5) the correlation between the results of the e-nose in the field, where the samples were taken directly with the portable e-nose over the compost piles, and the laboratory analysis where the samples collected in 60l-Tedlar bags were analysed as explained in the materials and methods section. As can be seen in the figure, the response of the e-nose in the field and



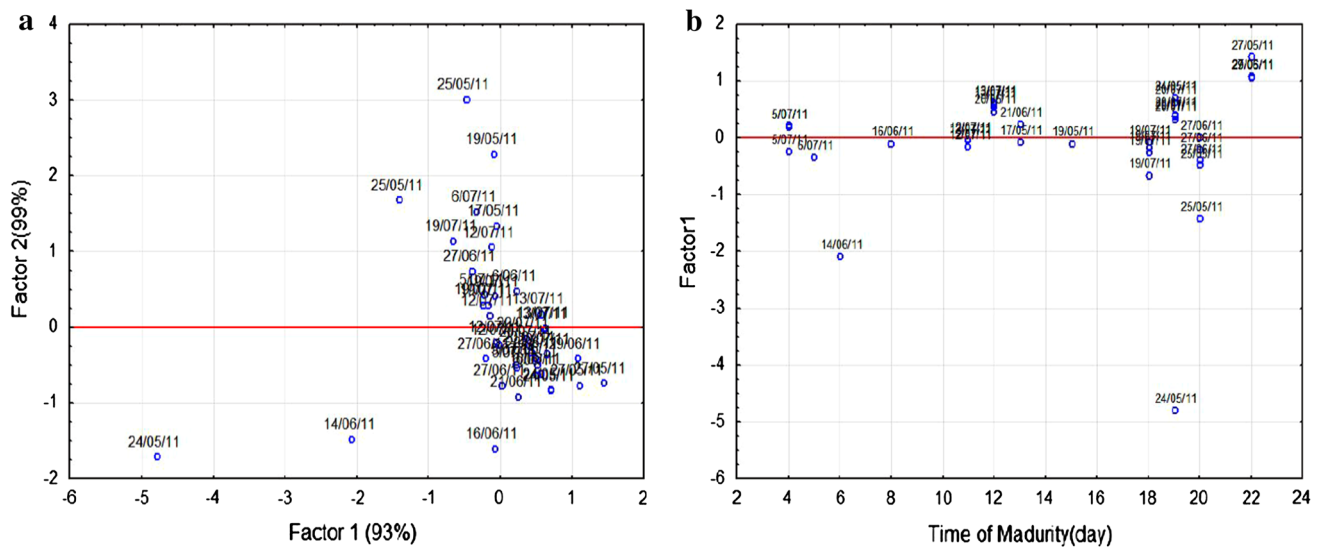
**Fig. 5** Correlation between the e-nose results in the field and in the laboratory measurement



**Fig. 6** Illustration by PCA of quantitative chemical family concentration and sensor sensitivity

in the lab during 6 days of monitoring was very similar. It should be noted that all the sensors showed a good correlation between responses in both the field and in the laboratory. These results highlight the capacity of the e-nose to monitor in real time and indicate the importance and advantages of monitoring in situ with the portable e-nose. Figure 6 shows a PCA analysis performed with the normalised variable taking into account the results of the 24 measurements made in the field and in the laboratory. It is possible to observe the sensitivity of the TGS822 sensor to alcohol compounds and the sensitivity of the TGS842 sensor to hydrocarbons. Moreover, the TGS2610 and TGS2620 sensors have similar sensitivities. These results highlight the sensitivity of the e-nose to some odorous chemical compounds.





**Fig. 7** **a** Illustration of PCA: Score plots of the e-nose measurements, factor 2 versus factor 1. **b** Illustration of PCA: Factor 1 versus Time of maturity (days)

PCA was also performed (Fig. 7a) with all the measurements obtained in all the compost piles (forty observations) with the portable e-nose. The odourless samples and the response of the TGS842 sensor (only sensitive to hydrocarbons) were not considered. The combined response of the sensors permitted obtaining PCA factors and provided global information on the chemical profile and chemical concentration of all the compounds. The PCA, with the data described above, explained 93 % of the total data variance. The PCA plot highlights the non-conforming measurements carried out on May 25th, June 14th and June 16th.

When considering the new components (factors 1 and 2) rather than the sensor responses, factor 1 versus maturity time was represented so that non-conforming measurements could also be highlighted (Fig. 7b). The results revealed some abnormal data which can be explained. June 14th coincided with a high odour concentration value (the maximum value before a decrease in the odour concentration). On June 24th the composting piles were turned, which may have increased the gas emissions. This activity could also have influenced the measurement taken on June 25th. If we consider these new components rather than the sensor responses and we plot this factor as a function of time, exceptional measurements can be highlighted.

#### Comparison of Dynamic Olfactometry, E-nose and GC–MS

To highlight the importance of using these approaches as complementary techniques, their characteristics are compared in this section.

Firstly, while the e-nose is able to monitor continuously in real time, GC–MS and dynamic olfactometry only allow

us to analyse a sample collected from one point of the odour emission in a short period of time (a few minutes). Moreover, only the e-nose allows doing analyses directly without previous sampling in tedlar or nalophan bags in the case of dynamic olfactometry and in Tenax TA<sup>®</sup> sorbent in the case of GC–MS.

On the other hand, it should be noted that GC–MS and dynamic olfactometry are expensive techniques. Although e-noses are available with a wide variety of prices, for this kind of applications a low-cost sensor device is sufficient. For instance, in this study we used a simple e-nose device developed by the ULg team whose cost is significantly lower than GC–MS and dynamic olfactometry.

Finally, although the e-nose and GC–MS give us more information than dynamic olfactometry, the data processing is more tedious. The e-nose allows responses to be obtained each second from a number of sensors during an unlimited period of time. This information must then be processed continuously by statistical methods to interpret it. On the other hand, analyses by GC–MS allow obtaining a wide amount of chemical compounds that must be identified and quantified.

#### Conclusions

The results show that the odour concentration of the green compost emissions was not linearly correlated with the total VOC concentration. However, by exploring the concentration of the chemical families as a function of maturity time, we have determined that the acid compounds can explain the odour trend.

This study shows that the odour of green compost emissions begins to increase during the first step of the composting process (a peak odour concentration of 714 ou<sub>E</sub>/m<sup>3</sup> was observed day 6), while a continuous decrease occurs from day 12 to the end of the monitoring process. The same trend was observed in the evolution of acid compounds, both of which are associated to high microbiological activity and the degradation of organic matter.

Thanks to the use of these two techniques, GC–MS and dynamic olfactometry, it was possible to understand the odour evolution of the compost emissions and to identify the chemical family responsible for the odour variation.

Although the e-nose was unable to accurately predict the odour concentration and maturity time when using common linear multivariate approaches such PCA, a positive correlation between the sensors and the odour concentration was obtained. The combined responses of the sensors (the PCA factors) provided global information about the chemical concentration of all the compounds and highlighted some specific events. The aeration activities of the compost piles led to an increase in VOC emissions, which were detected by the e-nose. Compared to dynamic olfactometry and GC–MS, the advantage of the e-nose is that it is capable of monitoring emissions and odours in real time. The e-nose could be used as a rapid and easy detection tool to monitor the composting process in the field as well as compost quality (acid compounds).

In short, pile compost monitoring must be carried out using these complementary techniques:

- *Dynamic olfactometry* as it allows us to evaluate the odour concentration as a function of biological activity during the composting process.
- *GC–MS* as it allows us to detect which chemical compounds are responsible for odours during periods of high emissions. For instance, acid compounds which are one of the most odorous families.
- *Electronic nose* as it is capable of monitoring emissions and odour in real time. Although the e-nose was unable to accurately predict the odour concentration and maturity time when using PCA, the combined responses of the sensors (factors) provided global information about the chemical concentration and highlighted some specific events.

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