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RESEARCH ARTICLE

Evaluation of the odor impact of some environmental gaseous pollutants: calibration of the methodology and preliminary results

Valentina Coccia[1,2] · Mattia Manni[1] · Alessandro Petrozzi[1] · Andrea Nicolini[1,2]

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Abstract

During the last decades, it has been observed a growing interest on odor impact because of the frequenter social acceptability problems about energy plants handling and processing stored organic materials (e.g., biogas plants, landfills, farms, distilleries, etc.). In this context, the UNI EN 13725:2004 indicates the "dynamic olfactometry method" as validated, recognized, and adequate measurement procedure for estimating the odor concentration. This protocol is carried out by a panel of specifically trained and selected human receptors, but alternative analytical methodologies are currently under discussion. This work aims to describe the initial steps of a wider research toward the definition of a new analytical protocol for monitoring odor concentration. The alternative methodology is here presented through the implementation of a case study: stored organic materials exploited on an energy plant in Central Italy. The paper describes the preliminary activities related to the survey of the case study (i), the definition of alternative methods and devices for conducting emissions sampling (ii), and the adopted experimental approach (iii). Finally, preliminary results are also presented (iv). The resulting protocol, once validated, could be employed by local authorities to measure both the odor impacts and the effectiveness of specifically designed mitigation strategies.

Keywords Odor impact · Low-speed wind tunnel · Social acceptability · Dynamic olfactometry · Round robin test

Nomenclature

OIA	Odor impact assessment
OT	Odor threshold
MS/GC	Mass spectrometry-gas chromatography
OU	Odor unit
M1	Type one molecules (hydrogen sulfide, dimethyl sulfide, ammonia, and ethyl mercaptan)
M2	Type two molecules (terpen, linalohol, nerol, geraniol, citronellol)
LSWT	Low-speed wind tunnel
ILC	Inter-laboratory comparison

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✉ Mattia Manni
manni@crbnet.it

[1] CIRIAF, Interuniversity Research Center on Pollution and Environment "M. Felli", CRB Section, Biomass Research Center, Via G. Duranti, 63, Perugia, Italy

[2] Department of Engineering, University of Perugia, Via G. Duranti, 63, Perugia, Italy

Introduction

The investigation of the odor emissions from agricultural and industrial facilities is an uncertain field of study since the phenomenon is strictly affected by subjective perception. During the last decades, regulations and standards have been focusing on this topic because a significant part of urban citizens moved to traditional rural areas (Blanes-Vidal et al. 2009; Guiterrez et al. 2014; Libby and Sharp 2003; Melse et al. 2009a, b; Pillai et al. 2010; Walgraeve et al. 2015) and livestock zones became larger and more concentrated (Blanes-Vidal et al. 2009; Melse et al. 2009a, b; Donham et al. 2007). Those factors led to an increment of both complaints about odor nuisance, and social acceptability problems regarding energy plants which handle, and process stored organic materials (e.g., new biogas plants, landfills, farms, distilleries, etc.). Although minimizing the olfactory impact of those facilities represents one of the main challenges of these years (Longhurst et al. 2004), the local discomfort caused by those odor sources is still difficult to be quantified. As reported on (Capelli et al. 2012), for performing an odor impact assessment (OIA) procedure, it is necessary to sample and determine the frequency of measurement. But the studies carried out by

Barczak and Kulig (2017) shows how difficult it can be conducting such assessments in a country—Poland in this case—that does not have specific regulations. The Italian case does not differ from the Poland one: the European Standard UNI EN 13725:2004 (2004) has not been yet integrated on the regional regulations, except in Lombardia and Abruzzo. Hence, several measurement methods were proposed as well as different possible interpretations of the results. Despite of it, only the European Standard would be considered in this study.

On this basis, there are different instrumentations and workflows which can be employed for evaluating the odor impact on population. Among those, the mains are dynamic olfactometry (i), sensory analysis as electronic nose (ii), and chemical analysis as gas chromatography (iii). The dynamic olfactometry (i) is the most widely applied method and it provides an opinion—statistically defined—on the perceptibility and magnitude of the investigated odor samples by means of a controlled dilution made by olfactometer. This method does not provide the identification of the chemical species but leads to the definition of their odor threshold (OT) that corresponds to the number of dilutions necessary for nulling the odor perception. The number of dilution value is used predominately in the USA instead of odor concentration (ou/m^3). The panelists are invited to judge the perceptibility of the sample, by determining the number of dilutions which equals the OT. This procedure has been largely investigated by several studies in literature, evaluating the OT of specific molecules such as hydrogen sulfide (Greenman et al. 2004; McGinley and McGinley 2004), mercaptans (Vermeulen and Collin 2002; Vermeulen et al. 2003), and ethanol (Nagata 2003; Cometto-Muniz and Abraham 2008). The results can be used on specific dispersion models for predicting the impact on the neighboring areas (Nicell 2009; Nicolas et al. 2008; Nimmermark et al. 2005; Romain et al. 2013; Yu et al. 2010), evaluating the efficiency of mitigation strategies (Friedrich and Kosmider 2012; Hansen et al. 2014; Martens et al. 2001; Melse and Moi 2004; Miller et al. 2004), and testing the impact of new facilities (Nicell 2009; McGinley 2002; Munoz et al. 2010). Then, sensory (ii) and chemical (iii) analyses represent two alternative methodologies for measuring odor impacts which are not scientifically accepted yet, even if the newest regulations—currently under review—are oriented toward including those among the accepted techniques. Both are strictly connected to the dynamic olfactometry that up to now has represented the only interface with the human sensitivity. Indeed, the electronic nose required an initial calibration (*Training Stage*) that is performed through olfactometers and panelists. The device acquired information about the odor footprint of molecules contained on the proposed samples to successively estimate the odor concentration (ou_E/m^3) by evaluating the presence of those molecules. Among the advantages guaranteed by this device, the electronic nose can monitor in continuous the source and record the discomfort events. The frequency of those events and their magnitudes permits to evaluate the phenomenon (Gralapp et al. 2001).

The here presented study aims to investigate the quantitative correlation between the concentration of the molecules in odor mixtures—measured by analytic instruments (MS/GC)—and the odor concentration expressed as odor units (OU) exceeding the OT. A similar approach has been already assessed on some studies present in literature whose main goal is to correlate the chemical composition and the odor concentration (Kim 2011; Blazy et al. 2015; Hansen et al. 2016; Wu et al. 2016, 2017). The analyzed case studies range from pig slaughterhouse (Blazy et al. 2015; Hansen et al. 2016) to landfill (Wu et al. 2017). Hence, the proposed workflow would give an original contribution through the implementation of a different case study from the ones mentioned above: stored organic materials from a distillery factory, used for feeding an energy plant, are investigated.

Indeed, the produced molecules which could be related to the odor emissions can be different depending on the process and the conducted activities. In that regard, the literature review demonstrated that agro-industrial facilities like the case study produce two main molecules:

i. Type 1 molecules (M1): these molecules are emitted during the activities of composting, storing piles, and handling of biomass such as hydrogen sulfide, dimethyl sulfide, ammonia, and ethyl mercaptan (Guitiérrez et al. 2015);
ii. Type 2 molecules (M2): they are specific molecules emitted by the storage, handling, and processing of organic material employed in the vinery factories, which are aromatic molecules such as terpen, linalohol, nerol, geraniol, and citronellol (De Rosa and Castagner 1994).

Concerning the quantitative measurement of the concentrations of M1-type chemical compounds, some empirical correlations between molecule concentration and OT can be found in literature (van Gemert 2011). Thus, MS/GC-panel campaigns are combined on characterization protocol allowing to determine the concentrations of chemical species producing one OU. Regarding M2, this cluster of molecules has not been investigated in detail yet. Among those molecules, one of the more experienced compound is limonene (van Gemert 2011), but it is not comparable in advance to the investigated odor mixtures.

The paper describes the activities related to the survey of the case study (i), the definition of methods and devices for conducting emissions sampling (ii), and the adopted experimental approach (iii). Preliminary results are also presented (iv).

Methodology

The here presented workflow for OIA was developed by following the logical framework described on Fig. 1. Once the research questions and the goals had been detailed, the methodology was defined by considering the state of the art about

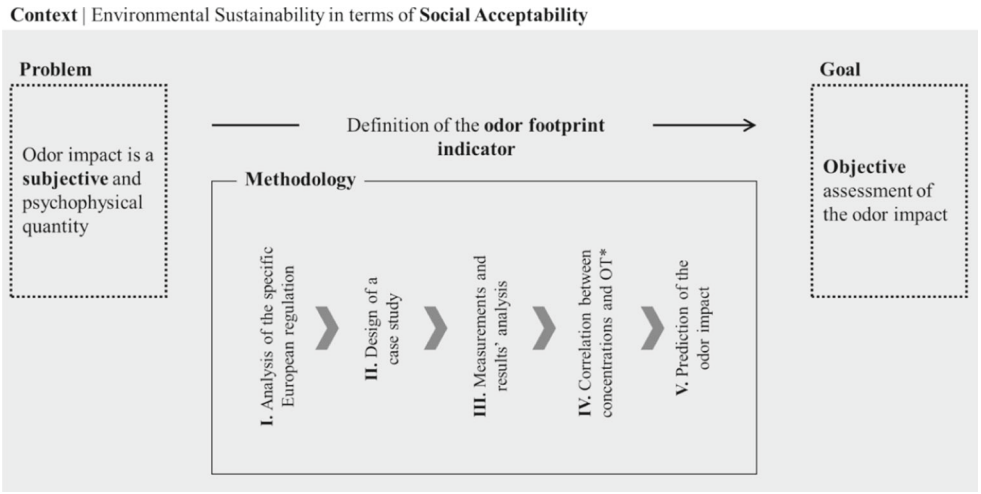

Fig. 1 Logical framework of the research. *OT stands for Odor Thresholds

olfactometry and odor assessment. In particular, it was chosen a case study in which the measurement could have been conducted in compliance with the current European standards. The study area is located in Central Italy (Fig. 2) and it is a facility storing organic materials. The company produces a significant odor impact on a mainly natural territory, as demonstrated by the continuous complaints of people who live in the adjacent areas. Prior to the space-time scheduling of the sampling campaign, a state of the arts was carried out in order to disclose the type of molecules to be possibly detected: this stage of the workflow was necessary due to the multiplicity of employable methods of detection and their dependency on chemical structure of the odorous molecules.

Since the study concerning the odor emissions was extended to the entire area of the factory production, a complete survey of the site was carried out. The odor sources were properly identified and classified according to the regional recommended regulations (Linea Guida Regione Lombardia 2012).

Each odor source requires a proper sampling device that was suggested by the specific protocol. In fact, the sampling devices are commonly classified depending on the nature of the emission source in which they are employed. In general, the totality of the odor sources can be grouped in three different clusters:

- Active areal sources (e.g., bio-filters through which a flow of forced air is conveyed, rich in pollutants to be collected)
- Passive areal sources (e.g., piles of material involved in biochemical processes and that are not subjected to the forced circulation of the air flow)
- Conveyed sources (e.g., ducts conveying the flow rates of treated effluents)

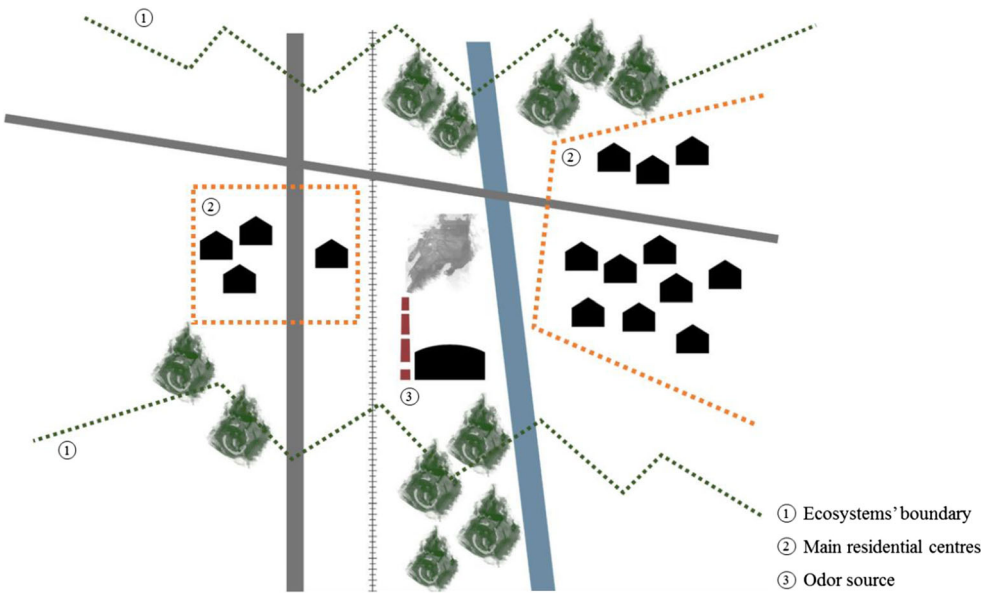

Fig. 2 Schematic view of the study case. The odor source is located in a semi-natural context, between two residential centers

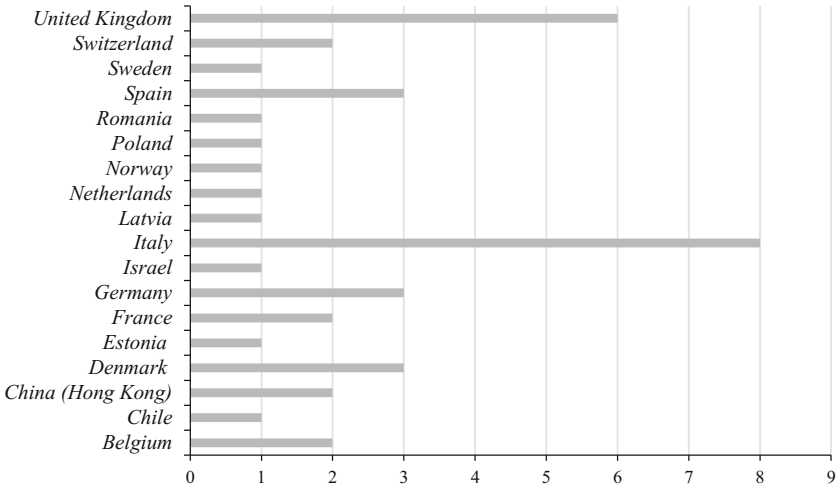

Fig. 3 Geographical distribution of the participants

In the chosen case study, up to 12 passive areal sources were identified.

Once the grid had been defined, a complete space-time monitoring program was planned, and the low-speed wind tunnel (LSWT) (European Commission 2004) was chosen as sampler.

The first sampling campaign was carried out in a low intensity period of activities for the distillery; this allowed to define a proper base-line for odor emissions if compared with high intensity period of activities. Three gaseous mixtures were collected in three different odor sources of the facility:

- *Odor source n. 1*, a pile of olive husk
- *Odor source n. 2*, a pile of marcs
- *Odor source n. 3*, a purification tank for the water treatment

Three coupled odorous mixtures were collected from the three different odor sources: (i) sample 1 (S1) from odor source n. 1; (ii) sample 2 (S2) from odor source n. 2, and (iii) sample 3 (S3) from odor source n. 3.

The three collected mixtures were firstly analyzed by MS/GC technique in order to verify the lack of health-related problems for panel members then submitted to dynamic olfactometry tests, which were conducted at CIRIAF-CRB research center, to estimate the OT.

Inter-laboratory comparison

To assess the level of reliability of the results, the olfactometry lab in Perugia took part on inter-laboratory comparison (ILC), a proficiency test during which different clusters of panelists from different laboratories analyze the same sequence of samples and then compare the results (Maxeiner and Manneck 2004).

According to the protocol provided by Olfasense GmbH, the task for the participants is to set the value of the dilution factor at the panel threshold $\overline{z}_{ite,pan}$, and to specify the value of the odor concentrations in compliance with EN 13725:2004 (European Commission 2004). The panel threshold is computed as the geometric mean of the individual threshold estimations $\overline{z}_{ite}$ following the retrospective screening and it is without units. As shown on Fig. 3, up to 40 laboratories from 18 different countries took part to the ILC, which was organized in five stages: samples' preparation (a), samples' storage (b), odor threshold estimation (c), results' analysis (d), and final submission (e). During the first phase, the 10 samples, which are 1-butanol gases with different concentrations (Table 1), are transferred from cans into sampling bags. The transferring was conducted through a control valve which permits to regulate the flow from the can to the bag. The pressurized gas cans supply about 12 l. The valve has been flushed with fresh air before opening a new can. Then, the nalophan bags are

Table 1 Analytical concentration and homogeneity test of the used testing gases

Content	Concentration	Measurement uncertainty* ($k = 1$)	Measurement uncertainty* ($k = 2$)	Analyzed by
1-butanol				
Carrier gas: *nitrogen*	22.5 ppm (μmol/mol)	±1.4%	±2.7%	Westfalen AG, D
1-butanol				
Carrier gas: *nitrogen*	56.9 ppm (μmol/mol)	±1.2%	±2.5%	Westfalen AG, D

*The maximum measurement uncertainty for 1-butanol according EN 13725:2003 is 5.0%

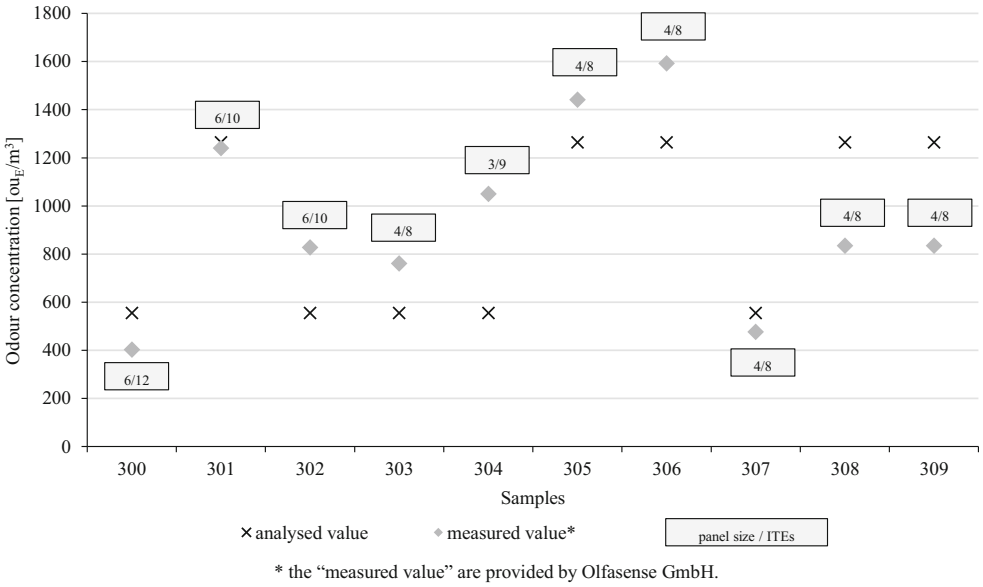

Fig. 4 Comparison of the measured concentration values with the analyzed ones. *The "measured value" is provided by Olfasense GmbH

stored (b) at room temperature (18–25 °C). Once the measurements are performed (c), the results are recorded on the specific form provided by Olfasense GmbH (d). Finally, the documents are submitted to an external coding institute before being compared (e) for ensuring the data anonymity for both participants and for the organizers Olfasense GmbH.

Among the participants, only 50% fulfills the requirements of precision under repeatability conditions (r) and accuracy (A_{od}) according to EN 13725. Regarding the others, about 33% satisfied at least one quality parameter (r or A_{od}), while the remaining are not able to achieve an adequate performance level at all. In conclusion, the ILC experience has represented for CIRIAF Olfactometry Lab a significant opportunity for comparing its activities and measurement protocols with the ones followed by other European and non-European laboratories. In particular, the evaluation of the results led to the enhancement of the procedures of both panelists' selection and definition of dilution steps. In fact, up to three different groups of panelists were involved, and different dilution steps were employed during the ILC tests, permitting to assess how they could affect the measured concentrations. The ILC test's results, which are reported on Fig. 4, show that the second group of panelists—involved for measuring the samples' series 303–306—is more sensitive than the others—involved for measuring the samples' series 300–302 and 307–309—and used to recognize the 1-butanol at lower concentrations if compared to the results achieved by 50% of participants which fill both the requirements. In addition to that, the higher dilution steps coupled with a minor number of panelists seem to be the reason that led to the most significant errors (304, 308, 309), confirming the conclusions presented by Hove et al. (2017). All of those observations regarding panelists' selection and measurement's protocol were taken into account during the analysis of the case study in order to improve the accuracy and the repeatability.

Table 2 Chemical composition of the odor mixtures S1, S2, and S3, summarized by classes of compounds

	Concentration [mg/m^3]		
Classes of compounds	S1	S2	S3
Halogen derivatives	0.522	0.409	0.382
Nitrogen compounds	–	–	0.017
Saturated hydrocarbons	0.009	0.018	0.015
Unsaturated hydrocarbons	–	–	0.002
Aromatics hydrocarbons	0.007	0.006	0.019
Acids	0.060	0.057	0.042
Alcohols	0.006	0.004	0.014
Aldehydes	0.086	0.033	0.157
Ketones	0.011	0.007	0.020
Esters	–	0.005	0.006
Other oxygen compounds	0.002	0.001	0.006
Thiols	–	–	–
Thioesters	–	–	0.008
Total	0.703	0.540	0.688

Preliminary results and discussion

The first analysis regarded the chemical characterization, and it was performed by using mass spectrometry-gas chromatography (MS-GC) techniques. This method is largely accepted by the scientific community (Zhao et al. 2018; Huang et al. 2018) and allows to determine both the kind of substances and the corresponding concentrations in S1, S2, and S3. Up to 63 substances were detected with remarkable differences of

Table 3 Odor concentration of the S1, S2, and S3 mixtures, measured by panel, according to UNI EN 13725:2004

	Units	S1	S2	S3
Odor concentration	OU_E/m^3	47	80	110

concentration among the three samples, and they were classified in 13 chemical classes of compounds. Table 2 shows the results of the detected chemical species and their measured concentrations. The *halogen derivatives* turned out to be the most present specie among the ones detected in all of the three samples, while *thiols* were not found at all. Furthermore, the S3 is the only sample characterized by the presence of *nitrogen compounds*, *unsatured hydrocarbons*, and *thioesters*, although in low quantities—lower than 2.5%.

Successively, the samples have been analyzed in order to evaluate the odor concentration according to EN 13725:2004. Each sample was submitted to a panel of qualified human receptors using dynamic dilution-based olfactometry methodology (Orzi et al. 2018; Guffanti et al. 2018). Table 3 summarizes the resulting odor concentration of the mixtures and is expressed in *European odor units per cubic meter* (OU_E/m^3), which corresponds to the number of dilutions necessary to no longer detect any odor for the 50% of the human panelists.

As mentioned above, the purpose of the work is to investigate the correlation between measured concentrations of the substances and odor concentration, mainly considering the synergy effects which may occur between odorous substances. The correlation between sensory analyses and chemical evaluation represents a topic largely discussed among the research groups focused on olfactometry (Bonte et al. 2017). It may be not linear and probably a more complex function is required for describing these mg/m^3 to OU_E/m^3 dependences.

Data processing was conducted as follows. The concentrations corresponding to the odor thresholds values in air for each substance were collected from other studies in literature (van Gemert 2011). The state of the arts highlights the impossibility of defining a threshold value for 4 of the 63 substances identified. Then, the odor concentration value of the extra-threshold chemical species in the three mixtures was calculated, following a logarithmic trend (1):

$$OI_i = \frac{1}{\log(F_s)} \cdot \log(X_i) + A_i \quad (1)$$

F_s is the dilution factor used in the panel analysis
X_i is the concentration of the i-th substance
A_i is a constant term (characteristic for each substance)
$OI_{,i}$ is the odor intensity at X_i concentration (for the i-th substance)

The term A can be determined for each substance from its threshold value ($X_{0,i}$), which corresponds by the definition to 1 OU_E/m^3 and it is calculated as follows:

$$A_i = 1 - \frac{\log(X_{0,i})}{\log(F_s)} \quad (2)$$

Knowing the correlation law, in which the dilution factor (F_s) assumes the same value employed in the dynamic olfactometry test for consistency, the odor intensity higher than the odor threshold was determined.

As an example, the correlation mg/m^3 to OU_E/m^3 is reported for tri-methylamine on Fig. 5.

Some similar calculations allowed to determine the odor intensity (extra-threshold values) of the other 12 detected class of compounds (Fig. 6). The results showed

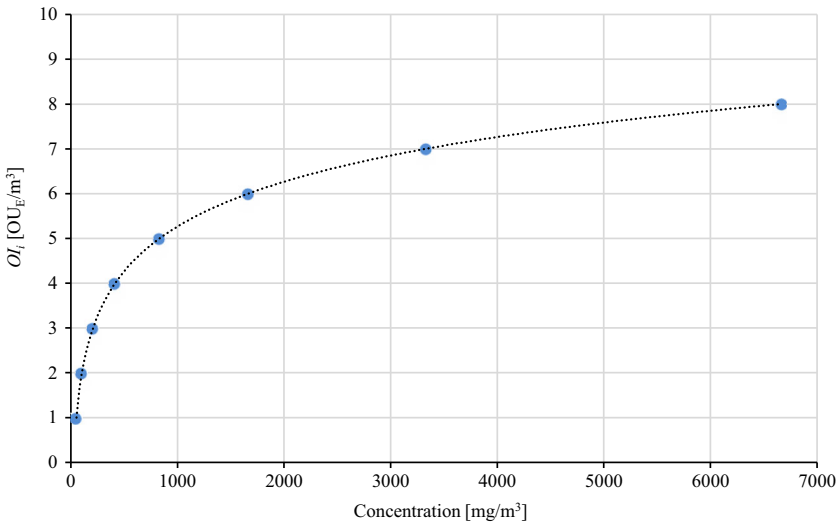

Fig. 5 Correlation odor intensity—substance concentration for tri-methylamine

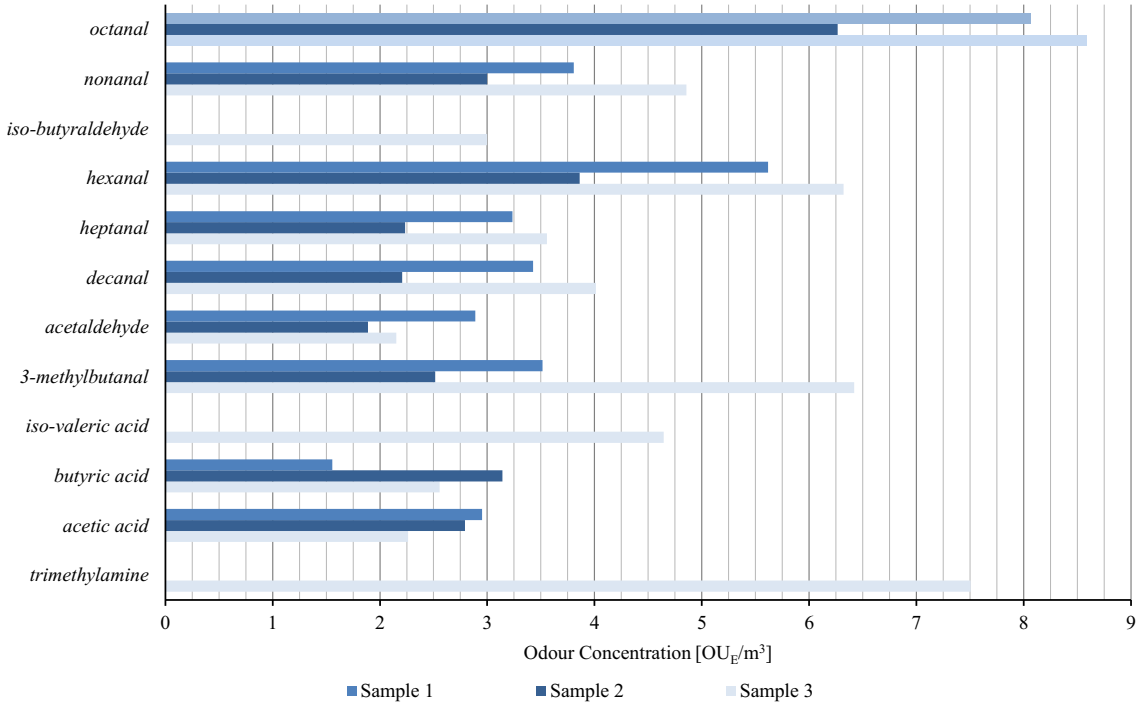

Fig. 6 Calculated odor intensities of the 12 extra-threshold chemical species in S1, S2, and S3

that some substances have less influence in the odor impact if compared to others: the measured *aldehydes* in S1 and S3 have higher concentrations than in S2, which globally, however, has an intermediate odor intensity. It can also be observed that *tri-methylamine*, which is one of the substances with the lowest odor threshold (hence easily perceived), is detected only in S3 (the most odorous), with an odor intensity of about 7.5 OU_E/m^3.

Near future works

The calculated values of odor concentration for each sampled mixture would be used in order to find the mathematical correlation between the estimated and the panel-measured values of odor intensity. The qualitative form of the correlation could be expressed by the following equation (3):

$$F(OU_{calc})_{Si} = \left[OU_{panel}\right]_{Si} \quad (3)$$

F	is the mathematical operator allowing to pass from the model to the panel measurement
OU_{calc}	is the calculated value of the odor intensity
OU_{panel}	is the measured value of the odor intensity according to UNI EN 13725:2004
Si	is the i-th sampled odor mixture

The definition of the correlation, once further validated by means of in-field measurement campaigns, should permit to predict the odor intensity for specific classes of compounds after knowing their concentrations.

Conclusions

The study presents the preliminary activities carried out by CRB Research Centre on Biomass, in defining an innovative methodology for relating the odor intensity to the concentrations regarding particular classes of chemical compounds, produced from agro-industrial activities. The project consists in the assessment of a case study involving a local factory, where a sampling campaign was conducted using the LSWT device. Then, the collected mixtures were analyzed by both MS/GC and panel techniques.

The initial phase of the measurement campaign was carried out in a low intensity period of activities for the factory leading to the definition of a background odor for the facility. They were identified as 63 chemical species, belonging to 13 different classes of compounds.

The initial data processing was also presented, particularly focusing on the calculation methodology for the extra-threshold odor intensity of the detected species. To this aim, a Weber-Fechner-type law was chosen. Results showed that the synergy among chemical species in producing odor impact is the major problem to be overcame in modeling these phenomena. In addition to that, the environmental impact of the odor sources should be further evaluated by means of dispersion models, accounting the specific meteorological conditions of the case study.

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