

Olfactometric testing as a method for assessing odour nuisance of biogas plants processing municipal waste

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Abstract: Biogas plants processing municipal waste are very important investments from the point of view of waste management and also the sustainable development of urban infrastructures. They may also have a potentially negative impact on the environment in the form of odour emission. Olfactometry is the main method for odour impact assessment. Field olfactometry allows for performing a wide range of tests, the results of which are practically instantaneous. The purpose of this work is to provide a tool for assessing the odour impacts of municipal management facilities, including biogas plants processing municipal waste and evaluating the correctness of processes carried out in these plants, namely the method of field olfactometry. In order to compare obtained olfactometric results with the concentration of chemical compounds, chromatographic tests were also carried out using the Photovac Voyager portable chromatograph (hydrogen sulphide – H_2S and dimethyl sulphide – $(CH_3)_2S$). The results of the odour concentration tests are in line with the results of odorant concentration tests and indicate that c_{od} is strongly related to the concentration of hydrogen sulphide. Thanks to this method, it is possible to find a relationship between odour nuisance, technological processes used in the plant and the type of treated waste.

Introduction

Municipal solid waste treatment plants are potential sources of odour nuisance (Di Nardo et al. 2019, Pawnuik and Sówka 2019). This nuisance may be related to the emissions of individual compounds such as volatile sulphur compounds, volatile nitrogen compounds and volatile organic and inorganic compounds (Young and Parker 1983, Chianese et al. 2015). They are dispersed in the air as gases or as aerosols that spread in the atmosphere (Fabbri et al. 2014). Many of the studies conducted so far have been related to the odour impact of landfills. Research carried out on the active surface of municipal waste landfills by Ding et al. (2012), Fang et al. (2012), Kim et al. (2005) and Li et al. (2005) has shown that dimethyl sulphide (DMS), as an odorant, can be a cause of odour nuisance problems in landfills.

Moreover, odour emissions can be characterized by high variability over time, depending on the meteorological conditions (Drew et al. 2007). The continuous release of low-concentration unpleasant odours can be equally strenuous, in terms of a nuisance to the population, as a periodic release of unpleasant odours at high concentrations because the effect potentially accumulates over a prolonged period of exposure (Daskalopoulos et al. 1997, Meišutovič-Akhtarjeva and Marčiulaitienė 2017).

The main objectives of waste treatment plants should be to produce as many fractions as possible for further use and to

dispose of waste that cannot be used otherwise. The processes, both recovery and disposal, are most often carried out in mechanical and biological installations of waste processing (Połomka and Jędrzak 2019). Disposal is carried out with the participation of micro-organisms under aerobic conditions (aerobic stabilization) or anaerobic (anaerobic stabilization). Anaerobic processes using methane fermentation, in addition to the benefits associated with the disposal of waste, are also characterized by energy benefits (Zentner 2019). Biogas produced in fermentation chambers is utilized for the generation of electricity and/or heat thanks to the biogas plant. At biogas plants processing municipal waste in Poland, the batch material for the fermentation process is the most common fraction of mechanically produced waste from the mixed waste stream. Only one of them is given a fraction of biodegradable waste from separate collection to the fermentation chambers (Wiśniewska et al. 2019).

Biogas plants processing municipal waste also have a negative impact because they are the source of the emissions of odours. These compounds (Wiśniewska 2018) originate from both the mechanical part, including the preparation of the batch for the process fermentation, as well as from the biological part including the dewatering of the digester and its aerobic stabilization. The fermentation process is carried out without air access and is therefore encapsulated and does not cause the emission of odorants (Rosik-Dulewska 2012).

There are a few methods for quantitative analysis of both odours (sensory methods) and the individual compounds that produce a negative olfactory impression (analytical and sensor methods) (Wiśniewska 2020). Analytical methods include, among others, gas chromatography (GC) and gas chromatography coupled with mass spectrometry (GC-MS). These methods make it possible to separate individual odorants in the gas mixture, identify them and quantify them. None of these methods, when used alone, will give full information on the odours emitted (Grzelka et al. 2018; Wiśniewska 2020).

An example of using the sensing method is, among others, electronic nose (e-nose) and gas detectors (Capelli et al. 2014; Wiśniewska 2020). The working of the electronic nose is based on a calibration model. To develop such models, signals from electronic nose sensors and odour intensities expressed in verbal scale are used (Szulczyński & Gębicki 2019). The electronic nose can be used for “in-situ” tests. An unquestionable disadvantage of the device is its sensitivity to changes of temperature and relative air humidity (Capelli et al. 2014; Nakamoto and Sumitomo 2003).

Olfactometry is a quantitative technique for determining the odour concentration range (Munoz et al. 2010, Szyłak-Szydłowski 2014). The olfactometric tests are used to determine the odour concentration of the process gases (Badach et al. 2018, Maurer et al. 2018). The olfactometric methods can be divided into static and dynamic (indirect – determination under laboratory conditions; and direct – determination in the field). The dynamic olfactometry method is based on air analysis at the source. Field conditions (“in-situ” designation) or laboratory conditions (“ex-situ” designation) may be used. In the latter case, it is necessary to take a sample of gas into a bag made of suitable material and analyze it under laboratory conditions. During storage and transport, the gas sample is exposed to adsorption and condensation processes (Munoz et al. 2010). Measurement methods being used to analyze odours and to assess odour annoyance with their advantages and disadvantages are fully described in the paper (Conti et al. 2020).

Dynamic olfactometry, which is also field olfactometry, is the most commonly used method and provides information – statistically defined – on the sensitivity and size of the odour samples tested by means of a controlled dilution by the olfactometer. This method does not identify the chemical compounds in the mixture of the test gases, but leads to the determination of their odour threshold, which corresponds to the number of dilutions necessary to reduce the perception of odour. This procedure has been largely examined by several studies in the literature evaluating the odour threshold of specific odour-causing chemicals (odorants), such as ammonia, hydrogen sulphide and dimethyl sulphide (Coccia et al. 2018, Greenman et al. 2004, McGinley et al. 2004; Wiśniewska et al. 2019).

The aim of the work is to check whether the field olfactometry method can be used to carry out the recognition in waste management plants, which include biogas plants processing municipal waste, as well as to determine the sources of odour emissions and the degree of their intensity. The use of dynamic field olfactometry allows for many more measurements to be made than in the case of determining odour concentration in the laboratory (Benzo et al. 2012, Kolasińska et al. 2017, Newby and McGinley 2004).

The article presents the results of two series of odour tests (odour concentration) and one series of chemical tests (hydrogen sulphide and dimethyl sulphide) carried out in six municipal mechanical and biological waste treatment plants, using a methane fermentation process in the biological part and having a biogas installation. The purpose of this work is to present a tool for assessing the odour impacts of municipal facilities, including biogas plants processing municipal waste, and assessing the correctness of processes carried out in these plants using the field olfactometry method. The purpose of the application of chemical tests consisting of hydrogen sulphide and dimethyl sulphide concentrations determination by means of a portable gas chromatograph was to confirm the reliability of odour tests. Those pollutants characterize biogas plants and accompany anaerobic decomposition of waste.

Materials and methods

The tests involve chromatography and olfactometric measurements and the odour assessment method of sensory evaluation on a six-stage scale, where “0” means no smell and “5” very strong fragrance (Wiśniewska et al. 2019), at six biogas plants processing municipal waste in two measurement series. The Nasal Ranger® field olfactometer (St. Croix Sensors Inc.; Stillwater, Minnesota, USA) was used to determine the odour concentration at each measurement point. The device enables gradual dilution of the polluted air with purified air in a known ratio by means of two interchangeable control valves. The flow rate of the analyzed gases was 20 dm³/min. The first valve allows for an equal dilution of D/T: 2, 4, 7, 15, 30 and 60, and the other: 60, 100, 200, 300, 500. The accuracy and reproducibility of the dilutions using the first control valve is ±10% and the second one ±5%. The panelist, performing olfactometric determinations prior to each of the measurement series was subjected to Triangle Test according to ISO 4120:2004, which consists in indicating among the presented sticks the one soaked in a fragrance, n-butanol (International Organization for Standardization 2004). The sensory evaluation was performed in situ. Based on the results obtained in two repetitions, the odour concentration was calculated based on the following formulae in accordance with the European norm PN-EN 13725:2007 Polish Committee for Standardization 2007):

$$Z_{YES} = (D/T)_{YES} + 1,$$

where:

Z_{YES} means the dilution ratio (–) at which the odour was perceptible;

$(D/T)_{YES}$ means the dilution ratio (–), corresponding to the moment when the odour was perceptible for the first time,

$$Z_{NO} = (D/T)_{NO} + 1,$$

where:

Z_{NO} means the dilution ratio (–) at which the odour was imperceptible;

$(D/T)_{NO}$ means the dilution ratio (–) corresponding to the moment when the odour was imperceptible just before the dilution $(D/T)_{YES}$,

$$Z_{ITE} = \sqrt{Z_{YES} \cdot Z_{NO}},$$

where:

Z_{ITE} means the assessment of the individual threshold, expressed as dilution ratio (-).

Values of the odour concentrations were calculated based on a geometric mean of the set of all individual estimations (Z_{ITE}) for a given measurement point:

$$c_{od} = \sqrt[n]{\sum_{i=1}^n Z_{ITE,i}},$$

where:

n – means the number of all estimates.

Chromatography tests involving determination of hydrogen sulphide and dimethyl sulphide concentrations were carried out using the Photovac Voyager field-portable chromatograph (Perkin Elmer Inc., United Kingdom). The chromatograph is equipped with a photo-ionization detector (PID). The carrier gas used for determination was high-purity nitrogen (N_2). The chromatograph uses the technique of precolumn backflushing to enable fast analysis times. It has separate columns for the determination of different compounds. Two of them were used: one for DMS determination and second for H_2S determination. Samples of process gases were led directly to the device using a built-in pump. In Table 1 the characteristics of the columns used for testing is presented. The obtained results are the arithmetic mean of three parallel measurements. In addition, standard deviations were calculated.

The Odour Activity Value (OAV), also called sensory stimulation strength of single-compound is defined as the ratio of a specific odorant concentration (C_i) to its odour threshold (OT_i) value (Ravina et al. 2020, Yang et al. 2015). For hydrogen sulphide and DMS OTs equal respectively 0.0081 ppm and 0.001 ppm (Wiśniewska et al. 2019). It was calculated for the results, according to the formula:

$$OAV = \frac{C_i}{OT_i}$$

Each measurement series shows the relationship between the odour intensity and odour concentration determined based on the D/T parameter read from the field olfactometer Nasal Ranger® (Di Nardo et al. 2019, Szulczyński et al. 2018)

at designated measurement points constituting sources of odour nuisance. In the first measurement series, the impact of hydrogen sulphide and dimethyl sulphide concentrations on odour concentration is also presented. Evaluations of the obtained results were carried out concerning the technological processes in the analyzed plants.

Results and discussion

From July 2018 to May 2019, two measurement series were conducted in six waste treatment plants equipped with biogas installations in Poland with locations shown in Figure 1.

Local visits to the plants (Wiśniewska et al. 2018) made it possible to identify and characterize odour sources in individual plants. Table 2 and Figure 2 show the sources of odours in biogas plants, taking into account the method and location of technological processes.

The results of measurements obtained in the biogas plants under study are shown in Figures 3–8. Standard deviations were indicated for odorant concentrations. In any biogas plant, they exceeded 10% of the average values. Additionally, OAV was included among the measurement results.

When analyzing Figure 3, it can be observed that, at most measurement points, the odour intensity (i) changes with the odour concentration (c_{od}). In most odour sources, a higher odour intensity was accompanied by a higher odour concentration. During the measurement series carried out at the analyzed plant, the various plant buildings (A – waste storage plant, B – mechanical part plant, C – fermentation preparation plant and D – digestate dewatering plant and the digestate oxygen stabilization plant – the first stage) were open, which resulted in dispersion of odours during the process carried out. Dewatered digestate is directed to an uncovered box in the analyzed plant, which also may cause an increased odour emission. According to the results presented in the graph, the sources of the biggest odour emission are the fermentation preparation plant and the digestate dewatering and aerobic stabilizing plant (the first stage). These measurement points (C and D) in the first series of measurement were also characterized by the biggest concentration of odorants, especially dimethyl sulphide. The odour concentration on the biofilter surface (G) at the level of 6 ou/m³ (series 1) and 4 ou/m³ (series 2) and the high concentration of hydrogen sulphide (series 1) may be due to the structure of the deodorization installation of process gases (the open biofilter), which is not equipped with a chemical scrubber (before biofiltration), or improper and insufficient care of the filter bed (surface 400 m²).

Table 1. The field portable gas chromatograph characteristic

Parameter/Column	1	2
Compound	(CH ₃) ₂ S	H ₂ S
Detector	PID (10.6 eV)	PID (10.6 eV)
LOD [ppm]	0.001	0.001
LOQ [ppm]	0.005	0.005
Retention time [s]	103	32
Analysis time	660	660
Dimensions	20 m × 0.32 mm × 1 μm	25 m × 0.32 mm × 12 μm

At the Tychy plant, as at the Jarocin plant, the source of the biggest odour emission is the fermentation preparation plant (C) and digestate dewatering and stabilizing plant (D). The digestate dewatering system in this plant is the same as that used in Jarocin. The results of the odour concentration tests are in line with the results of odorant concentration tests and indicate that c_{od} is most strongly related to the concentration of hydrogen sulphide. The biggest concentration of hydrogen sulphide during the first measurement series was

also observed in fermentation preparation plant (C). The second highest result of hydrogen sulphide concentration was recorded at the digestate dewatering plant (D). In the case of this biogas plant, smaller differences were observed between individual measurement series compared to the installation in Jarocin. However, where the differences were clearest, similar relationships were noted as in Jarocin – the increase in the odour intensity accompanies growth in the odour concentration.

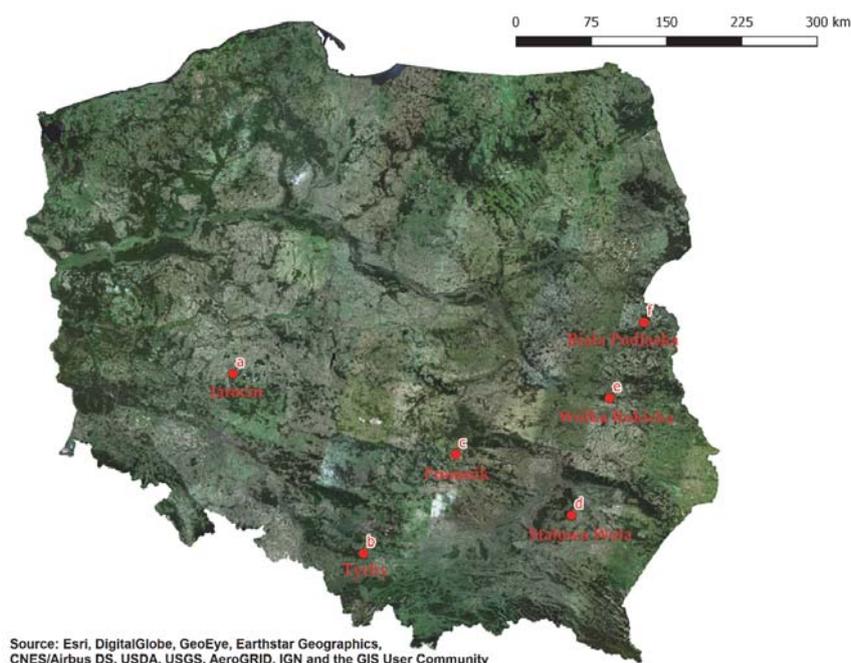


Fig. 1. Location of biogas plants processing municipal waste in Poland

Table 2. Odour sources at the examined biogas plants (Wiśniewska et al. 2019)

Mark of the odour source	Biogas plants location					
	Jarocin	Tychy	Promnik	Stalowa Wola	Wólka Rokicka	Biała Podlaska
	Dates of measurement series					
	2018-07-21 2019-02-27	2018-09-27 2019-03-05	2019-04-12 2019-05-15	2018-08-30 2019-02-19	2018-08-28 2019-02-19	2019-04-26 2019-05-15
Name of odour source						
A	Waste storage					
B	Mechanical part					
C	Fermentation preparation (in processing buildings)					Fermentation preparation (at technological field)
D	Digestate dewatering				–	
E	–	1° digestate oxygen stabilization			–	1° mixed waste fraction 15–80 mm oxygen stabilization
F	Oxygen stabilization	–	Oxygen stabilization			
G	Biofiltration (surface of open biofilter)					

– means that the sources do not occur at the plant (as resulting from the applied waste treatment technology or practiced technological regime) or was not tested during the measurement series.

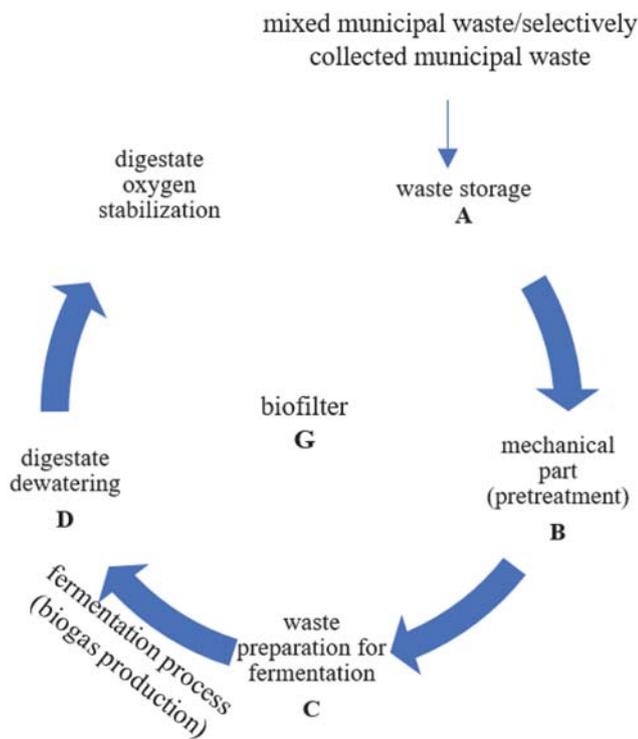


Fig. 2. General scheme of the technological line processes at the examined biogas installations with indication of odour sources

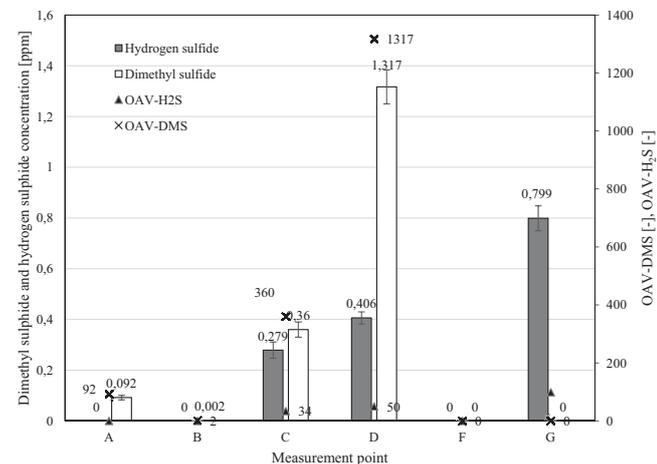
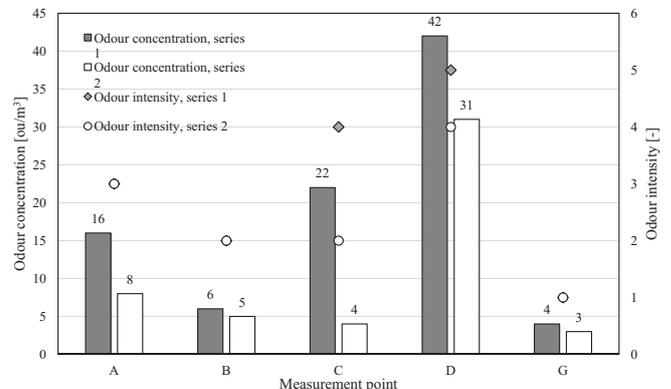
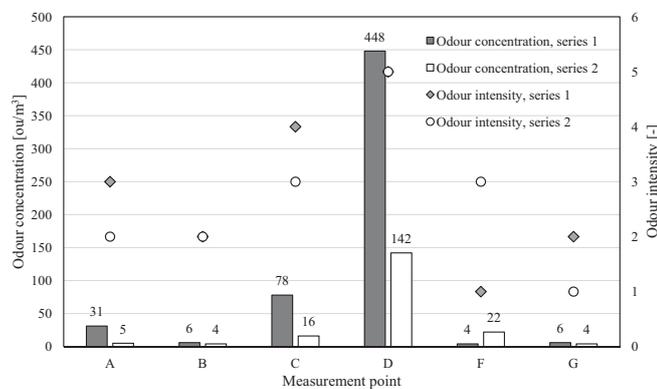


Fig. 3. Measurement results from the biogas installation in Jarocin

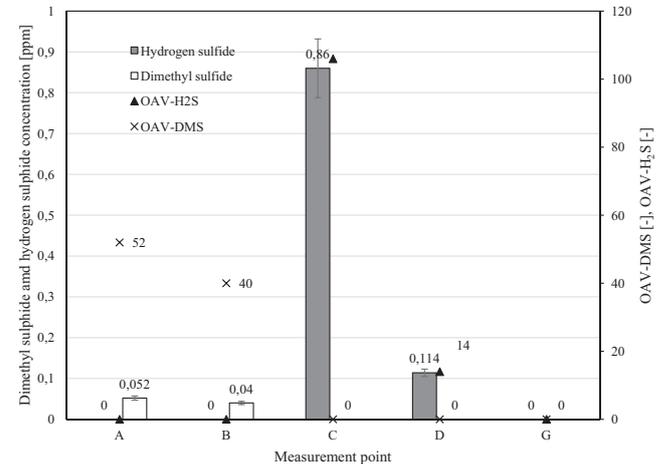


Fig. 4. Measurement results from the biogas installation in Tychy

At the biogas plant in Promnik, high odour nuisance was associated with a high concentration of both dimethyl sulphide and hydrogen sulphide. Unlike other installations, the plant in Promnik is operated with closed gates, maximizing the encapsulation of technological processes. Also, in this case the source of the biggest odour and odorant emission (both dimethyl sulphide and hydrogen sulphide) is the digestate dewatering plant – D ($c_{odl} = 42 \text{ ou/m}^3$, $\text{H}_2\text{S} = 0.267 \text{ ppm}$, $\text{DMS} = 0.997 \text{ ppm}$ – during the 1st series). The second result of high dimethyl sulphide concentration was observed at fermentation preparation plant (C), which was also accompanied by one of the higher odour concentration values (11 ou/m^3). In Promnik the second stage of digestate oxygen stabilization is carried out under a roofed shelter, while in Jarocin it takes place in an open-air prism field. Odour concentrations obtained in Promnik are at a much lower level than in Jarocin, which may indicate a higher degree of waste stabilization in earlier stages of the process, as well as lower exposure to meteorological factors.

At the plant in Stalowa Wola, as at the plants in Jarocin and Tychy, technological processes are carried out in buildings with open gates. At most odour sources, a bigger odour intensity was accompanied by a higher odour concentration. Big odour concentrations were observed on the surface of the biofilter (G), which may be a result of improper care for the filter bed (surface 550 m^2), as well as improper operation of the deodorization installation for process gases. At this measurement point, the biggest concentration of hydrogen sulphide was also observed during the first measurement series (1.373 ppm). The highest

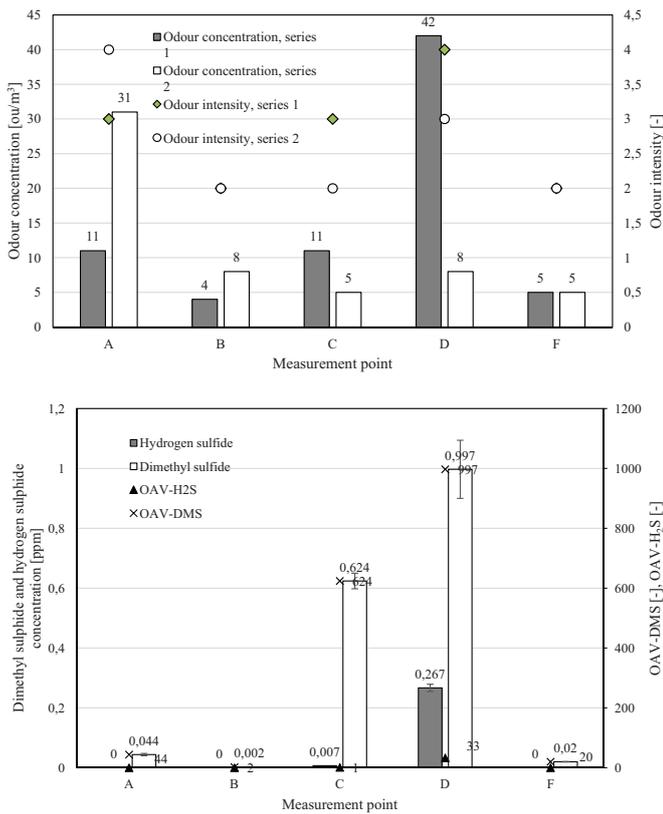


Fig. 5. Measurement results from the biogas installation in Promnik

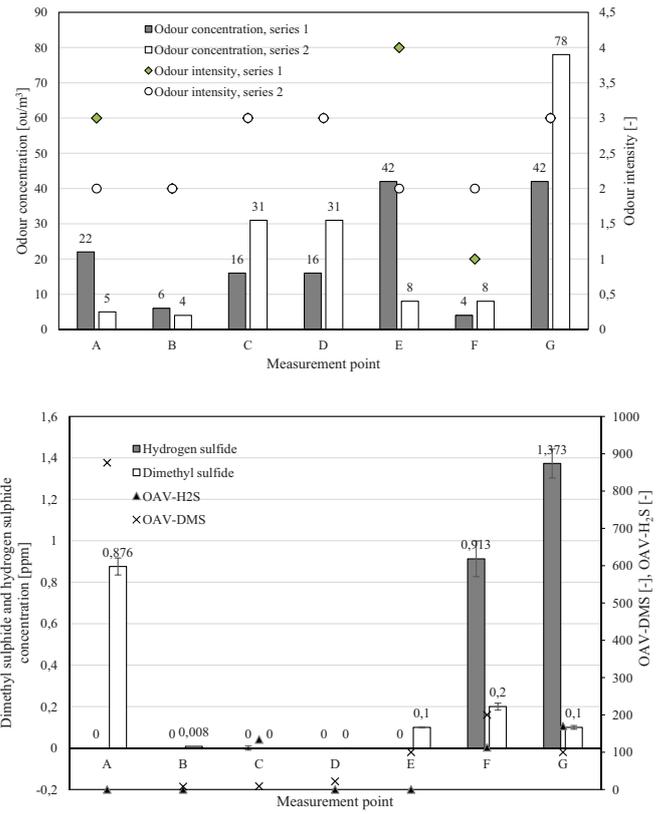


Fig. 6. Measurement results from the biogas installation in Stalowa Wola

concentration of dimethyl sulphide was in turn accompanied by a high concentration of odour in the waste storage plant (at the beginning of the technological process) – A.

Special attention should be paid at Wólka Rokicka biogas plant to the very big odour concentration accompanied by the highest concentration of dimethyl sulphide during the first series of measurements in the waste storage plant (A). At the time, the plant was filled with mixed municipal waste. Big odour concentration in this source may indicate the beginning of uncontrolled waste decomposition processes under anaerobic conditions as a result of too long storage. Moreover, the plant differs from the other installations analyzed in terms of technology. The fermentation process is not carried out in a traditional fermentation chamber, but in a tunnel, which is opened each time it is loaded and unloaded, thus increasing odour emissions. The highest concentration of hydrogen sulphide (1.905 ppm) was noted during the oxygen stabilization process (F), where the third highest cod result (16 ou/m³) was observed.

The plant in Biała Podlaska, unlike other plants, carries out the fermentation process of biodegradable waste collected selectively. This is probably one of the reasons for lower odour emissions than others during waste storage at the beginning of the process line. In addition, the digestate is not dewatered but it is mixed with green waste (structure-forming material) and stabilized in the open air (F). This process is associated with significant odour emissions. The highest odour concentration was observed there in both measurement series. This highest odour concentration during the first measurement series was accompanied by the highest concentration of both hydrogen sulphide and dimethyl sulphide. At the Biała Podlaska plant,

as at the plants in Jarocin, Tychy, Wólka Rokicka and Stalowa Wola, unit processes and operations related to mechanical processing are carried out in open buildings. However, the fermentation preparation takes place in the processing yard, which leads to lower results compared to the plants where the fermentation preparation is carried out in the plants (due to the spread of odours to neighboring areas).

The above charts also show the calculated OAVs to analyze the contribution of odorants tested. The larger the OAV, the more likely that compound would contribute to the overall odour of a complex odour mixture (Yang et al. 2015). While the OAV value of DMS is higher (in five out of six analyzed biogas plants), therefore this odorant is found as the most significant in research conducted. It can be assumed that it has a greater impact on the concentration of odour and its intensity in the analyzed biogas plants. Only at the Wólka Rokicka biogas plant the highest OAV was determined for H₂S.

On the basis of the presented results, a summary of odour nuisance parameters for particular technological operations (odour sources) used at the analyzed biogas plants together with factors influencing them was prepared – Table 3. In turn, Table 4 shows the correlation coefficients (based on correlation matrix) for odour and odorant concentrations for Biała Podlaska plant, where these coefficients are the highest in comparison with other plants. In other biogas plants, these correlations are not so clear, which means the impact of other odorants on odour concentration. The paper (Wiśniewska et al. 2020) indicates a distinct influence of VOCs and ammonia on the odour nuisance associated with municipal solid waste processing in biogas plants.

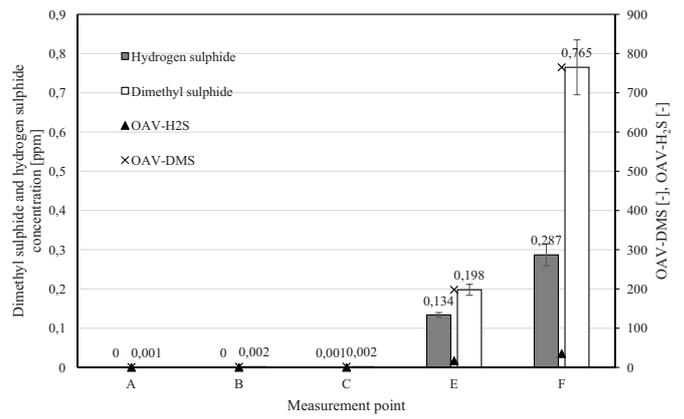
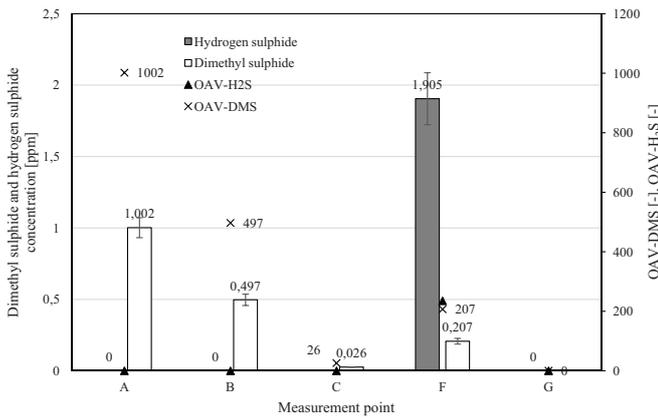
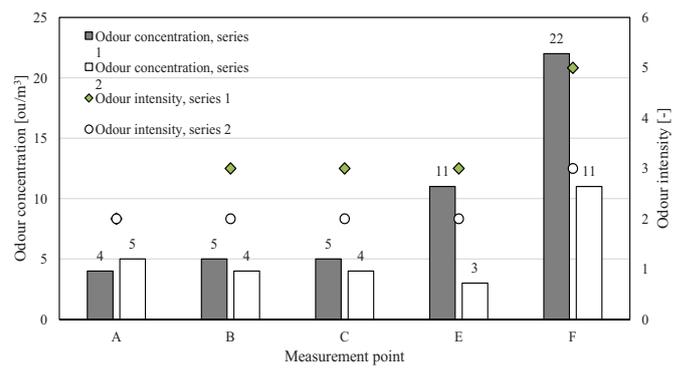
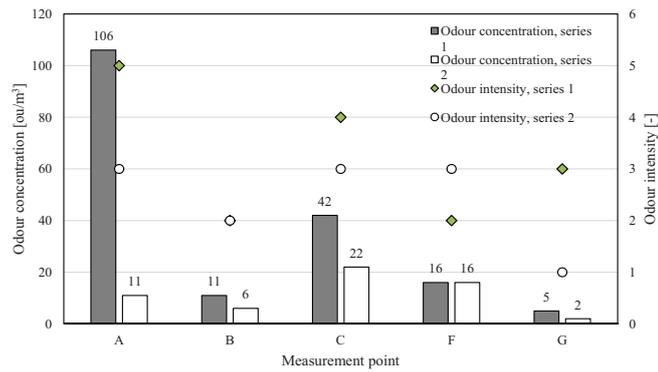


Fig. 7. Measurement results from biogas plant in Wólka Rokicka

Fig. 8. Measurement results from biogas plant in Biała Podlaska

Table 3. Summary of odour nuisance parameters for particular technological operations (odour sources) used at the analyzed biogas plants together with factors influencing them

Technological operation	Odour nuisance parameters					Odour nuisance factor
	Odour concentration [ou/m³]	Odorant concentration [ppm]		OAV [-]		
		H ₂ S	DMS	OAV-H ₂ S	OAV-DMS	
Values range						
Waste storage	4÷106	<0.001	0.001÷1.072	<0.123	1÷1072	type of treated waste: • mixed • selectively collected
Mechanical part (incl. fermentation preparation)	2÷42	<0.001÷1.092	0.001÷0.650	<0.123÷135	1÷650	type of technological halls: • open • closed
Digestate dewatering	8÷448	<0.001÷0.430	<0.001÷1.384	<0.123÷53	<1÷1384	technology of methane fermentation: • dry • semi-dry
Digestate oxygen stabilisation	4÷42	<0.001÷2.087	<0.001÷0.835	<0.123÷258	<1÷835	no evidence

Table 4. Correlation coefficients between odour concentration and the concentration of the tested odorants for biogas plant in Biała Podlaska

Odorant	C _{od}	C _{H₂S}	C _{DMS}
C _{od}	1	0.99	0.99
C _{H₂S}	0.99	1	0.97
C _{DMS}	0.99	0.97	1

Conclusions

The field olfactometer, allowing for direct measurement of odour concentrations at multiple points within a plant is not only a suitable method for assessing the odour concentration of biogas plants processing waste, but also an appropriate method of process control carried out in such biogas plants, as confirmed by the obtained test results.

The achieved results showed the relationship between odour intensity and odour concentration for processes carried out in the analyzed plants. For many odour sources, a higher odour intensity was accompanied by a higher odour concentration. This is in line with Weber-Fechner's law, according to which there is a relationship between the intensity of olfactory perception and the odour concentration, which constitutes the theoretical basis for the perception of olfactory sensations by the human sense of smell (Huang & Guo 2018).

In most odour sources at the analyzed biogas plants, odour concentration and intensity were associated with the processes used in the plant and with the type of treated waste. Noteworthy is the varied concentration of odours at the stage of waste storage, which is related to both the type of waste delivered to the plant (mixed or collected selectively) and the time of storage. Less odour nuisance accompanies the processing of municipal waste collected selectively, at least at the storage stage – due to the lower nuisance of the dry fractions (raw materials) and shorter storage time (increased collection frequency) in the case of selectively collected bio-fractions.

Odour emission depends on technological processes, including the type of fermentation technology, which can be implemented as dry (garage) – carried out in special tunnels; or semi-dry – carried out in traditional fermentation chambers. In most analyzed biogas plants where digestate dewatering is carried out (in semi-dry fermentation technology), this element of the processing line was characterized by the greatest odour nuisance (measured both by odour concentration and intensity). This technological operation is accompanied by emissions of both dimethyl sulphide and hydrogen sulphide. Among the odorous technological operations, the input preparation for the fermentation process (especially in the case of operations conducted in open halls) and the digestate oxygen stabilization should also be mentioned.

OAV analysis shows that among the odorants tested (hydrogen sulfide and dimethyl sulphide), it is the presence of DMS that has the greatest impact on odour mixture and contributes to its overall odour. At the same time the analysis of correlation between odour and odorant concentrations shows that this correlation occurs only at one biogas plant, in Biała Podlaska, what indicates that in the remaining analyzed plants odour is caused more by chemical compounds other than those measured.

The odour concentration and intensity also depend on the location of individual technological processes – in the open air, under a shelter or in a hall – closed or open. These variations cause differences in the spread of odours and in the exposure to meteorological factors.

Not only technological processes related to waste treatment, but also solutions in the field of deodorization (type of deodorization installation and method of its operation) affect odour emission. Installations for process gas purification may prove to be an important source of odour nuisance.

Field olfactometry enables the control of individual processes and unit operations as well as the control of odour problems. It is a simple method which gives immediate, practically instantaneous results. In addition, it is a reliable method, which is confirmed by the obtained test results. The highest odour concentrations accompanied the highest odorant concentrations (dimethyl sulphide or hydrogen sulphide or both) at many measuring points, such as waste storage plants (at the beginning of the process line), preparation fermentation plants, digestate dewatering plants and digestate oxygen stabilization.

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