Application of a Gas Sensor Array to Effectiveness Monitoring of Air Contaminated with Toluene Vapors Absorption Process

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ABSTRACT

This article demonstrates the application of a gas sensor array to monitor the effectiveness of the absorption process of air stream purification from odorous compounds (toluene vapors). A self-constructed matrix consisting of five commercially available gas sensors was used. Multiple linear regression (MLR) was selected as the statistical technique used to calibrate the matrice. Gas chromatography coupled with a flame ionization detector (GC-FID) was used as a reference analytical technique, which enabled to obtain reliable quantitative determinations of toluene concentration in the samples. A commercially available absorption liquid dedicated to non-polar compounds was used as an absorbent. The process was carried out in two identical systems: in first, pure toluene was absorbed and in the second, toluene vapor contaminated with acetone. This approach allowed verifying the selectivity of the prepared MLR calibration model for process control in the case of the presence of more or less expected pollutants in the treated gas. The results obtained with the gas sensor array were related to the reference technique and they confirm the usefulness and advisability of using these devices to monitor the absorption processes as a cheaper and more time-efficient alternative to chromatographic methods. The root mean square error (RMSE) in absorptivity determination between the results received with the analytical and sensor techniques was 0.019 and 0.041 when treating pure toluene vapors and its vapors with acetone, respectively. Compared to instrumental techniques, sensor matrices are technologically less complex, useful for laboratory purposes, as well as showing application potential for field studies. However, it is necessary to develop more sensitive and selective chemical gas sensor arrays and better master advanced data processing and identification techniques.

Keywords: gas sensors, sensor array, absorption, deodorization, process control.

INTRODUCTION

Emission of undesirable odorous substances, in addition to their undeniable impact on life and health of people and other living organisms, causes deterioration of living comfort, degradation of agricultural land and reduction of attractiveness of natural areas [Szynkowska et al. 2009]. Moreover, exposure to odor nuisance compounds can cause depression, anger, fatigue or overall mood disturbance [Wysocka et al. 2019].

The odor nuisance of a given gas stream is caused by the presence of volatile substances, even in very low concentrations due to their low olfactory thresholds. Fragrance (smell) is a mixture of different chemicals, but only a small fraction of them is responsible for a specific odor. For example, less than 300 different compounds could contribute to nuisance odor from swine production facilities [Pearce et al. 2010, Phung 2006]. Volatile odor nuisance substances can be divided into two groups of compounds. The first of these is volatile organic compounds (VOCs) which in the simplest terms are defined as all carbon-containing compounds except carbon monoxide, carbonic acid, carbon dioxide, metal carbonates, carbides and ammonium carbonate. A more precise definition of VOCs was proposed by the United States Environmental Protection Agency (US EPA), defining them as organic compounds with a vapor pressure exceeding 0.1 millimeter of mercury (mm Hg) under normal

conditions [Revah et al. 2005]. The other one is volatile inorganic compounds (VICs), which are simply inorganic gases of all kinds, which can include hydrogen sulfide, sulfur dioxide, carbon disulfide or ammonia.

The most common sources of odor emissions reported in the literature include municipal facilities such as landfills [Liu et al. 2020, Malovanyy et al. 2021, Tansel & Inanloo 2019, Talaiekhozani et al. 2018, Zhang et al. 2021], sewage treatment plants [Lewkowska et al. 2016, Sa et al. 2022, Zarra et al. 2008] and composting facilities [Cheng et al. 2019, Han et al. 2020, Han et al. 2019], as well as poultry farms [Amon et al. 1997, Guffanti et al. 2018] and livestock facilities [Andersen et al. 2013, Kim et al. 2008, Yao & Feilberg 2015]. Currently, the odor nuisance generated by such plants has become a flashpoint between them and the residents of the neighboring areas. The constant flow of complaints forces these institutions to take steps to counteract the generation of odors. One of the possibilities is the use of deodorization techniques for emitted gases, including absorption treatment processes.

Absorption is a process in which, due to the exchange of mass between the contaminated gas and the absorbing liquid (absorbent), one or more components of the purified gas mixture (absorbate) are absorbed by the contacting liquid. This process takes place in the entire volume of the absorbent and the necessary condition for its realization is the solubility of the absorbate in the applied liquid described by Henry's law (valid for moderate pressures and low concentrations).

Chemical absorption processes in particular are widely used in gas deodorization. Among other things, they are used to remove sulfur and nitrogen compounds from treated gas streams. An example is the recovery of elemental sulfur by hydrogen sulfide oxidation in the so-called Claus processes. This technique allows the recovery of sulfur from streams with high hydrogen sulfide concentrations and is reliable for daily sulfur production exceeding even 20 tons [Busca & Pistarino 2003]. A study reported in [Fruedenthal et al. 2005] analyzed the effect of the absorbent used on the efficiency of odorants removal from gases from the chocolate factory and fat and oil refineries. The key odorants include the following compounds: dimethyl sulfide, pentanediol, trimethylamine and acetic acid.

The most commonly used technique for assessing the effectiveness of the absorption gas treatment is gas chromatography coupled with mass spectrometry (GC/MS) or with a flame ionization detector (GC-FID). However, its use for continuous monitoring of industrial processes in real time (online mode) is difficult due to high operation costs or the need to supply high purity gases. In such cases, gas sensor arrays seem to be an ideal solution for this type of application. These devices reduce the time and cost of a single analysis and have grown strongly in popularity in recent years. This is evidenced by numerous publication reports on their application in various fields: from controlling biofiltration or methane reforming processes [Dobrzyniewski et al. 2021, Liang et al. 2020, Rolewicz-Kalinska et al. 2021, Rybarczyk et al. 2020], over applications in food or perfume industry [Dymerski et al. 2013, Gębicki et al. 2015, Mohd et al. 2020, Tan & Xu 2020] and monitoring of odor nuisance [Gębicki et al. 2014], to medical usage for early diagnosis of diseases [Saidi et al. 2020, Smulko et al. 2022].

In general, sensor matrices are devices that are used for simultaneous, sensory evaluation of multiple chemical compounds from different sources. Odorants are recognized thanks to the use of an array of non-selective chemical sensors, sensitive to different substances belonging to the same group of compounds. The detection capabilities of odorous compounds by the sensors are much lower than those presented by their biological counterparts, the olfactory epithelial cells [Gębicki et al. 2016]. However, on the other hand, they allow the identification of substances that are undetectable to the human nose, i.e. substances that do not have a smell [Di Francesco et al. 2001, Patel 2014]. The measuring system that shapes the sensor matrix analyzer consists of four main components: a sampling system, a detection system (a matrix of non-selective gas sensors), a data collection system and a signal identification (recognition, processing) system.

The use of a sampling system ensures the elimination of external factors that may interfere with the sensors and enables the achievement of stable and repeatable measurement conditions. The detection system is a matrix of gas sensors. Most often, commercially available sensors are used, not only because of their low prices, but also to allow easy reproduction of the prepared array. The output signal from the sensor array is multidimensional, so it is necessary to use a data acquisition system which is responsible for signal averaging and recording in the appropriate

form. The most important part of the analyzer is the signal identification systems. Using appropriate statistical methods and mathematical models, it allows to predict the concentration of the substances to be analyzed. The most commonly used methods for calibration of the gas sensor array are multiple linear regression (MLR), principal component regression (PCR) and partial least squares regression (PLSR).

This paper presents the use of a self-constructed gas sensor array for monitoring a deodorization process of the air stream based on the absorption method. The prepared matrix consisted of five commercially available gas sensors and the MLR model was used as the calibration method. The study was conducted on a laboratory scale and was designed to monitor the efficiency of absorptive removal of toluene from the air stream. Toluene is a hydrophobic odorous compound found, inter alia, in petroleum and the tolu tree. For this reason, toluene vapors are emitted into the atmosphere during fuel production or processes carried out in municipal waste treatment plants, such as composting. Moreover, it is commonly used in the industry of paints and varnishes, as well as in the tanning or perfumery industry for the production of disinfectants, washing agents, paints, adhesives and thinners. It is characterized by sweet, pungent, aromatic odor and a quite low olfactory threshold resulting in odor nuisance. However, its toxic effect on humans and other living organisms seems to be more dangerous. Inhalation of toluene vapors has been proven to be harmful to the respiratory, circulatory, reproductive, nervous and immune systems and, in its liquid form, to cause skin dryness and eye irritation. Examples of the adverse effects of toluene on humans depending on its concentration are shown in Table 1. As a

water-insoluble aromatic hydrocarbon, toluene can be difficult to remove from the air stream using biofiltration processes (poorly biodegradable), where the hydrophilicity of pollutants is one of the main factors affecting the efficiency of the process. Therefore, deodorization of the air stream contaminated with toluene vapors using absorption methods seems to be justified both from an engineering and technical point of view and also for safety reasons.

A commercially available liquid dedicated to the absorption of non-polar compounds was used as an absorbent. Gas chromatography coupled with a flame ionization detector (GC-FID) was used as the reference method to which the results obtained with the gas sensor array were referenced and compared. The use of gas chromatography to control the absorption removal of toluene has already been reported in recent publications [Chen et al. 2021, Ramos et al. 2020, Zhang et al. 2021], but the use of a gas sensor matrix for this purpose seems to be a novel approach. The conducted research consisted of three stages:

- Stage 1 – determination of breakthrough times of toluene and acetone on the applied absorbent;
- Stage 2 – process of absorptive treatment of the air stream from toluene vapor;
- Stage 3 – process of absorption air purification from toluene vapors with the addition of acetone.

The third stage was carried out in order to verify the suitability of the prepared sensor array with the developed MLR calibration model for monitoring the process of absorptive toluene removal from the air stream in the presence of other polluting compounds. Acetone, which is an organic solvent with high polarity, was selected as the contaminating compound for toluene vapor.

Table 1. Adverse effects of toluene on living organisms [Grant 1986, Zenz et al. 1994]

Concentration [ppm]	Signs and symptoms
50–100	Eye and upper airway irritation (after 6.5 hours), headache, no impairment in reaction time or coordination
100–200	Mild irritation of the eyes and throat
200–300	Noticeable sensation of eyes irritation, incoordination (after 8 hours exposure)
400–500	Lachrymation, severe throat and eye irritation
500–800	Dilation of pupils, fatigue and impairment of reaction (after 8 hours), slight pallor of fundi
1500	No lethal risk with exposure time not exceeding 8 hours
4000	Rapid impairment of reaction time and coordination, depression and probably death (after one hour or longer exposure)
10000–30000	Probable lethal threat after a few minutes

Additionally, like toluene, it is used in the production of dyes, paints, varnishes, cleaners and nail polish remover. Acetone is also proven to be emitted into the atmosphere from, for example, composting processes. It follows that its presence in waste gases from various industrial plants accompanied by toluene is highly probable.

MATERIALS AND METHODS

Experimental setup

The measuring stand used during the conducted research is presented in Figure 1. The purified air was directed into a toluene-filled barbotte using a mass flow controller (MFC 1). The air flow rate through the barbotte was 5 ml/min. The air stream contaminated with toluene vapors was then diluted using zero air generator which flow was controlled by a second mass flow controller (MFC 2). The gas mixture prepared in this way was directed to the absorption column. The volumetric flow rate of the gas mixture through the column was 100 ml/min. A commercially available absorbent Genosorb® 1843 (Clariant, USA) was used as the absorption liquid. According to the manufacturer, this liquid is dedicated and suitable for removal e.g. alcohols, ketones, aromatics, hydrocarbons, aromatic hydrocarbons and ethers. The basic physicochemical properties of the absorbent used are summarized in Table 2. Gas samples were taken and the inlet and outlet of the installation and analyzed using gas chromatography and a gas sensor array. TEDLAR film bags were used to collect the gas samples so that both measurement methods could be performed. The process was carried out until toluene concentration at the outlet of the installation reached the level of the inlet concentration. Samples were taken at five-hour intervals (approximately) or more frequently, and analysis of each of them was repeated three times.

Gas sample analysis

Gas chromatography analysis

Gas chromatography coupled with a flame-ionization detector (Varian CP-3800, VarianAnalytical Instruments, USA) was used as a reference method in this study. The concentration of toluene and acetone in gaseous samples was determined using a DB-624 column (60 m × 0.32 mm × 1.8 μm; Agilent Technologies, USA) and nitrogen as a carrier gas. The following chromatographic program parameters were used: injector temperature: 150°C; oven temperature: 150°C; FID detector temperature: 250°C; carrier gas flow rate: 3.5 ml/min; split ratio: 10; inject volume: 0.5 ml. Calibration was performed using the external standard
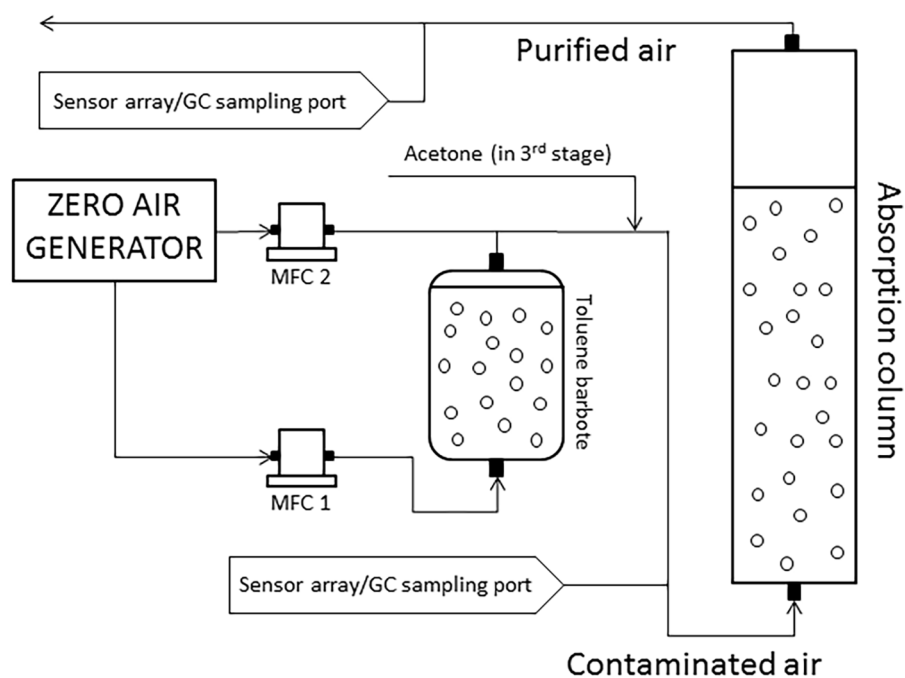

**Figure 1.** Absorption system used during the research

Table 2. Basic physicochemical properties of Genosorb ® 1843

Physicochemical properties	Unit	Value
Molar mass	g/mol	270
Appearance	-	Dark liquid, weak odor
Viscosity (at 20°C)	mm²/s	4-5
Boiling range (at 1013 mbar)	°C	>250
Vapour pressure (at 20°C)	mbar	<0.002
Density (at 20°C)	g/cm³	0.93
Water absorption (at 20°C)	%m/m	3
Solubility in water (at 20°C)	%m/m	1.5
Electrical conductivity (at 25°C)	S/m	1.10×10^9
Specific heat (at 50°C)	J/kg·K	2.20
Thermal conductivity (at 20°C)	W/m·K	0.1493
Flash point	°C	154
Ignition point	°C	205

method otherwise known as the absolute calibration method. In order to determine the calibration curve for toluene, gas mixtures were prepared in TEDLAR bags with defined concentrations of this compound. Ten gas mixtures were prepared with toluene concentrations ranging from 5 ppm to 80 ppm. This scope was selected based on publications reporting measured toluene concentrations in the vicinity of municipal landfills, composting facilities or paint manufacturing factories, where it ranged from concentrations of single ppm to as high as 100 ppm [Brosseau & Heitz 1994, de la Rosa et al. 2006, Eitzer 1995, Jafari et al. 2009, Kim et al. 1995, Nie et al. 2018, Thetkathuek et al. 2015]. For each prepared gas mixture, the chromatographic analysis was repeated three times. Figure 2 shows the calibration curve for toluene obtained from the analyses performed.

Gas sensor array

The constructed prototype of gas analyzer consisted of five commercially available gas sensors. Four TGS metal oxide semiconductors (MOS) sensors manufactured by Figaro Engineering Inc. (Mino, Osaka, Japan) and one photo-ionisation detector (PID) manufactured by Alphasense Metek (Great Notley, Braintree, United Kingdom) were used. Each of the sensors used in the array was located in an individual measurement chamber made of a material that does not adsorb contaminants, which was polytetrafluoroethylene (PTFE). The individual measurement environment allowed for the elimination of the influence of external factors on the signals from the sensors, and the non-adsorbing materials meant that subsequent analyzed samples were
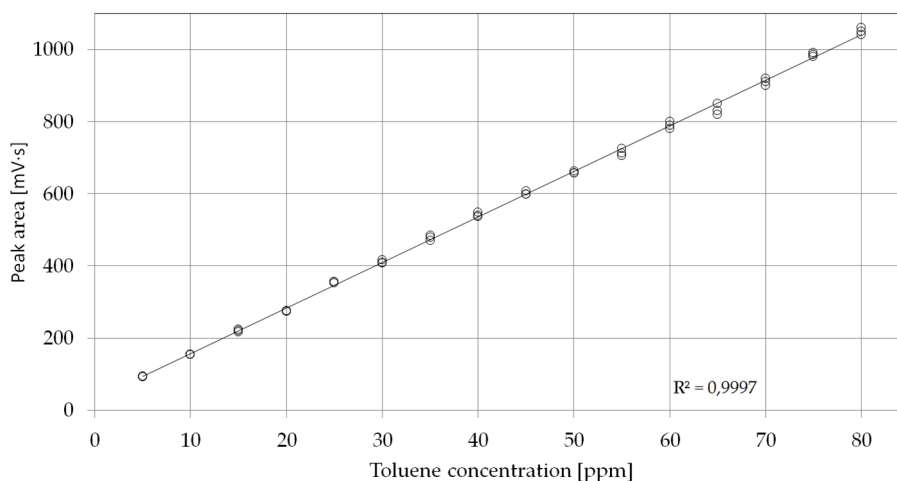
Figure 2. Gas chromatography calibration plot for toluene

not contaminated with the residues from previous analyzes. All electrical connections and circuits have been made in accordance with the manufacturer's recommendations. Table 3 presents the basic parameters and characteristics of the sensors selected for the constructed matrix.

Figure 3 shows a block diagram of the measurement system that enables the analysis of gas samples with the use of a constructed array. The pneumatic system consisted of a reference line (1) and a measurement line (2), and it was possible to switch between this two track thanks to a three-way valve (3). The reference line was connected to the valve's normally open channel and used to pass clean air through the chamber. This allowed the sensors to be restored to their initial parameters before subsequent analyzes. Through the measuring line, which was connected to the normally closed channel of this valve, gas samples were directed to the matrix of sensors (4). Replaceable dust filters (5) were located between the two position valve and the inlets of the individual line. The flow of the gas sample through the system was forced by a membrane pump (6). The gas sensor matrix worked in stop-flow mode: the sample flow time through the sensor chamber and the stopping time were both equal to 40 seconds. Then clean air was passed through the measurement chamber, and the sensors were flushed of any contaminants until the next analysis was performed.

The multilinear regression was used as a calibration method for the prepared gas sensor array. It is a statistical technique that makes it possible to predict the value of a dependent variable from the values of independent variables. In this particular case, the dependent variable was toluene concentration, and the independent variables were signals from gas sensors. The general equation of the MLR method is as follows:

$$y = \alpha_0 + \alpha_1 x_1 + \alpha_2 x_2 + \cdots \alpha_n x_n \qquad (1)$$

where: y is a dependent (predicted) variable; α_0 is an intercept; α_n are the regression coefficients and x_n are independent variables.

The sensor array was calibrated using the same gas samples that were used to determine the calibration curve for toluene in the case of chromatographic analyzes and the results from the *Stage 1* of the research where the breakthrough time of toluene through applied absorbent was determined using gas chromatography only. As a result of the conducted analyzes, a mathematical MLR model was obtained, showing the linear relationship between the signals from the sensors and the concentration of toluene, with the simultaneous rejection of irrelevant independent variables.

Data analysis and calculations

Data analysis and other calculations were performed using RStudio desktop (v.1.0.143)

Table 3. Models of chemical sensors used in the constructed matrix

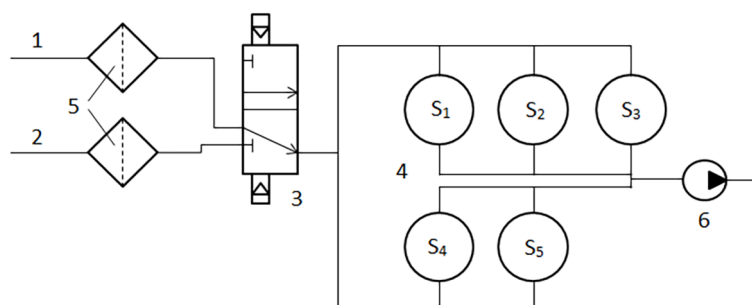| Indication	Sensor type	Model	Target gas
S_1	Metal oxide semiconductor	TGS2600	Air contaminants
S_2	Metal oxide semiconductor	TGS2602	VOCs and odorous gases
S_3	Metal oxide semiconductor	TGS2603	Air contaminants
S_4	Metal oxide semiconductor	TGS823	Organic solvent vaporous
S_5	Photo ionisation detector	PID-A12	VOCs with ionisation potentials <10.6 eV

Figure 3. Block diagram of pneumatic system used for gas sensor array measurements

software. For the calculation of the sensor signal the following relation was assumed:

$$\Delta S = S_{max} - S_0 \qquad (2)$$

where: S_{max} is the maximal signal value and S_0 is the sensor baseline value determined for synthetic air.

On the basis of such relations the model based on the MLR method was developed and was characterized by a determination coefficient (R^2) equal to 0.9860. The equation of the obtained model was as follows:

$$C_{toluene} = -360.743 + 151{,}696 \cdot \Delta S_2 - \\ - 43{,}221 \cdot \Delta S_4 + 786{,}316 \cdot \Delta S_5 \qquad (3)$$

As indicated by Eq. 3, in terms of the calibration performed, the signals from the first and third sensors were found to be statistically insignificant. The effectiveness of the absorption process

was calculated based on the values of parameter called absorptivity (A):

$$A = \frac{C_{out}}{C_{in}} \qquad (4)$$

where: C_{out} and C_{in} – concentration of toluene at the outlet and inlet gas stream, respectively.

RESULTS AND DISCUSSION

In the first stage of the study, the breakthrough time of toluene and acetone on Genosorb® 1843 (Clariant, USA) was determined. For toluene, this time was about five thousand minutes and was approximately ten times longer than for acetone. The experimentally determined breakthrough times of both substances are shown in Figure 4.
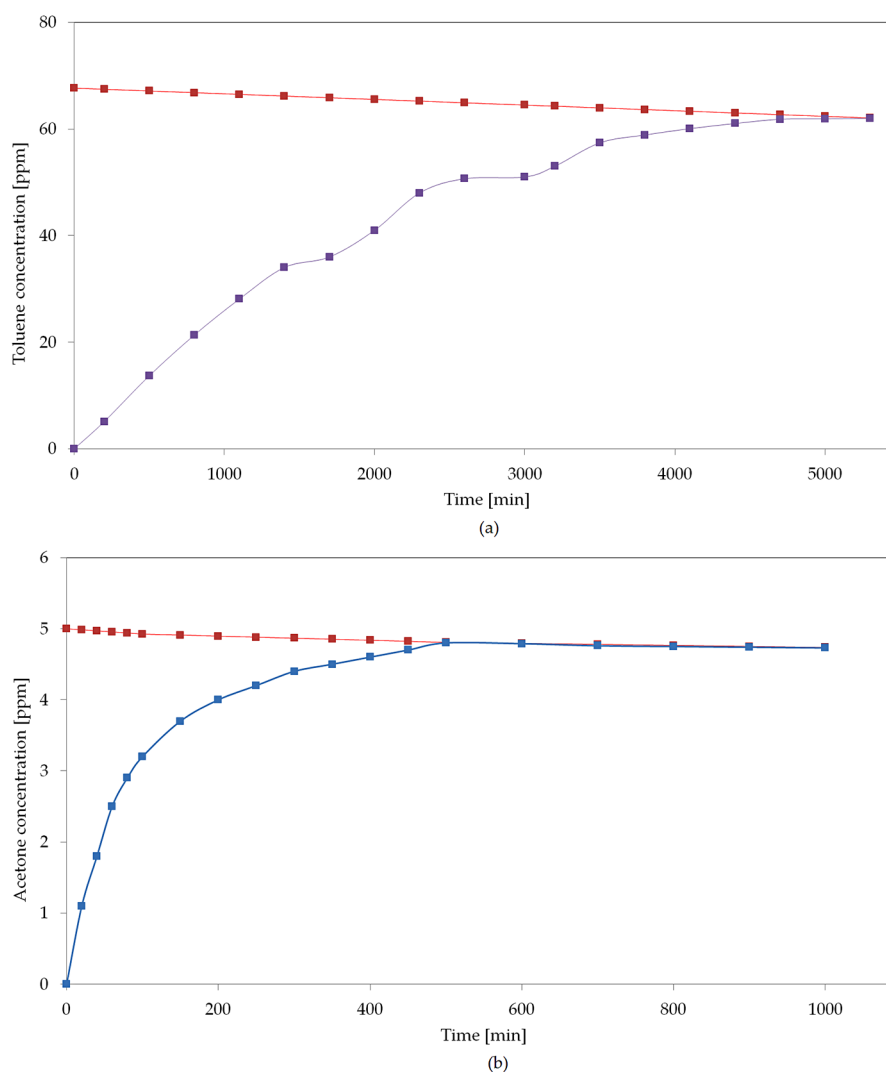

Figure 4. Breakthrough times of toluene (a) and acetone (b) on Genosorb

Based on the results obtained in the first stage and the gas mixtures used in the chromatographic calibration process, the gas sensor array was calibrated using the MLR method. Calibration plot of the prepared model is presented in Figure 5.

In the second stage of the research, the process of air absorption purification from toluene vapors was monitored and controlled in terms of the concentration of this compound at the inlet and the outlet of the installation. Chromatographic and sensor techniques were used for this purpose. Figure 6 presents the variation of toluene concentration in the polluted and treated air stream over time. The Root Mean Square Error was determined to compare the results obtained using the gas sensor array with the reference method. For the inlet stream it was 1.3 ppm, and for the outlet stream its value was equal to 1.6 ppm.
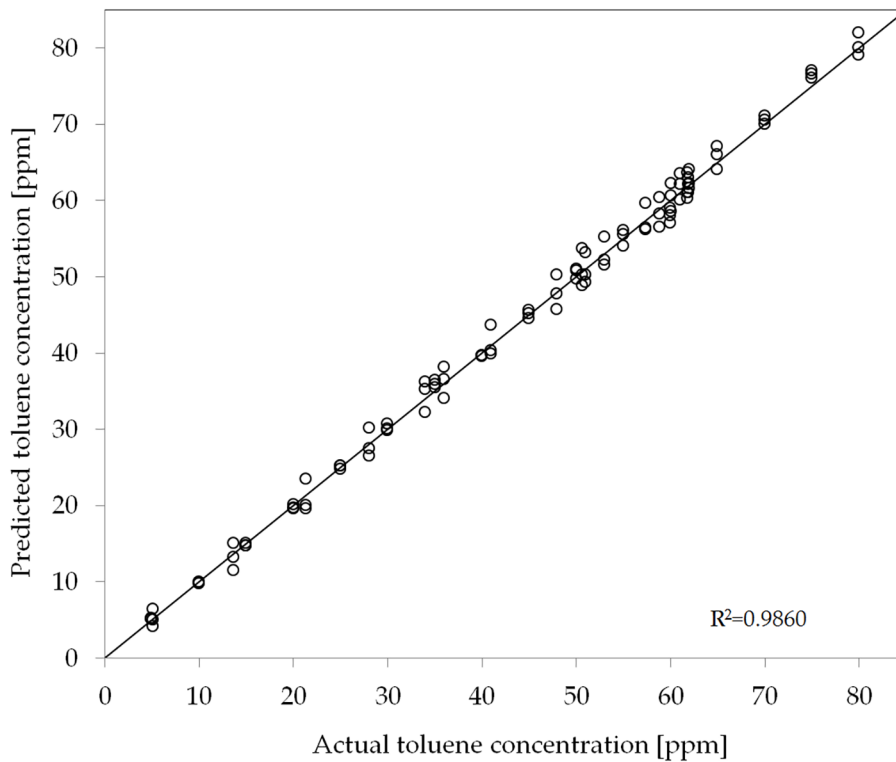

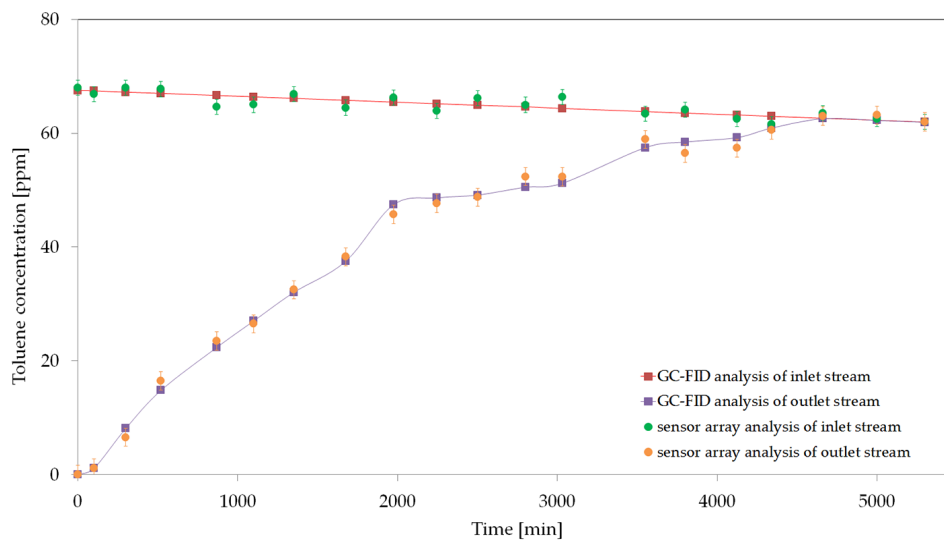**Figure 5.** Multiple linear regression correlation plot

Figure 6. Changes in toluene concentration at the inlet and outlet of the installation as a function of the process duration

In the third stage, the performed process of absorbing toluene removal from the air stream was repeated, but this time acetone was added as contaminant. Concentration of acetone in the inlet stream was set at 5 ppm level. The same measurement techniques and unchanged calibration MLR model for the prepared sensor array were used. Changes in toluene concentration over time in this case are shown in Figure 7. For this process, the RMSE was 1.7 ppm and 3.1 ppm for the inlet and outlet stream respectively. In order to determine the applicability of the developed MLR model, the absolute errors between chromatographic and sensor measurements were determined for the process carried out in Stage 2 and Stage 3 (Figure 8). On the basis of Figure 8, it can be concluded that
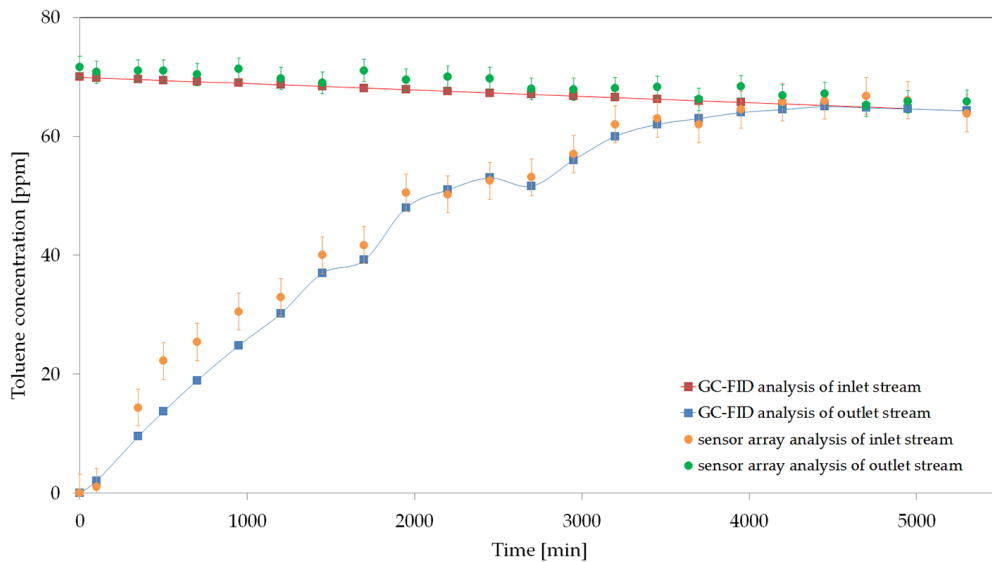

Figure 7. Changes in toluene concentration at the plant inlet and outlet as a function of process duration with acetone addition
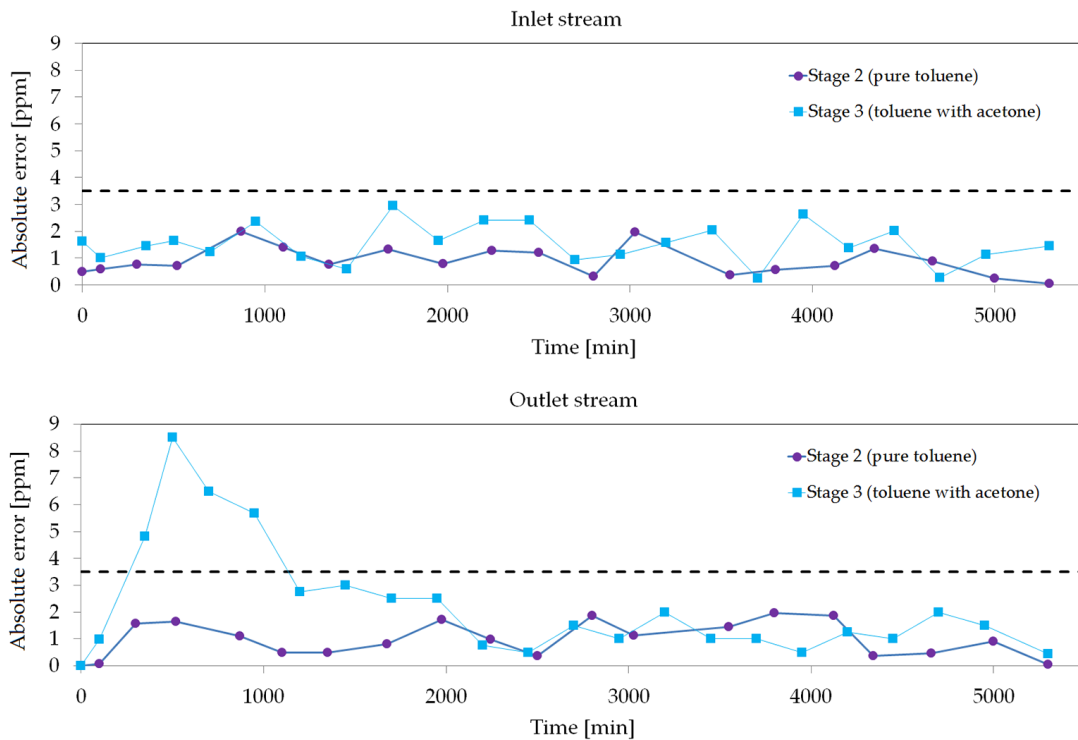

Figure 8. Absolute errors between chromatographic and sensor measurements for stage two and stage three processes

the prepared model predicts the concentration of toluene in the presence of an interfering substance slightly worse, but the differences are acceptable. In each of the conducted processes, the inlet toluene concentration was about 70 ppm and varied slightly during the processes. Assuming that the 5% error in the gas sensor array with the MLR model concentrations prediction is acceptable, this means that the difference between the chromatographic and sensor measurements should not be higher than 3.5 ppm. Figure 8 shows that even in the presence of an interfering substance, this value was not exceeded at any stage of the process when the inlet concentration was measured. The error values were slightly higher than in the case of the process without acetone addition, in which they do not even exceed 2 ppm, but still within

the accepted error limits. In the case of the outlet streams, the situation is somewhat more complicated as the concentrations of the toluene and acetone vary over time. For the absorption of toluene from the air stream (Stage 2), the developed MLR model predicts the concentration of this substance with high accuracy in relation to chromatographic analyzes, and again the measurement error does not exceed 2 ppm. However, when removing toluene with acetone from the air stream (Stage 3), the model prediction error exceeded the acceptable range at some point in the process. It was related to the variation of the acetone concentration in the outlet stream, and the greatest error were recorded approximately when the acetone broke through the absorbent used (about 500 min). Then the error between the chromatographic and sensor
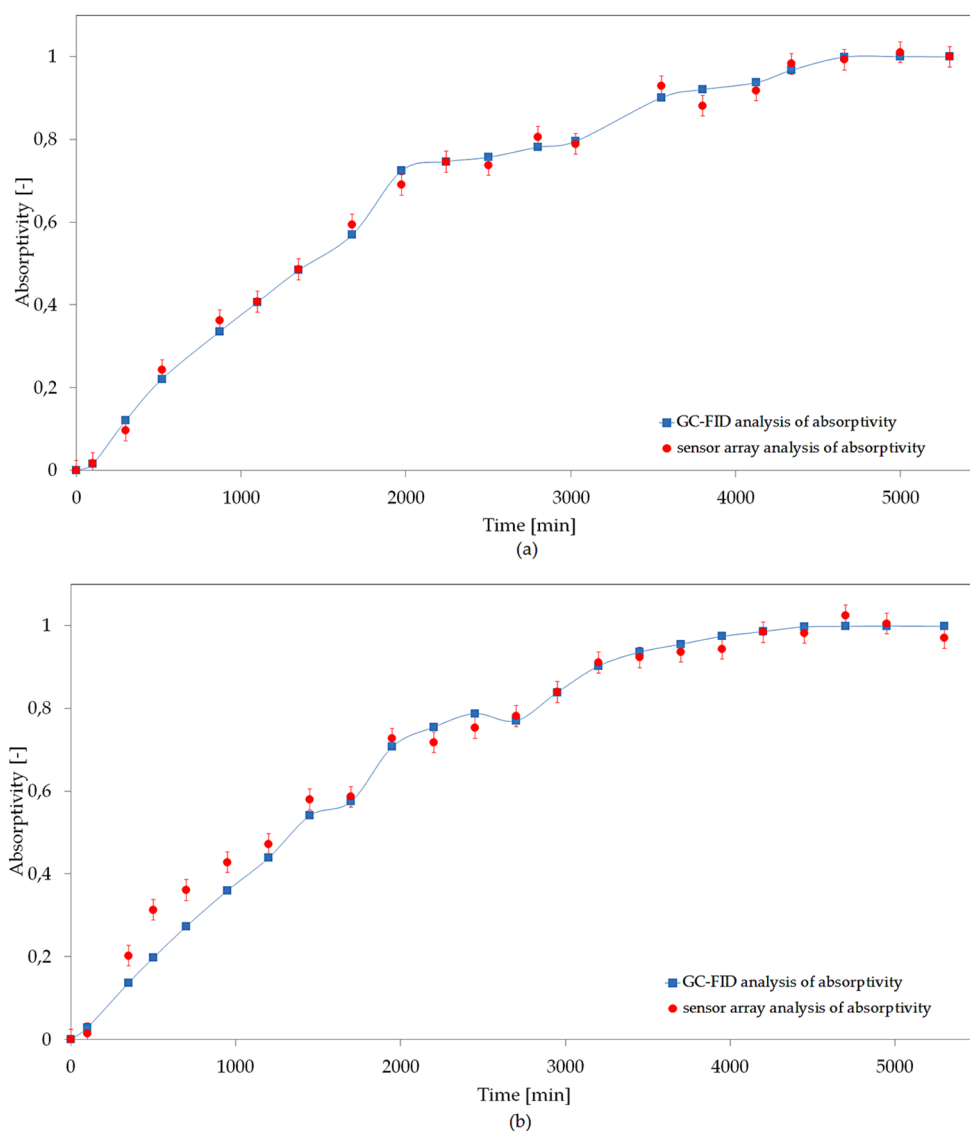

Figure 9. Experimental absorption breakthrough curves of Genosorb: (a) pure toluene absorption process; (b) the process of toluene absorption with the addition of acetone

measurements reached the maximum value of 8.5 ppm. After this time, the error values in subsequent measurement points gradually decreased and after about 1000 minutes of the process, they returned to the acceptable range and this was the case until the end of the research. This means that the values of errors in predicting the concentration remained at the same level as in the case of the absorption purification of toluene itself from the air stream. The final step of the study was to determine the efficiency of the conducted absorption air treatment process. Figure 9 presents the dependence of absorption effectiveness as a function of process duration. The RMSE for the entire process was 0.019 (1.9%) in Stage 2 and 0.041 (4.1%) in Stage 3.

Despite the fact that sensor matrices arouse great interest and expectations, during environmental tests they often do not provide satisfactory results. Currently, most industrial enterprises do not regularly use them as the main method of analyzing the odor air quality or process monitoring, but they find application in research activities. This is caused by many factors: from improper operation, both in the measurement phase and during data analysis, to technical limitations related to the instability and lack of the repeatability of the gas sensors. The effectiveness of sensor matrix analyzer is also strongly influenced by environmental parameters, mainly humidity and temperature, whose variability is difficult to control. Moreover, gaseous process streams emitted from industrial plants often exhibit much more complexity than model laboratory mixtures on which the process of "learning" of the constructed device was implemented. These differences in chemical composition could cause misinterpretation of odor by the devices and at the same time require continuous improvement of all four sensor matrices forming systems. On the other hand, small improvements of the proposed sensor system such as sample cooler, dilution module or systems controlling temperature and pressure inside the device would allow them to be used as measuring elements in automation systems to online management of processes carried out in various types of industrial plants. An interesting solution seems to be the use of the gas sensor arrays as supporting devices for olfactometric measurements. Calibration of the sensor array using a field olfactometer along with an appropriately selected mathematical model would enable the use of this type of device to monitor air quality in terms of odor concentration. Such a solution could be useful for municipal waste treatment plants and landfills that are constantly struggling with human complaints. The use of the gas sensor arrays in this form would allow them to constantly control the odor nuisance at the site and verify the validity of incoming plaintions.

CONCLUSIONS

On the basis of the conducted research, it has been shown that the constructed gas sensor array together with a properly selected calibration mathematical model can be successfully applied to monitor and evaluate the effectiveness of the absorption process of air stream purification from toluene vapors. The selected and developed for this study MLR model was characterized by high agreement and compliance with the results obtained by gas chromatography, which was treated as a reference technique. This confirms the feasibility and validity of using sensor matrices as an alternative to chromatographic methods while maintaining the results at the similar levels of quality. The study also demonstrated the high selectivity of the developed MLR mathematical model in relation to acetone as an interfering substance.

The main advantages of sensor analyzer from the point of view of monitoring the absorption processes are: real-time (online) process control without taking any samples, identification of odorants with very low olfactory thresholds, uniqueness of the generated signal (different set of signals for each tested mixture), they could be constructed as portable devices (measurement in the immediate vicinity of pollution sources) and have sensor matrices in the form of easily replicable modules, which allows to adapt the device to the expected composition of the process gas stream. In conclusion, it should be noted that compared to other analytical techniques, sensor arrays are technologically less complex, useful for laboratory purposes, as well as showing application potential for field studies. However, there is a need to continuously develop more sensitive and selective arrays of chemical gas sensors and to be fluent in advanced data processing and identification techniques.

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