Semi-quantitative Monitoring of VOCs Emission Decay based on Gas Sensor Array and Graphical Display

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Abstract: We propose a method of evaluating volatile organic compounds (VOCs) emission decay semi-quantitatively. The method utilizes continuous measurements performed with gas sensor array and the simultaneous visualization of measurement data. Individual VOCs are evaluated against the graphically displayed quantitative criteria, which describe the stages of the emission process. Based on the displayed information, human operator evaluates the gas mixture composed of the emitted VOCs. In order to visualize the data together with the quantitative criteria the two-dimensional feature space was proposed. A number of issues were discussed in this work regarding the selection of the adequate feature spaces and suitable methods of their partitioning in order to reflect the stages of the VOCs emission process in the structure of the feature space.

1 INTRODUCTION

Over the last decades it has become evident that many products can be major contributors to the pollution of the indoor environment with volatile organic compounds. These substances are found in wide variety of everyday products such as floor coverings, solvent-based paints/coatings, adhesives, sealants, printing inks, many consumer products, organic solvents and petroleum products. VOCs contained in products tend to diffuse to the surface of the products and then they are emitted to the air over time. When these emissions occur indoors, building occupants are exposed via inhalation. Unfortunately, some of the emitted VOCs may affect the perception of the indoor air quality, e.g. in the form of odor nuisance, eye and airway irritation. Thus, there is an increasing awareness and emphasis on receiving the information, whether concentrations exceed levels that may affect human health or comfort. Therefore manufacturers are increasingly asked or required to demonstrate that vapor-phase emissions of chemicals of concern from their products under normal use conditions comply with various voluntary or regulatory acceptance criteria. In practice, it is difficult to accomplish this requirement. In general, the mechanism of VOCs release from products and the factors influencing the emission process are complex and can involve the diffusion of VOCs within the product and their evaporation from the surface to the ambient air. Additionally, the emission rate of VOCs may be affected by the concentration of these species in the air, decomposition related to the hydrolysis or heat, oxidation, chain breakdown resulting in the smaller and more volatile molecules as well as the sorption on the material surface. All these phenomena cause that the process of VOC emission is time dependent. For these reasons, there is the absence of a uniform procedure for modelling and calculating the emission rate of VOCs.

Other approach requires manufacturers to have their products periodically tested for VOC emissions by independent laboratories using designated reference test methods (for example, Test Method D6007, ISO 16000-9, and ISO 16000-10). This strategy very often uses chromatography and spectroscopy which are accurate and precise. However, reference methods for testing chemical emissions from products are typically expensive, too time-consuming and impractical for routine emission monitoring in a production environment. So, there is a great need for rapid, easy-to-use, inexpensive, portable instruments for real-time, continuous or periodic measurements performed in situ or on-line,

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that do not require specialist technicians. This demand can be fulfilled by the devices equipped with the solid-state chemical sensors. Among solid-state chemical sensors, tin oxide semiconductor sensors developed by Taguchi are highly sensitive to volatile organic compounds (VOC) and other reducing gases. Unfortunately, they cannot be used as individual devices for qualitative and quantitative analyses, because of very low selectivity. This problem may be solved by the use of the measurement system consisting of a suitable sampling system, an array of gas sensors with partial or overlapping sensitivities and a pattern recognition component capable of discriminating between simple as well as complex gas mixtures (Jurs et al., 2000).

In practice, there is frequently a need for rapid characterization of volatile compounds emission. Such measurements are performed in an analysis mode termed “semi-quantitative”. They are not designed to offer the ultimate accuracy but to provide the results with the sufficient accuracy in both an analysis time and labour efficient manner (Lemke et al., 2005, Dias et al., 2011). The aim of this work was to propose a method of semi-quantitative analysis of VOCs multi-component gas mixtures which are emitted from different products.

Several assumptions were made in our studies. The first one was related to the instrumental aspects of semi-quantitative analysis. The elaborated system should be based on: (1) semiconductor gas sensor array, (2) dynamic mode of operation and (3) pattern recognition module. Sensor array usually generates highly dimensional response to the target gas. Therefore it is difficult to extract the information useful for solving the problem under investigation from the obtained data. Graphical methods offer a simple way of analyzing data in an exploratory manner (Scott et al., 2007). For that reason, it was assumed that the graphical method of data analysis could be used in the system for semi-quantitative measurements. Additionally, we assumed that from the start, the emission of VOCs decreases over time and the decay process is relatively fast. In our work, semi-quantitative analysis was designed to provide the information which allows for comparing the concentration of the selected species with the standards using minimum effort and in close to real time. It is assumed that the investigated substances belong to the brad category of VOCs are they are known in advance. By definition, the accuracy of the method should be in the range of ± 30 to 50 %.

2 MATERIALS AND METHODS

2.1 VOCs Gas Mixture

In order to test the approach we focused our attention on the four-component gas mixture. It was composed of toluene, hexane, acetone and ethyl acetate, see Fig. 1 and Table 1. These VOCs are widely used in many commercial products and by means of evaporation from the surface they find their way to the surrounding air. Fourteen series of gas mixture samples were experimentally examined.

Table 1: Initial concentrations of four VOCs in each of fourteen measurement series [ppm].

Series	Toluene	Hexane	Acetone	Ethyl acetate
1	101.0	302.0	650.2	146.8
2	110.4	299.6	640.3	149.7
3	122.0	296.6	628.1	153.2
4	136.7	292.9	612.7	157.7
5	155.9	288.0	592.5	163.5
6	182.0	281.4	565.2	171.4
7	219.5	271.9	525.9	182.8
8	277.9	257.0	464.5	200.5
9	381.8	230.6	355.5	232.1
10	618.0	170.7	107.6	303.7
11	874.2	0.0	0.0	325.8
12	959.1	0.0	0.0	240.9
13	1064.9	0.0	0.0	135.1
14	1090.9	0.0	0.0	0.0

Each series of gas mixture samples started from different initial concentrations of VOCs (Table 1).

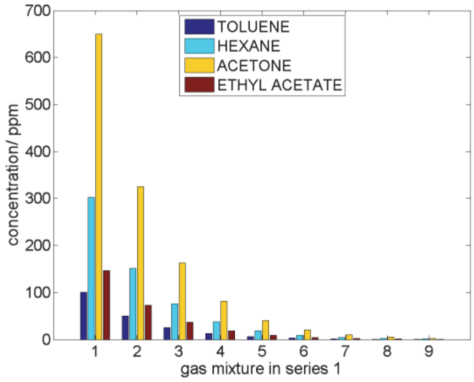

Figure 1: Single measurement series of gas mixtures.

There were chosen decreasing concentrations within series, in order to simulate the decay of VOCs emission process in a manner which is shown in Fig. 1. The measurements were repeated three times.

2.2 Gas Sensor Array

The experimental setup consisted of several functional blocks. Pure and dry air was produced using zero air generator manufactured by Horiba. The installation for standard gas preparation included vaporizer in the form of the heated glass coil with an injection port, Tedlar bag (12 l) and the chromatography syringe (Hamilton). The module for delivering and control of the gas flow was equipped with the diaphragm pump, mass flow controllers, valves and Teflon gas line. Sensing array was equipped with the set of six commercially available Taguchi Gas Sensors made by Figaro Engineering Japan. There were applied: TGS2620, TGS821, TGS825, TGS826, TGS2104, TGS2602. Each sensor was mounted inside its own, specially designed, airtight, flow-type test chamber. Chambers were made of aluminum. The chambers were provided with gas inlet and outlet and they were connected parallel using Teflon-tubing. Such configuration allowed for the simultaneous exposure of the sensors to the same gas. The sensor array was attached to the module for delivering and control of the gas flow. The chambers were also equipped with electrical connections. Each sensor was connected to the voltage supplier and electronic circuit. The output signals were measured in the form of voltage variations on the load resistance with the time resolution of 1 s. The ADC (Maxim 1231) was used for analog to digital signal conversion. Signals were recorded by the application developed in LabView. Personal computer with the suitable software was used for data storage, processing and analysis. The system was operated in the dynamic mode. The sensor signal was collected for a predefined period of time. The responses of sensor array at each time point of exposure to the test gas were considered as distinct sources of information about the state of the gas mixture. They were the candidates to become the basis of semi-quantitative gas mixture assessment.

3 MEASUREMENT DATA VISUALISATION FOR GAS MIXTURE MONITORING

Semiconductor gas sensor array was chosen as the source of measurement data to be applied for VOCs emission decay monitoring. Due to their operation principle sensors provide the data which contains wide range of information about the tested gases. Very accurate information may be acquired from sensor array data provided that the adequate pattern recognition methods are applied (Bermak et al., 2006). This requirement comes from the fact that the data has multivariate character and it is usually featured by a considerable redundancy. In this work, we proposed a simple manner of analyzing gas sensor array measurement data. It was designed to satisfy the objective of performing the semi-quantitative assessment of emitted VOCs gas mixture. The principal element of the concept was the visualization of the data.

There are many thinkable ways of sensor array data visualization, radar plots being most popular among them. However, the graphical representation of measurement data, which is to serve semi-quantitative assessment of gas mixture, imposes certain requirements on the method. The chosen domain of graphical representation shall allow for: 1) simultaneous visualization of the data and the criteria of its division, 2) establishing the semi-quantitative relationship between the data and the criteria, based on the visual inspection.

In this work there was exploited the concept of employing feature space as the visualization domain. In principle, the feature space encompasses the representations of all possible states of gas mixture. There was performed a division of the feature space. The division resulted from solving the gas mixture classification problem in that space. The division criteria had the quantitative character i.e. they referred to the quantitative parameter(s) of gas mixture. As a result, there were separated the sub-spaces containing representations of gas mixture compositions, which complied with distinct criteria. The criteria were displayed in the feature space as the decision boundaries.

We chose two-dimensional feature spaces for displaying the criteria and the measurement data. More dimensions were not allowed to assure comprehensive visualization. The current state of gas mixture was represented as a wandering point in the feature space. The coordinates of the point were defined by the result of measurement obtained upon dynamic exposure of sensor array to the investigated gas.

It was proposed to monitor gas mixture evolution by simultaneously analyzing the change of its main components. Separate display panel was dedicated to demonstrate the fate of each component of gas mixture. The evolution of single component was carried out against quantitative criteria. Depending on the application they could refer to concentrations of substances or other quantities describing environmental impact of the test gas e.g. odor intensity. The background of the display was to provide visually the information on parts of the feature space which represent particular stages of the emission process decay. The partitioning of the feature was guided by the mentioned criteria. The gas mixture component evolution would be evaluated by looking at the location of the data point representing current measurement results against the background information. The combined assessment of gas mixture would be based on the human judgment. It shall utilize the information about all or just selected components of the gas mixture, presented in the display panels.

A number of issues had to be considered while designing a visualization of measurement data in the proposed manner. The main ones were: 1) the selection of features to form feature spaces, 2) the selection of method to organize the feature space in a way that its different regions correspond to the subsequent stages of evolution of gas mixture composed of the emitted VOCs. In both cases the selection criteria had to be articulated and applied.

We proposed to anchor the feature space in a defined time point of exposure to the test gas in the dynamic mode of operation.

There were proposed two kinds of feature spaces for consideration. The first kind was formed by single time point signals of two different sensors. The main advantage of using the time point sensor signal as the feature is the direct transfer of the sensor measurement result to the display, as the data pre-processing is not required. In such case, there are a number of available feature spaces to choose. They may be associated with different time points of exposure and also different pairs of sensors may be considered. The main drawback of this solution is that features of this kind are not orthogonal. The second kind of feature was the principal component (PC) resulting from the transformation of the data associated with a single time point of sensor array exposure to the test gas with principal component analysis. This kind of feature represents the combined information provided by all sensors in the array. The features are orthogonal, but the measurement data preprocessing step is required for obtaining them. It was allowed that the feature spaces selected for different components of gas mixture are associated with different time points of exposure to the test gas.

In this work, we applied an exhaustive search across the pool of all two-dimensional feature spaces in order to find the best ones.

The simplest way to display measurement results which allows for tracing the gas mixture evolution in feature space is to use the data on the reference gas mixtures as the 'background', as shown in Fig. 2a.

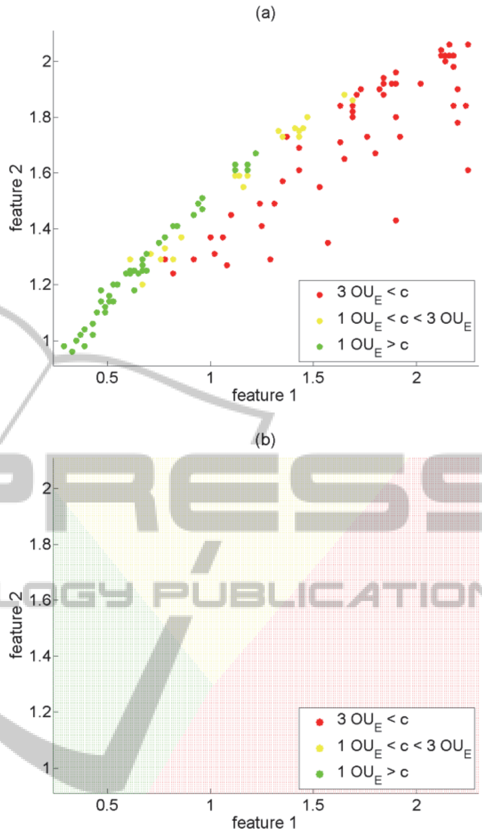

Figure 2: Two options of the background information visualization on the gas mixture evolution in feature space: a) with the use of reference gas measurement data, b) by applying feature space division.

The data may be shown using different colours or shapes which indicate separate categories of reference gases, associated with different stages of VOCs emission decay process. The way to evaluate the current state of gas mixture is by comparing the location of the measurement data point with the location of reference data points on the display. This simple solution has one major drawback. The test gas mixture may be evaluated if the result of the current measurement shows up in the part of the display which is populated by data points associated with the reference gas mixtures. While moving out of this range the assessment becomes dubious due to lack of reference. In practical applications, such situation is very likely.

This major shortcoming may be overcome by applying a classification engine for feature space structuring. It is a way of dividing entire feature space in an objective manner, see Fig. 2b. Two issues shall be addressed in relation to that operation. These are: the method of dividing feature space and the formulation of the classification problem.

There are basically utilized the distance principle and the decision boundary concepts while developing classifiers (Snopok and Kruglenko, 2002). In the first approach the class assignment is performed by calculating the data point distance to different classes and by choosing the least distant class. The second strategy involves the division of the feature space into subspaces by imposing decision boundaries which are in general multidimensional surfaces (in 2D space these are lines). They divide the space into disjoint regions associated with distinct classes. The advantage of the second approach consists in proposing the division of entire feature space directly. Although far from the regions populated by the calibration data points the division shall be treated with caution the feature space is reasonably well structured close to them. Moreover, the class assignment of the new data is related to its location with respect to the displayed decision boundaries. In the particular application considered in this work, the methods based on decision boundaries would be favored also for another reason. We found that the set of points representing the gas mixture evolution has a characteristic layout in the feature space, see Fig. 2a. They are not arranged into a number of disjoint clusters but they rather form one stretched grouping. Due to the continuous transition between the stages of gas mixture evolution the data points representing subsequent stages are neighbours. In such circumstances, the methods designed to cope well with compact and disjoint clusters are inappropriate. In this work, Discriminant Analysis was applied for feature space partitioning. It is the most simple and least time consuming classification method based on decision boundary concept. We tested whether the liner technique (LDA) or Quadratic Discriminant Analysis (QDA) is more suitable for our application.

The way of formulating the classification problem gains importance while applying multi-criteria division of the feature space. It is a necessity upon monitoring the evolution of gas mixture. Willing to discriminate *k* stages of gas mixture emission decay one may decide to apply one division of feature space into *k* classes or *k*-1 two-part divisions. While the first approach is quite inflexible, the other leaves space for some adjustment of the feature space structure to the relative importance of the individual criteria. In this work there were applied the criteria allowing to assess VOCs emission process regarding their odor impact (CIWEM's, 2011). We were willing to discriminate between the following three stages of gas mixture evolution: odor intensity below 1 OU_E/m^3, odor intensity between 1 OU_E/m^3 and 3 OU_E/m^3 and odor intensity higher than 3 OU_E/m^3. In principle, the examination was performed for each component of gas mixture separately.

Finally, one shall articulate the criteria of selecting the best feature space. In this work we considered two criteria. The first criterion was the misclassification rate of reference gas mixtures. This objective criterion indicated how successful was the particular feature space together with the way of its partitioning in assigning training patterns, representing different stages of VOCs emission process to the appropriate classes. There was also applied other, subjective criterion. It referred to the correspondence between the logics of feature space fragmentation and the logics of gas mixture evolution. Namely, there were examined the arrangement, in particular the neighbourhood, of distinct fragments of feature space, representing the sequence of VOCs emission decay stages.

4 RESULTS AND DISCUSSION

The accuracy of reference gas mixtures classification was the first criterion employed for the assessment of data visualization method dedicated to semi-quantitative gas assessment using sensor array. The criterion was aimed to represent the accuracy of feature space division with respect to the stages of VOCs emission process. The following aspects of visualization were examined with this criterion: 1) selection of feature space coordinates (single sensor related or sensor array related), 2) method of feature space partitioning (linear or nonlinear), 3) formulation of feature space division problem (*k*-1 divisions into two-part, or one division into *k* parts).

The misclassification rates of reference gas mixture patterns are presented in Table 2, Table 3 and Table 4. The values shown in the tables are medians. They provide a statistical indication of misclassification rates obtained in feature spaces, which were built separately for each time point of sensor signal obtained upon dynamic exposure. Due to the fact that misclassification rates were low for most of the time points, the error rates shown in Table 2, Table 3 and Table 4 well represent minimum misclassification rates achievable with the particular approach. The pairs of best sensor are quoted in Table 5.

Table 2: Error rates while applying k-1 two-part divisions in best feature spaces, by means of LDA. Criterion I: 1 $\mathrm{OU_E/m^3}$, Criterion II: 3 $\mathrm{OU_E/m^3}$.

VOC	Best sensor pair		PC1 - PC2	
	I	II	I	II
Toluene	13.49	7.14	9.52	18.25
Hexane	2.38	4.76	7.94	14.29
Acetone	7.94	8.73	15.87	11.11
Ethyl acetate	11.90	7.94	14.29	8.73

Table 3: Error rates while applying k-1 two-part divisions in best feature spaces, by means of QDA. Criterion I: 1 $\mathrm{OU_E/m^3}$, Criterion II: 3 $\mathrm{OU_E/m^3}$.

VOC	best sensor pair		PC1 - PC2	
	I	II	I	II
Toluene	7.14	4.76	7.94	9.52
Hexane	3.17	0.00	3.17	0.00
Acetone	3.97	1.59	7.94	3.17
Ethyl acetate	12.7	5.56	12.70	7.94

Table 4: Error rates while applying one division of feature space into k parts.

VOC	LDA		QDA	
	best sensor pair	PC1-PC2	best sensor pair	PC1-PC2
Toluene	23.20	26.98	13.49	19.50
Hexane	3.97	11.11	3.17	5.56
Acetone	13.49	23.02	8.73	14.29
Ethyl acetate	15.08	16.67	11.11	12.70

Table 5: Best sensor pairs.

VOC	k-1 two-part divisions		one division into k parts	
	LDA	QDA	LDA	QDA
Toluene	TGS825, TGS2104	TGS825, TGS2104	TGS825, TGS2104	TGS826, TGS2104
Hexane	TGS825, TGS2602	TGS826, TGS2104	TGS825, TGS2602	TGS2620, TGS2104
Acetone	TGS826, TGS2104	TGS826, TGS2104	TGS826, TGS2104	TGS825, TGS2104
Ethyl acetate	TGS825, TGS826	TGS2620, TGS826	TGS825, TGS2602	TGS825, TGS2104

Based on the joint analysis of Table 2, Table 3 and Table 4, the application of k-1 two-class divisions of feature space resulted in lower misclassification rates as compared to one division into k fragments. The differences were considerable and in single cases they exceeded 10 %. In general, nonlinear classification offered better results than linear. Regarding kind of features applied for constructing the feature space it was shown that responses of best pairs of sensors outperformed principal components. The gain was less obvious while applying k-1 two-class partitioning (Table 2 and Table 3) than in case of one division into k classes (Table 4). It is noticeable in Table 5 that different sensor pairs were found best for tracing the evolution of the individual components gas mixture. Except for one case (Table 4, toluene) all considered methods allowed for the assessment of gas mixture evolution stages with an error not exceeding 20 %. In most cases the error was lower than 10 %. The obtained result is satisfactory in view of ±30 to 50 % accuracy expected from the gas sensor system which performs semi-quantitative analysis.

While choosing the criterion of best separation of classes as decisive, the obtained results indicate that, the best choice for visualization of gas mixture evolution is: 1) selecting responses of single sensors as coordinates, 2) applying nonlinear division of feature space and 3) using k-1 two-part divisions of feature space. However, while designing the visualization system the consideration of other aspects is also worthwhile.

The first of other aspects considered in this work is the reflection of the logics of gas mixture decay in the structure of feature space. It is shown in Fig. 3 that illogical divisions are likely to occur while applying nonlinear method of feature space division/data classification.

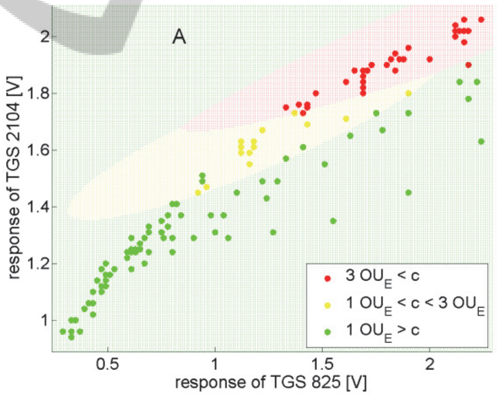

Figure 3: Illogical division of feature space realized by QDA.

From the logic of the arrangement of reference gases data points, it is unlikely to encounter low odor intensity gas mixtures in the region of feature space marked A (Fig. 3). The structure of feature space proposed by the classifier is questionable. The problem indicated by the presented example may be encountered both, when applying multiple two-class partitioning as well as one division into k parts. It comes from the fact that nonlinear classifier tends to tighten nonlinear boundaries around groups of points which belong to the same class. It result in lower misclassification rates (Table 2 to Table 4) but at some distance from the centers of the classes, the organization of the feature space may fail to agree with the logics of the variability of real object represented by the data. The linear solution, although less advantageous in terms of the classification performance is less likely to generate such misinterpretations.

The second of other visualization aspects considered in this work is associated with the multiple two-class approach to the feature space division. While choosing this strategy instead of one multiclass division, the designer of data visualization for the sensor system may decide about the priority of one or the other criteria of gas mixture state assessment. This problem is illustrated in Fig. 4. An example of linear division was chosen for showing, although the same possibility is available while using nonlinear classifier.

The region marked A, between the dashed lines (red and green), may host data points considered as representing highly odorous gas mixture or low odor intensity gas mixture depending on whether the priority was given to the decision boundary set at c = 3 OU_E/m^3 or to c = 1 OU_E/m^3. In the second case the field denoted with A shall be marked green.

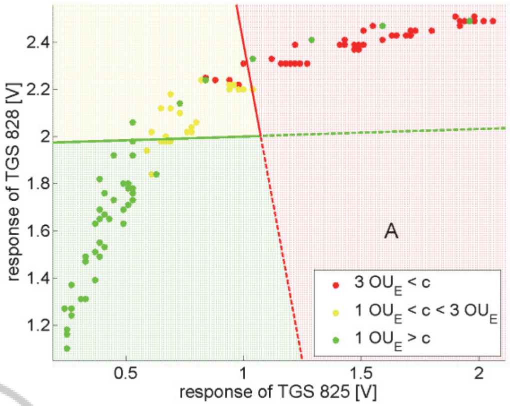

Figure 4: Prioritization of the criteria for gas mixture evolution stages detection.

The division shown in Fig. 4 would be favoured

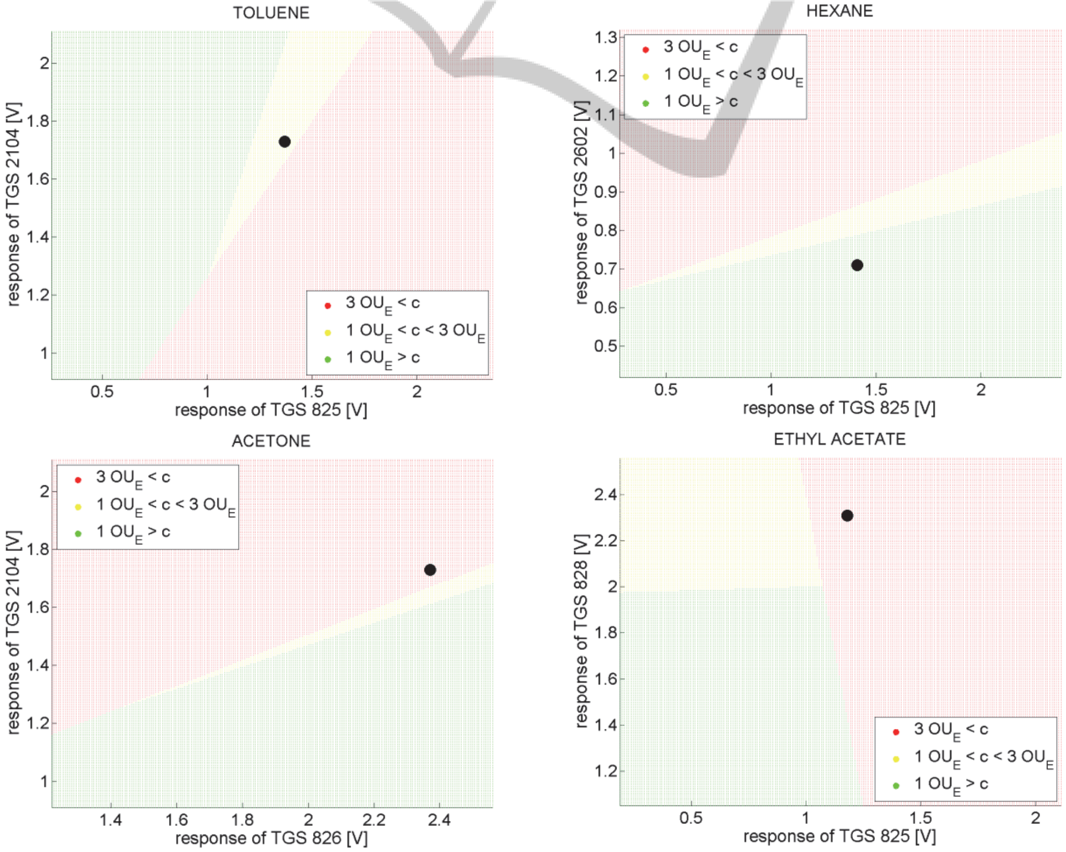

Figure 5: Sensor array measurement data visualization for semi-quantitative monitoring of VOCs emission decay process. There is shown a exmple of a display designed for four-component gas mixture. We were willing to discriminate between the following three stages of gas mixture evolution: odor intensity below 1 OU_E/m^3, odor intensity between 1 OU_E/m^3 and 3 OU_E/m^3 and odor intensity higher than 3 OU_E/m^3. Black dot indicates the current state of gas mixture.

if the underestimation of the gas mixture odor intensity was not desired, e.g. in environmental applications. However, one would rather choose the other way of dividing the feature space if avoiding too strict judgment of gas mixture odor intensity was most important. In case of a single multiclass partitioning the possibility of gas mixture assessment criteria tuning is not available. We additionally showed that in this case the classification errors are higher (Table 2 to Table 4).

Based on the above findings, we proposed the concept of gas sensor system for the semi-quantitative monitoring of multi-component VOCs mixture emission decay. The system consists of the semiconductor gas sensor array and the graphical display. Commercial TGS sensors are used. They are operated in the dynamic mode. The display of the sensor system is divided into panels, as shown in Fig. 5. Each panel hosts the graph of two-dimensional feature space. Separate graphs are used to address distinct components of gas mixture with the quantitative criteria. The responses of selected sensors are used as features (dimensions of feature spaces). Best pair of sensor is chosen for each substance individually. Linear division of feature space is performed using Linear Discriminant Analysis. Mulitple two two-class divisions are applied with the priority assigned to detecting gas mixtures with higher polluting potential. The integral elements of the feature space are the decision boundaries. They represent the quantitative criteria which separate different stages of gas mixture evolution. The tested gas mixture is represented as the wandering point in the feature space on each display. The diagnosis of gas mixture state results from the combined assessment which utilizes the displayed information about all, or selected components of gas mixture, and it is based on human judgment.

5 CONCLUSIONS

In this work there was proposed the concept of gas sensor system for the semi-quantitative monitoring of the emission process decay of multi-component VOCs mixture. The principal idea for realizing the assessment was to visualize the evolution of gas mixture components against the objective, quantitative criteria. It allows for fast human judgment on the condition of the mixture as a whole, based on the visual inspection of the graphical display.

For visualization we proposed to use the two-dimensional feature spaces. They were defined for each component of gas mixture individually. Classifiers were applied in order to partition feature spaces according to the criteria which discriminate between different stages of VOCs emission process. The measurement data resulting from an ongoing measurement was displayed in the feature spaces. Its location with respect to the distinguished fragments of feature spaces was indicative for the current state of gas mixture.

Based on our investigation, the most difficult problem of sensor system design was the lack of objective criteria to decide whether the proposed division of feature space is adequate. It was shown that the criterion of lowest misclassification rate of reference gas mixture patterns was insufficient for assuring that the obtained partitioning of feature space follows the logic of gas mixture evolution. The objective criteria regarding neighborhood and sequence of regions corresponding to particular classes would be needed to evaluate the candidate feature spaces in that respect. In this work we dealt with these problems in a heuristic manner.

We think that the proposed solution may be interesting for the constructors of sensor systems addressed to environmental monitoring applications.

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