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HUMIDITY AS A DISCRIMINATIVE FACTOR IN ALCOHOLS RECOGNITION

Measurements were carried out using the gas sensor array consisting of six TGS. Responses of this device to such alcohols as methanol, ethanol, propanol and butanol were investigated. Water vapour influenced sensor responses to each alcohol. On the basis of experimental results it was possible to establish the discriminative factor. It improved the results of recognition of alcohols in humid air. Methanol and ethanol were successfully recognised in 100% cases. The percentages of correct recognition of propanol and butanol were 85% and 95%, respectively.

1. INTRODUCTION

The use of semiconducting gas sensors for environmental purposes is limited because these devices are strongly affected by an ambient moisture. Water vapour is always present under standard environmental conditions. Additionally, it may also be among the products of hydrocarbon oxidation on the semiconductor surface. The concentration of water vapour can vary within a wide range. Therefore, it is difficult to estimate the influence of humidity on the response of gas sensor.

Humidity plays an important role in sensing mechanism of semiconductor gas sensors. It is known from experimental results that water molecules can be physisorbed or chemisorbed on a metal oxide surface. These phenomena depend on a temperature. The physisorbed molecules in the form of neutral chemical species are entirely desorbed at a temperature of about 150 °C [1].

Chemisorbed water molecules, just as reducing gases, increase the electrical conductivity of the *n*-type semiconductor such as SnO_2. This effect has not been clearly explained yet. Most likely, two processes are involved:

- transfer of electrons from adsorbed water to metal oxide material,
- dissociation of water molecules and chemical reduction of oxygen lattice atoms.

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Water has an electron-donating properties, therefore the charge carriers from this donor can be injected into the conduction band of a tin oxide. This results in the decrease of electrical resistance.

The adsorbed water molecules can also dissociate on the semiconductor surface. Hydrogen atoms and hydroxyl groups are produced in this process:

$$H_2O_{ads} \rightarrow H_{ads} + OH_{ads}.$$

Hydrogen atoms are attracted to the oxide sites and reduce the atoms of oxygen lattice. The oxygen vacancies are one of the products of this chemical reaction:

$$H_{ads} + O_{lat} \rightarrow OH_{ads} + V_o.$$

The vacancies V_o can diffuse into the bulk, where they act as electron donors causing a decrease in electrical resistance. The chemisorbed hydroxyl groups are bound to metallic atoms because these sites present cationic properties. For this reason the water chemisorption on a semiconductor results in the formation of a hydroxylated surface. Hydroxylation of the metal oxide surface takes place at a relatively high temperature. The hydroxyl groups desorb at a temperature ranging from 250 up to 500 °C. They may behave like an electron donor on a semiconductor surface.

The details of SnO_2 interaction with adsorbed water were discussed by many authors. KOHL [2] has assumed that two types of OH groups are involved in this mechanism: one of them includes the atoms of oxygen lattice, and the other is bound to lattice tin. According to this assumption two reactions can be written:

$$H_2O + Sn_{lat} + O_{lat} \rightarrow (HO \cdot Sn_{lat}) + (O_{lat} \cdot H)^+ + e,$$

$$H_2O + Sn_{lat} + O_{lat} \rightarrow 2(HO \cdot Sn_{lat}) + V_o.$$

The oxygen vacancies migrate to the bulk where they are the donors of electrons. The above reactions explain the increase of electrical conductivity in tin oxide caused by water-vapour adsorption.

IONESCU [3] has proposed the hypothesis that water reacts dissociatively with one type of lattice oxygen on the SnO_2 surface, but in two different ways, giving rise to two different types of OH^- ions. One of them, i.e. OH_4, replaces the reactive oxygen sites without producing any free carriers (no change in conductance):

$$Sn^{2+}O^{2-} + H^+ + OH^- \leftrightarrow Sn^{2+}(OH_A^-)_2.$$

The other OH_B groups generate free carriers without blocking the oxygen sites:

$$2Sn^{2+}O^{2-} + H^+ + OH^- \leftrightarrow Sn^{2+}O^- + Sn^{2+}e^- + 2OH_B^-.$$

This equation leads to the formation of $Sn^{2+}O^-$ groups. They are the equivalent of free holes. The $Sn^{2+}e^-$ groups dissociate rapidly and produce electrons, which can

contribute to conduction of electricity or in oxygen chemisorption. Reaction results in the increase of conductance.

The other mechanism of water vapour interaction with SnO_2 was presented by YAMAZOE et al. [1]:

$$H_2O + O^{2-} + V_o^{n-} \rightarrow 2(OH)^- + ne^- .$$

It is also hypothesized that the changes of conductance are induced by surface charge resulting from the preferential alignment of water dipoles.

The experimental investigations show that the presence of surface hydroxyl groups limits the oxygen adsorption up to 320–340 °C even if the per cent of oxygen approaches 100 [4].

Water molecules may compete with reducing gases in the chemisorption process [5], [6], because these compounds react with the same active sites on the surface of tin oxide. This effect was observed for CO, CH_4, H_2. These gases and water vapour share the same oxygen sites. The previously chemisorbed water molecules can occupy available adsorption sites and reduce the sensitivity of the sensor, which is especially important for ambient air with high humidity levels. For example, in the case of propane and butane the sensor sensitivity decreases with increasing a concentration of water vapour [7].

Moisture in the atmosphere causes slow response of SnO_2 to NO_x [8]. Water molecules adsorbed on the semiconductor surface can act as catalyst or participate in chemical reactions. For example, a synergistic effect can be observed between CO and H_2O. Due to such an effect hydroxyl species may react with CO producing formate species ($-COOH^-$) [9]. This reaction affects the sensor response to CO.

On the surface of tin oxide ethanol behaves like CO, but the mechanism of this interaction is different. At a low humidity water molecules accelerate the dissociation of ethanol and thus the sensor sensitivity increases. As relative humidity increases, the contribution of water molecules to the conductivity of the tin oxide semiconductor is greater than that of ethanol. Therefore the sensor sensitivity to ethanol decreases.

Based on available experimental results we accept a hypothesis that the water vapour, when present in the mixtures of air and reducing gases (volatile organic compounds), can be used as a factor contributing to the diversity in a measuring process.

The aim of this work is to characterize the semiconducting gas sensor array in a wet, contaminated air in order to show that the humidity influence may be considered as a positive factor in the process of identification of volatile organic compounds in the air.

2. EXPERIMENTAL

Among the various metal oxides having gas sensing properties, tin oxide is very often applied in commercial gas sensors, first of all in detecting the leakage of reduc-

ing gases and some volatile toxic substances. This semiconductor is a widely used TGS from Figaro Engineering, Japan. We chose these sensors because of their large commercialisation, relatively low price and high sensitivity to volatile organic compounds. The following Taguchi Gas Sensors were applied in our multisensor array: TGS800, TGS822, TGS824, TGS825, TGS880 and TGS883.

The array was placed inside a glass cell. This chamber was specially designed to characterize the gas sensor properties (responses) under properly controlled physical and chemical conditions. The glass cell was connected by a teflon gas line to the equipment for the preparation of pure air and also air with different concentrations of water and organic volatile compounds. Pure air was drawn from the zero air generator (Horiba, Japan). Gas mixtures were prepared by an evaporation method. A desired amount of water and liquid volatile organic compounds was injected into a glass spiral tube (coil) using liquid chromatography syringes. Then it was vaporised in a stream of pure air. The flow rate of the air was precisely set. The prepared gas mixture was collected in a Tedlar bag. A vapour concentration of H_2O and VOCs in this mixture was determined by a dosage of these compounds and the air flow rate.

The measurements were based on the principle of a mass flow system. All tests were carried out according to the same experimental procedure. The sensors were tested through the sequential exposures. At the beginning, the stream of pure air was pumped into the test chamber. Then sensors were exposed to a flow of prepared gas mixture from the Tedlar bag. The time of exposure was 5 minutes. Finally, a constant dry air flow was applied to clean the chamber and the array. The gases were pumped into the measuring system continuously at a constant flow rate of 100 dm^3/h.

Electrical measurements were carried out by means of a simple equipment containing a reference resistor and digital multimeter. Each sensor was connected to the reference resistor. The changes of its voltage during exposures of the array to reducing gases were treated as the response of the sensor. The voltage was measured by a digital multimeter.

The experiment was performed with the measuring system presented. Four alcohols were selected for the analysis: methanol, ethanol, propanol and butanol. These were homologous chemical compounds. Sensor responses to different concentrations of selected alcohols in humid air were gathered. Several humidity levels were investigated, as in a real atmosphere the humidity has constantly changed. The experimental ranges of the concentrations of alcohols and water vapour were presented in table 1.

Table 1

The concentration ranges of alcohols and water vapour measured by the sensor array

Analyte	methanol	ethanol	propanol	butanol	water vapour
Concentration range [mg/m^3]	32–238	32–237	32–241	32–243	$(4–20)\cdot10^3$

The concentrations of alcohols investigated ranged from ca. 30 mg/m^3 to 240 mg/m^3. In the case of water vapour, the concentration range was from 4 to 20 g/m^3, which corresponded to the relative air humidity between 20% and 100%, at 25 °C.

The sensor response was defined as the ratio of sensor response to measured analyte and its response to the reference concentration of ethanol vapour. The sensor response to the alcohol in humid air is denoted by $R_k^{i,j}$, where:

k – the sensor number,
i = 1...5 – gas sensors: TGS800, TGS822, TGS824, TGS825, TGS880,
k = 6 – humidity sensor: TGS883,
i – the alcohol concentration, $i = a1, ..., am$,
j – the water vapour concentration, $j = w1, ..., wm$.

3. RESULTS AND DISCUSSION

3.1. PRELIMINARY ANALYSIS OF SENSOR ARRAY RESPONSES TO ALCOHOL AND WATER VAPOUR

The influence of alcohols and water vapour on sensor array responses was investigated within the predefined concentration range. To analyse the alcohol influence, the responses of sensors were plotted against alcohol concentrations for a constant humidity level. The methanol example was shown in figure 1.

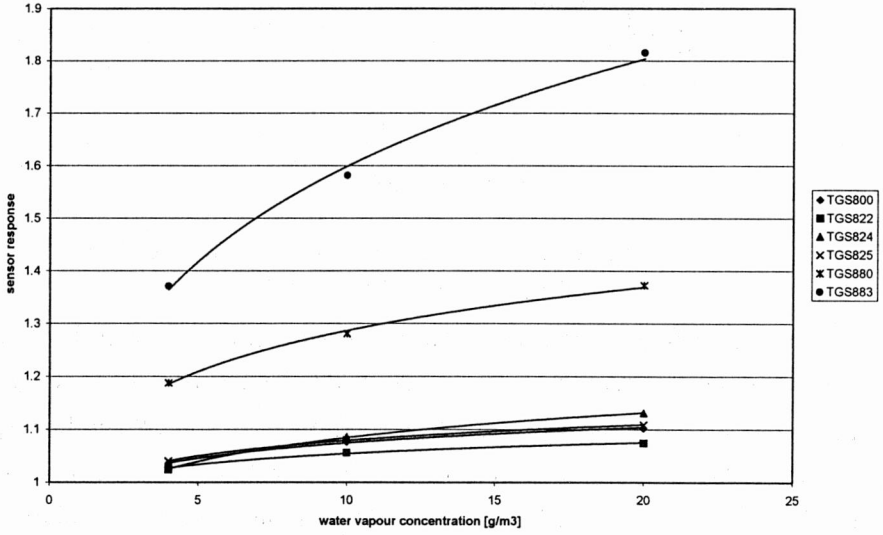

Fig. 1. Relationship between sensor responses and methanol concentrations at water vapour concentration of 4 g/m^3

The change in the minimum $a1$ and maximum am alcohol concentrations caused significant changes ΔR_1^j, ΔR_2^j, ..., ΔR_5^j in gas sensors responses (figure 1). Simultaneously, it was responsible for relatively small change ΔR_6^j in humidity sensor response. The mean value of ratios $\Delta R_1^j/\Delta R_6^j$, $\Delta R_2^j/\Delta R_6^j$, ..., $\Delta R_5^j/\Delta R_6^j$ was equal to 4 for all alcohols being analysed. This meant that on an average a gas sensor responded four times stronger to the change in alcohol concentration than the humidity sensor did. Actually the ratio ranged from 1.2 to 10. These findings were valid in our experimental range of analytes' concentrations.

The influence of water vapour on sensor array responses was also analysed in such a way that responses of sensors were plotted against water vapour concentrations for a constant alcohol concentration. The example for methanol was presented in figure 2.

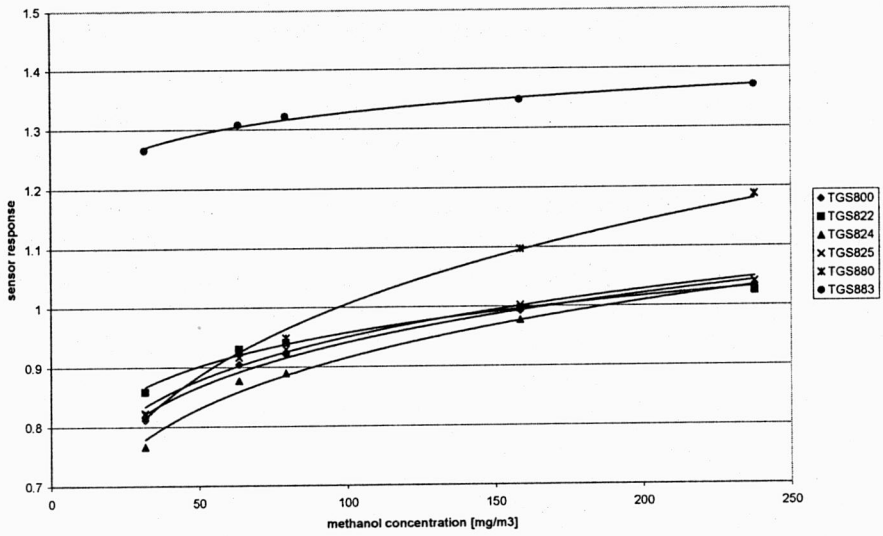

Fig. 2. Relationship between sensor responses and water vapour concentrations at methanol concentration of 238 mg/m^3

It can be seen in figure 2 that the change in the minimum $w1$ and the maximum wm water vapour concentrations caused a significant change ΔR_6^i in humidity sensor response. It also induced relatively small changes ΔR_1^i, ΔR_2^i, ..., ΔR_5^i in the responses of gas sensors. The mean value of ratios $\Delta R_6^i/\Delta R_1^i$, $\Delta R_6^i/\Delta R_2^i$, ..., $\Delta R_6^i/\Delta R_5^i$ was 4 for all alcohols being analysed. This meant that on an average a humidity sensor responded four times stronger to an alcohol concentration change than a gas sensor did. Actually the ratio ranged from 1.7 to 10. These findings were true in our experimental range of analytes' concentrations.

Each sensor in the array exhibited only partial selectivity with respect to chemical compounds, whose selectivity it measured. This investigation showed that both gas sensors and humidity sensor responded to alcohols as well as to water vapour. They only differed in the response intensity: gas sensors better responded to alcohols, while the humidity sensor stronger responded to water vapour.

As water vapour modified responses of gas sensors to alcohols and alcohols modified the response of humidity sensor to water vapour, this could become a valuable factor in the alcohol discrimination task. In order to take advantage of this factor, when solving the alcohol recognition problem, pattern recognition methods could be useful.

3.2. PATTERN RECOGNITION METHOD

Pattern recognition methods based on sensor array measurements are widely used for recognition of VOCs. Principal Components Analysis (PCA) [10]–[14], Discriminant Analysis (DA) [12], [15], different types of neural networks, e.g., multilayer perceptron [10], [11], Radial Base Function (RBF) networks [11], [16], Kohonen Maps [11] and also combined neural-fuzzy approach are most frequently used [17], [18].

The probabilistic neural network was used as a pattern recognition method in the analysis presented [19]. This type of RBF neural network may be used as a classifier in a correct recognition of the class, to which the input set of data belongs. The fractioning of input space is conducted in the course of supervised learning process. In that stage of network development, the value being assigned to the whole class of input sets is presented to the network as its expected output. Concurrently an input set of data is fed into the network, which belongs to this class. This is the task of a network teaching algorithm designed to calculate the neurone weights. Such a configuration of weights should allow a transformation of any input vector into an adequate value of the network output. Such presentation and calculation procedure is carried out for the whole set of input data. When the learning phase is finished, the network performance is tested upon new, unknown data vectors. The condition that values comprised in test sets are within the range of values included in learning data sets must be imposed. This way, the model ability to generalise in the whole input space is checked.

The classification task was to recognise each of four alcohols (methanol, ethanol, propanol and butanol) in humid air, irrespective of an alcohol concentration. To proceed with solving the problem, four pattern recognition models were built. Each model served for the recognition of one alcohol.

A set of about 100 input vectors was used for constructing the recognition models. The whole set consisted of four equal parts as there were four alcohols to recognise. In a learning phase, the network was presented with all but one input sets and all but one adequate network responses (1 – for the alcohol in question, 2 – for the remaining alcohols). The best conditions were settled for network generalisation ability by using a maximum possible number of input sets for network teaching. When the network

weights were calculated, the model was tested on a single input set, which was previously excluded from the whole teaching set. The procedure of network teaching and testing was repeated for each input data set. All necessary programming and simulations were done with the MATLAB software [19].

3.3. SENSOR ARRAY PATTERN OF ALCOHOL IN HUMID AIR

The recognition of alcohols was based on sensor array measurement data. These were arranged in data sets, which were then used as an input into pattern recognition models. We were aiming not only at selecting the data to be included in input sets, but also at obtaining the best possible alcohol recognition results. It has been already noticed that utilising water vapour influence on sensor array response to alcohols may be useful in that respect.

The most simple data set consisted of sensor array responses to any alcohol concentration (i) and any water vapour concentration (j) used in the experiment. This data set could be denoted in the following way:

$$I_a^{i,j} = \{R_1^{i,j} \; R_2^{i,j} \; R_3^{i,j} \; R_4^{i,j} \; R_5^{i,j} \; R_6^{i,j}\}, \tag{1}$$

where:

i – the alcohol concentration, $i = a1,..., am$,

j – the water vapour concentration, $j = w1,..., wm$.

A number of $I_a^{i,j}$ data sets were prepared according to equation (1) for the whole experimental range of the analytes' concentrations. They were used as the input sets for alcohol recognition models. Models were developed according to the procedure described in section 3.2. Finally, the performance of models was checked. The percentage of all data sets characterising the alcohol being correctly recognised was used as the indicator of model performance. The results of alcohol recognition were presented in table 2.

Table 2

Performance of a neural model recognising alcohols in humid air using $I_a^{i,j}$ input sets

Analyte	methanol	ethanol	propanol	butanol
Recognition rate [%]	67	53	20	33

The results presented in table 2 proved that alcohols were not successfully recognised, when sensor array response to any concentration of alcohol or water vapour was used as an input into the model. Recognition results for propanol and butanol were extremely confusing, i.e. 20% and 33%, respectively. Neural network was not able to derive patterns typical of those alcohols from such input sets at all. Methanol and ethanol were more recognisable for the network. Nevertheless, a correct recognition of 67% cases for methanol and 55% cases for ethanol was not acceptable.

The results obtained encouraged the development of another form of data set to be used as an input into the alcohol recognition model. We searched for the method that allowed us to treat a water vapour as a positive factor, which could enhance alcohol recognition. The final proposal was to build an input set consisting of sensor array responses, when the alcohol concentration was constant but the water vapour concentration was variable in a full experimental range of $w1$ ... wm (table 1). Adopting this approach, the alcohol should be recognised based on the following set of measuring data:

$$I_b^i = \{R_1^i \;\; R_2^i \;\; R_3^i \;\; R_4^i \;\; R_5^i \;\; R_6^i\}, \tag{2}$$

where:

$R_1^i = \{R_1^{i,w1} \;\; R_1^{i,w2} \;\; \ldots \;\; R_1^{i,wm}\}$,

$R_2^i = \{R_2^{i,w1} \;\; R_2^{i,w2} \;\; \ldots \;\; R_2^{i,wm}\}$,

...

$R_6^i = \{R_6^{i,w1} \;\; R_6^{i,w2} \;\; \ldots \;\; R_6^{i,wm}\}$,

i – the alcohol concentration, $i = a1,\ldots, am$,

$w1$ – minimum concentration of water vapour,

wm – maximum concentration of water vapour.

According to formula (2), each input set I_b^i represented sensory characteristics of alcohol in humid air at a fixed alcohol concentration (i). It has been noticed that the characteristic was stable for a particular alcohol. In our experimental range, its shape was almost independent of the alcohol concentration. Therefore the I_b type data set was proposed as a sensor array pattern of alcohol in a humid air.

The blueprint of alcohol in a humid air was derived based on the pattern proposed. To this end, the following indicator was used:

$$W_k^i(\text{alcohol}) = \frac{R_k^{i,wm} - R_k^{i,w1}}{R_6^{i,wm} - R_6^{i,w1}}, \tag{3}$$

where:

$R_k^{i,wm}$ – the response of the k-th gas sensor to the i-th concentration of alcohol and maximum concentration wm of water vapour,

$R_k^{i,w1}$ – the response of the k-th gas sensor to the i-th concentration of alcohol and minimum concentration $w1$ of water vapour,

$R_6^{i,wm}$ – the humidity sensor response to the i-th concentration of alcohol and maximum concentration wm of water vapour,

$R_6^{i,w1}$ – the humidity sensor response to the i-th concentration of alcohol and maximum concentration $w1$ of water vapour,

i – the alcohol concentration, $i = a1$ ... am,

k – the sensor number, $k = 1$ ... 5,

alcohol – methanol, ethanol, propanol, butanol.

The indicator (equation (3)) represents the ratio of each gas sensor response change to the humidity sensor response change at a fixed alcohol concentration and a humidity level changing from the minimum value $w1$ to the maximum value wm. Values of indicator were calculated for all gas sensors and for all alcohols at each experimental alcohol concentration.

As indicated before, the humidity sensor poorly responded to the change in alcohol concentration compared to the gas sensor response. For this reason, the humidity sensor was a valuable point of reference in characterising alcohol in humid air within a full range of alcohol concentrations.

Radial plots were used to present blueprints of alcohols in humid air. One plot was used for one alcohol. Each axis on a plot represented the indicator for one gas sensor in sensor array. The values of the indicator W_k^i(alcohol) were marked on each axis for all measured concentrations of alcohol. The points on all axes representing indicators calculated for the same alcohol concentration were shown in the form of line passing through all the points. Consequently, a set of lines was obtained. An exemplary plot for methanol was presented in figure 3. Three concentrations were chosen to show the scheme of plot construction.

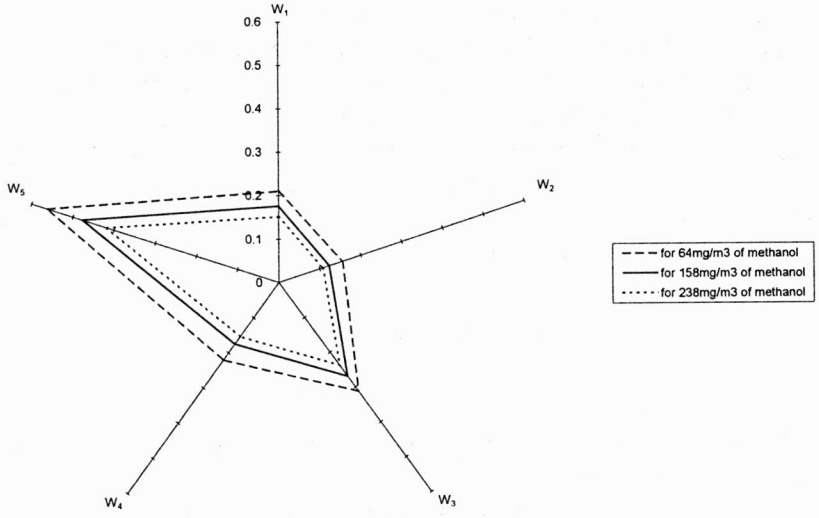

Fig. 3. Radar plot of the W (methanol) indicator at three selected concentrations of methanol in humid air

Further, only the outer line corresponding to maximum values of the indicator and the inner line corresponding to minimum values of the indicator were remained. The area between those two lines represented the space which comprised all indicator values calculated for the full range of our experimental conditions. In this area, all

lines (figure 3) could correspond to the entire range of alcohol concentrations. The figure obtained was considered as the blueprint of an alcohol in the sensor array. Blueprints of methanol, ethanol, propanol and butanol were presented in figures 4–7.

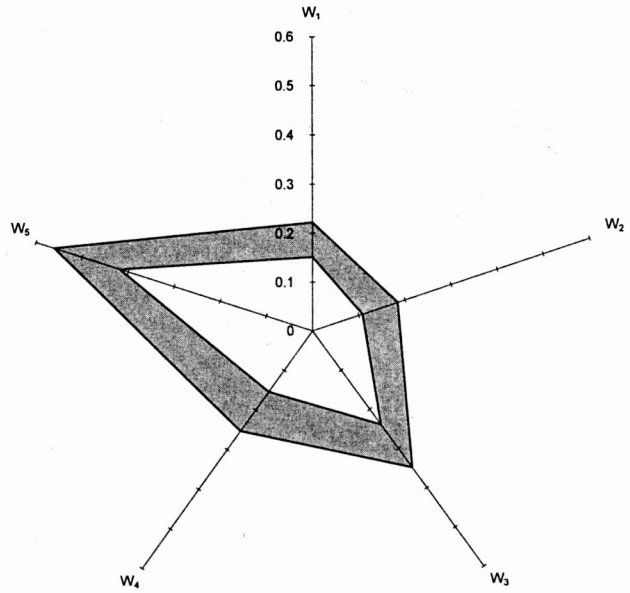

Fig. 4. The blueprint of methanol at *W* (methanol) indicator

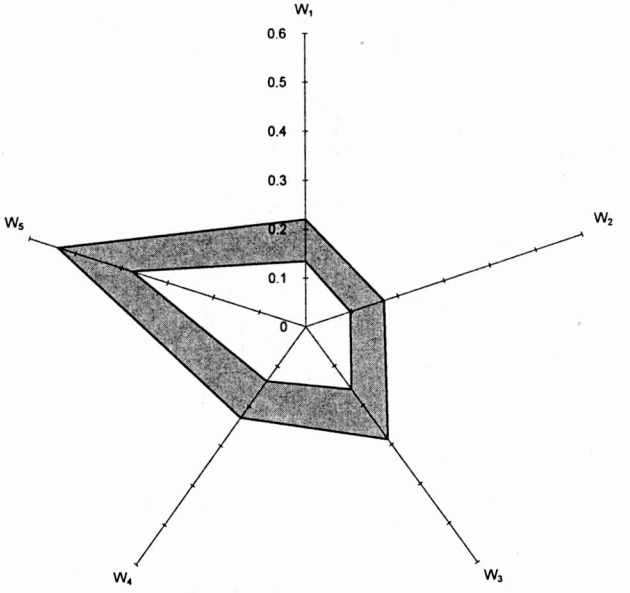

Fig. 5. The blueprint of ethanol at *W* (ethanol) indicator

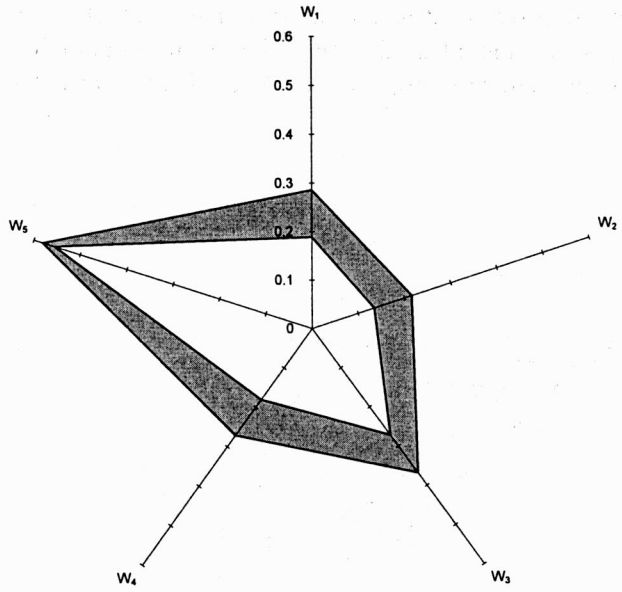

Fig. 6. The blueprint of propanol at *W* (propanol) indicator

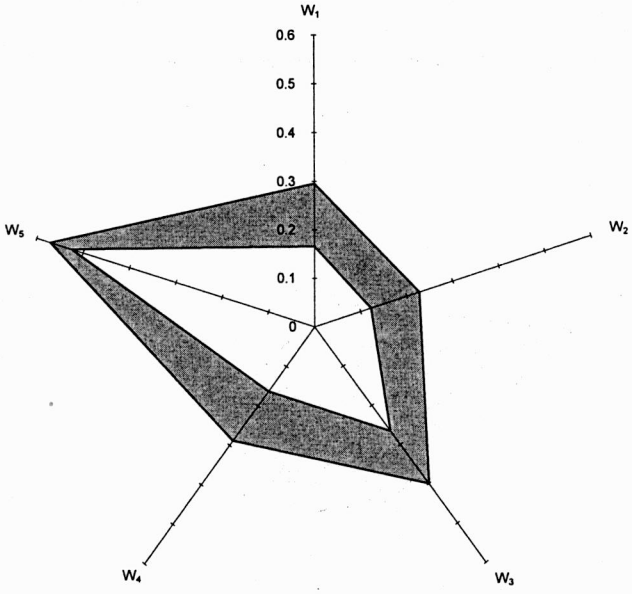

Fig. 7. The blueprint of butanol at *W* (butanol) indicator

One could analyse the blueprints in figures 4–7 with respect to their shape and/or size. The shapes of blueprints prove that the alcohols are very similar in the following pairs: methanol–ethanol and propanol–butanol. After taking into account the size of the blueprints, the difference between methanol and ethanol is clear. The sizes of the blueprints of propanol and butanol are almost the same. Propanol and butanol blueprints were almost identical as far as their shapes and sizes are considered.

As a new form of the input data set I_b^i was proposed, representing the pattern of alcohol in humid air, the recognition of alcohols in humid air could proceed. Models were developed according to the procedure described in section 3.3. Finally, performance of the models was checked. The percentage of all data sets characterising alcohol, which were correctly recognised, was used as the indicator of model performance. The results of alcohol recognition were presented in table 3.

Table 3

Performance of a neural model recognising alcohols in humid air using I_b^i input sets

Analyte	methanol	ethanol	propanol	butanol
Recognition rate [%]	100	100	85	95

The results in table 3 testify to a high performance of the pattern recognition approach being applied. Methanol and ethanol were always correctly recognised (100%). Very high recognition threshold was also reached for propanol (85%) and butanol (95%). Beyond any doubt, this result was a success. Propanol and butanol were unrecognisable when different type of input set was used (table 2).

One could try to identify the links between the recognition results presented in table 2 and alcohols blueprints shown in figures 4–7. Indeed, it could be noticed that an extremely close similarity of propanol and butanol blueprints corresponded to incomplete recognition success of those alcohols. On the other hand, a clear difference in the sizes of methanol and ethanol blueprints agreed with a 100% correct recognition by the model.

4. CONCLUSIONS

Based on the measurements of partially selective gas sensors' array we may conclude that water vapour can be a positive factor improving recognition of volatile organic compounds.

The preliminary analysis of measurement results for methanol, ethanol, propanol and butanol in humid air proved that both gas sensors and humidity sensor responded to alcohols and to water vapour. So the humidity influenced a pattern, which could be derived from sensor array measurement results, for each alcohol.

Taking account of this fact, a particular approach was proposed to the construction of data set, which was then used in the model for alcohol recognition. This set consisted of sensor array responses when the alcohol concentration was constant but the water vapour concentration changed within a full experimental range. This way, the humidity characteristics of alcohol in sensor array was proposed as a pattern of alcohol in a humid air.

The results of recognition of alcohols in humid air, based on humidity characteristics, were impressive. The neural models developed recognised propanol in 85% cases, and butanol – in 95% cases. The efficiency of methanol and ethanol recognition was 100%.

This result was very spectacular compared to the results of recognition that were based on more simple input data set consisting of sensor array responses to any alcohol concentration and any water vapour concentration in a full experimental range. In that case, the recognition results for propanol and butanol were extremely unsatisfactory (20% and 33%, respectively). Also the recognition results for methanol (67%) and for ethanol (55%) were not acceptable.

The success of proposed pattern recognition approach proved that water vapour could be a factor improving recognition of volatile organic compounds. A set of sensor array responses to a constant concentration of alcohol and a variable concentration of water vapour was proposed as a possible identifier of alcohol in humid air.

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WILGOTNOŚĆ JAKO CZYNNIK WYRÓŻNIAJĄCY W ROZPOZNAWANIU ALKOHOLI

Przeprowadzono pomiary za pomocą matrycy czujnikowej składającej się z sześciu czujników TGS. Przeanalizowano odpowiedzi matrycy na pary alkoholi: metanolu, etanolu, propanolu i butanolu w powietrzu. Odpowiedzi czujników na pary alkoholi zależą od wilgotności powietrza. Korzystając z tego, zaproponowano wzorce par alkoholi w matrycy czujników na podstawie wyników pomiarów par alkoholi w powietrzu wilgotnym. Wyróżnione wzorce umożliwiły poprawę wyników rozpoznawania alkoholi za pomocą matrycy czujników. Dzięki ich zastosowaniu uzyskano 100% skuteczność rozpoznawania metanolu i etanolu. Propanol i butanol były rozpoznawane odpowiednio w 85% i 95% przypadków.