Nowadays development of new technologies in various food freshness testing devices has a high potential and is one of the main objectives securing public health. Meat freshness evaluation becomes of paramount importance due to the growing consumer awareness of food quality and safety (Tománková et al. 2012, Rukchon et al. 2014; Ivanov et al. 2015).

In general, consumers are interested in healthy food with easy way to check its freshness. Chicken meat is the most popular kind of meat for consumers for its price, good taste, quick and easy preparation. However, chicken meat is highly susceptible to storage conditions and contamination that can cause meat spoilage (Šimoniová et al. 2013).

The rapid, non-invasive, reagentless, relatively inexpensive method with a possibility to use it everywhere would be an ideal method for the veterinary authorities that determine freshness of chicken meat on the spot or for consumers to check meat freshness by themselves. E-nose technology is an optimal solution for such a purpose that has been widely used in food analysis in recent years. It is proved that e-nose technology is a fast, simple, non-expensive and non-destructive method of food assessment and quality control (Gorska-Hdrczyczak et al. 2016). Compared with the analytical chemical and microbiological methods, e-nose technology offers an alternative approach to volatile compound detection in a rapid way.

The use of e-nose technology or various sensors has been proved to be developed and applied in various fields of life, such as food (Peris et al. 2009; Loutfi et al. 2015; Raudienė et al. 2017), environmental monitoring (Dentoni et al. 2012; Capelli et al. 2014) and medicine (de Heer et al. 2016). Application of e-nose technology to various meats is one of the main application areas in the food industry (Barbri et al. 2008; Hong et al. 2012; Dissing et al. 2013; Zohora et al. 2013). MOS sensors are widely used for food quality assessment (Sliwinska et al. 2014, Baietto
Moreover, these sensors are perhaps the commonly used gas sensors in the field of e-nose technology (Berna 2010). They have been more extensively used to make arrays for odour measurement than any other types of gas sensors (Zohora et al. 2013; Loutfi et al. 2015). The quality indicators and microbial spoilage of fresh chicken meat have been analysed by other authors (Song et al. 2013; Gorska-Hdrczyczak et al. 2016). Studies showed wide e-nose applicability in the market and demonstrated their potentially large possibilities in industrial practice. Nevertheless, there is a lack of information about the effective application of e-nose in daily consumer life. Furthermore, usually e-noses that are intended for chicken meat freshness detection have 8 or even more MOS sensors in their construction. Such devices are too expensive for the users in real life for rapid control of meat freshness.

Therefore, the goal of this work was (i) to develop a simple and low-cost customized e-nose system with specific sensors for rapid fresh chicken meat freshness detection and (ii) to determine VFA amount for spoilage detection of fresh chicken meat using traditional chemical analysis with the purpose to evaluate a correlation between the regulated concentration of volatile substances and measured parameters by MOS sensors.

In the present study, we propose a new and fast method for determination of chicken meat freshness, which analyses volatile compounds and gases in the meat headspace by using a portable e-nose. The results of this research can have practical application, which is the main advantage of this article.

MATERIAL AND METHODS

Sample preparation. One kind of meat samples (chicken) was used for this study. Fresh chicken samples were purchased from different providers: freshly processed chicken meat was brought directly from the farmer and it was also purchased from 4 different supermarkets. With the purpose to evaluate the response of MOS sensors to specific chicken odour, one of the test samples was the meat of yellow chicken that was fed maize (maize-fed chicken). A specific breeding method affects the taste, colour and odour of this type of chicken meat.

All meat samples for analysis were prepared by deboning and mincing carcass meat. Chemical composition (amount of proteins, minerals, fats) and moisture were measured in all test samples.

Moisture content was determined by drying samples at 105°C to a constant weight according to ISO 1442:1997. Nitrogen content was determined by the Kjeldahl method, according to ISO 937:1978. Total lipids were extracted from the samples using chloroform as solvent according to ISO 1444:1996. Total mineral content was determined by drying test samples at 500–600°C to a constant weight according to ISO 936:1998. The carbohydrate content was calculated. The pH value was determined by placing an N 1048A pH probe directly into homogenized samples (WTW 3110 pH-meter; WTW GmbH, Germany) and the electromotive force was measured. Calibration of used electrodes was performed at room temperature using phosphate buffers of pH 4 and 7. VFA content was determined by the steam distillation of an acidified aqueous extract of the samples in a Behr S 4 fully automatic steam distillation apparatus (Behr labor technik, Germany) and then by titrating the distillate.

Chroma Meter CR-400/410 (Konica Minolta, Japan) equipped with an aperture of 8 mm in diameter and standard illuminant D50 at a standard observation angle of 10° was used for the colour analysis of chicken meat samples. Colour was evaluated using the Commission Internationale de l’Eclairage (CIE) \(L^*\) (lightness), \(a^*\) (redness), and \(b^*\) (yellowness) system, where \(L^*\) is the intensity of colour (0 – absolutely black and 100 – absolutely white), \(a^*\) is the value from –60 (absolutely green) up to +60 (absolutely red), \(b^*\) is the value from –60 (absolutely blue) up to +60 (absolutely yellow). The Chroma Meter was calibrated with white plate before every measurement.

Samples were specially aged for experiments. They were stored in a fridge at a temperature of +4°C.
Electronic nose system. A new laboratory electronic nose prototype with a new generation MOS sensor system was created for meat freshness evaluation. Various sensors were used and tested in the research process with the purpose to evaluate and choose the best MOS sensor system. The selection of MOS sensors was based on chemical specificity and sensitivity. To develop a simple and low-cost customized electronic nose system it was very important to select parameters including size, cost and power consumption.

No MOS sensors were commercially available in the market for detection of specific volatiles that are released during chicken meat spoilage. So the complex of MOS sensors (that are sensitive to some volatile compounds) was selected for research.

A series of the experiments was started with the sensor array that was composed of three MOS sensors (SGX Sensortech, Switzerland) and two additional sensors (Figure 1).

The signals of additionally integrated sensors complement information of the main array and enable data correction of MOS sensors. Main characteristics of MOS sensors are given in Table 1.

Photo of the e-nose prototype with USB modification is presented in Figure 2 and software window for the processing of e-nose signals is presented in Figure 3.

MOS gas sensors are expected to be the best detectors of volatile organic compounds that are released during the meat spoilage process. The exact CH and NH$_3$ sensors were chosen as main sensors for the e-nose prototype. According to manufacturers’ declared characteristics, MOS sensors are mostly intended for exact gas detection and/or concentration measurement (for example CH sensor for CO gas detection and NH$_3$ sensor for NH$_3$ gas detection) but also they can fix other types of gases less or more. The fixation of other gases depends on the sensor coating sensitivity and its working range. O$_3$ sensor was added as a supplementary sensor that could extend usage and application of new generation MOS sensors for meat freshness evaluation.

The response of output signals of e-nose sensors was tested and fixed in critical concentration ranges: after a sudden increase in the concentration of the ‘sniffed’ gas mixture (Figure 4A) and after a sudden decrease in the concentration of the ‘sniffed’ gas mixture (Figure 4B). Both figures illustrate this response by voltage signal.

Figure 4A and B show typical curves of the used sensors that give an expressive response to chicken meat sample measurement. Each curve represents the sensor voltage (U) which increased in the first few seconds and stabilized in different time for each sensor: CH sensor at about 25$^{th}$ s, NH$_3$ sensor at about 300$^{th}$ s and O$_3$ sensor at about 180$^{th}$ s (Figure 4A). The time till the sensor stability phase was monitored. When the stabilization phase ends, the measurement phase starts.

After the period when the measurement result is fixed, the signals of sensors were restarted, i.e. come back to their baseline value. Right after that a new measurement can be started. The time needful to return to the baseline is different for every sensor: CH sensor reaches the baseline in approx. 35 s, NH$_3$ and O$_3$ – in approx. 500 s (Figure 4B). Given results show that CH sensor has higher sensitivity and significantly shorter stabilization duration than the other two sensors.

Table 1. Parameters of sensors

<table>
<thead>
<tr>
<th>Sensor number</th>
<th>MOS sensor</th>
<th>Substances for sensing</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>CH</td>
<td>hydrogen, ethanol, methane, propane, isobutane, ammonia, carbon monoxide, hydrogen sulfide</td>
</tr>
<tr>
<td>2</td>
<td>NH$_3$</td>
<td>ammonia, ethanol, propane, iso-butane, hydrogen</td>
</tr>
<tr>
<td>3</td>
<td>O$_3$</td>
<td>trioxysgen</td>
</tr>
</tbody>
</table>
RESULTS AND DISCUSSION

VFA measurement. For the next series of experiments the meat spoilage process was slowed down to enable better understanding and deeper analysis of the meat spoilage process. For this purpose, the samples were stored in a fridge at a temperature of +4°C. The variations of VFA in chicken meat during the aging process were measured by traditional chemical analysis method and results are given in Figure 5A.

Results showed that both meat samples remained fresh for the first 4 days and only after this period the spoilage of thigh meat was detected, i.e. an amount of VFA began to increase. Minced chicken breast meat (that is less fatty) began to spoil after 5 aging days and it was totally spoiled one day later than the thigh meat.

pH value. In parallel to VFA concentration measurement and analysis of MOS sensor signals, additional pH value measurements of meat samples were performed. pH value is one of the factors that characterize meat freshness. The obtained initial pH values of 6.02 and 6.21 for chicken breast and chicken thigh, respectively, fall into the limits of 5.6 and 6.2, which indicate that the product is fresh in accordance with Lithuanian legislative norms.

pH value was measured every day during all aging period. It was stated that pH of chicken meat increased in time.

Although the pH value should be one of the parameters for meat freshness evaluation, results did not give any obvious and reliable information about the spoilage process (Figure 5B).

There was no significant pH correlation with the signals of sensors. So the pH value is a contentious fact as a meat freshness indicator because its value varies depending on many factors (Duclos & Bihan-Duval 2007).

Colour analysis. Pigments oxidize during the meat storage process that causes a meat colour change. So the change of $L^*a^*b^*$ indicators was evaluated that characterizes variations of aging meat colour. Results of this analysis are given in Table 2.
Colour analysis of control samples did not give any significant tendencies analysing data of colour indicators: \( L^* \) (intensity of colour), \( a^* \) (redness) and \( b^* \) (yellowness). There was not noticed any correlation between \( L^*a^*b^* \) parameters and signals of the used MOS sensors.

**VFA and sensor signals correlation.** Main experiments with all meat samples were performed every 24 hours. VFA concentration was measured and compared with results of sensor signals (samples were stored in a fridge in at a temperature of +4°C).

Relations between the responses of CH, NH\(_3\), and O\(_3\) e-nose sensors and VFA concentration (equation that describes this process and reliability coefficient) during chicken meat aging are given in Figure 6. Performed experiments (with chosen CH, NH\(_3\) and O\(_3\) sensors) confirmed that the best results were gained using CH sensor.

In accordance with Lithuanian legislative norms (the legal act of the minister of Ministry of Agriculture of the Republic of Lithuania concerning the confirmation of technical regulation of meat and poultry freshness evaluation, No. 106-4772:2002) considering VFA concentration and evaluating signals of the used sensors, obtained curves (Figure 6) were distributed into 3 zones where the final result was defined as: meat is ‘fresh’, ‘unsafe’ or ‘spoiled’.

The highest correlation was observed between CH sensor output signals in the fresh chicken meat samples measured by e-nose and VFA concentration values measured using a traditional chemical method (\( R^2 = 0.89 \)) as shown in Figure 6. As the initial variable space, namely CH, NH\(_3\), and O\(_3\), was small, no data compression techniques like principal component analysis were adopted. Besides

<table>
<thead>
<tr>
<th>Storage day</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>L*</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>breast</td>
<td>56.07 ± 0.91</td>
<td>55.43 ± 0.67</td>
<td>63.15 ± 0.57</td>
<td>59.63 ± 0.67</td>
<td>60.22 ± 0.50</td>
<td>59.18 ± 0.53</td>
<td>58.84 ± 0.71</td>
</tr>
<tr>
<td>thigh</td>
<td>55.87 ± 0.55</td>
<td>56.70 ± 1.15</td>
<td>57.96 ± 0.72</td>
<td>58.21 ± 0.50</td>
<td>57.44 ± 0.66</td>
<td>57.64 ± 0.55</td>
<td></td>
</tr>
<tr>
<td>a*</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>breast</td>
<td>11.27 ± 0.60</td>
<td>10.17 ± 0.28</td>
<td>10.96 ± 0.53</td>
<td>10.18 ± 0.47</td>
<td>11.48 ± 0.18</td>
<td>12.25 ± 0.35</td>
<td>9.32 ± 0.25</td>
</tr>
<tr>
<td>thigh</td>
<td>16.75 ± 0.31</td>
<td>17.71 ± 0.29</td>
<td>17.68 ± 0.34</td>
<td>18.31 ± 0.23</td>
<td>19.27 ± 0.17</td>
<td>18.79 ± 0.27</td>
<td></td>
</tr>
<tr>
<td>b*</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>breast</td>
<td>12.17 ± 0.63</td>
<td>11.83 ± 0.27</td>
<td>13.44 ± 0.34</td>
<td>12.73 ± 0.36</td>
<td>14.61 ± 0.17</td>
<td>13.91 ± 0.33</td>
<td>14.92 ± 0.31</td>
</tr>
<tr>
<td>thigh</td>
<td>11.67 ± 0.19</td>
<td>11.01 ± 0.25</td>
<td>12.21 ± 0.28</td>
<td>11.86 ± 0.10</td>
<td>13.52 ± 0.17</td>
<td>13.61 ± 0.26</td>
<td></td>
</tr>
</tbody>
</table>

Table 2. Colour parameters characterizing the boneless meat colour change during its storage/aging

![Graphs](image)
it was decided to omit O_3 sensor data from statistical data analysis and the variable space was reduced to 2. Data mining involved linear discriminant analysis (LDA) using the raw data (measured voltages) of CH and NH_3 sensors. As the number of classes was known a priori, namely fresh, unsafe and spoiled, with reference to VFA concentration, this number was passed to LDA as an input parameter for the classification model building. Figure 7 presents the data scatter plot in CH-NH_3 variable space divided into three distinct classes: 1–'fresh', 2–'unsafe', 3–'spoiled'.

The sensor signals at different freshness groups according to the VFA level of meat are well separated. It is seen from the figure that several outliers exist in all groups. However, the achieved overall classification error rate was 12%.

CONCLUSIONS

The main contribution of this paper was to develop a low-cost customized e-nose, which can help the user to quickly determine chicken meat freshness. It could be modified for freshness detection of other kind of meat.

A high correlation ($R^2 = 0.89$) was achieved between developed e-nose signals and VFA concentrations determined by the traditional chemical method. These results prove that the developed e-nose has a potential for assessing fresh chicken meat freshness.

The performed study demonstrated that the created e-nose, equipped with at least two sensors involving NH_3, CH, was suitable for monitoring quality changes occurring during storage of fresh chicken meat.

The created e-nose prototype has an absolute advantage as a tool for the cheap and rapid detection and assessment of meat freshness compared to time-consuming and expensive chemical/microbiological methods.

The results of this research can have practical application, which is the main advantage of this article.

References


Received: 2017–11–14
Accepted after corrections: 2018–08–24
Published online: 2018–10–24