Odour Detection Method of Silk Products Based on Electronic Nose Technology

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Silk is a natural animal protein with special odour. Polyester yarns, similar in appearance to silk, are often used in silk production. Electronic nose (e-nose) technology can distinguish different gases by different pattern recognition methods. In this paper, e-nose technology was applied to obtain data sets of silk/polyester yarns with different mixing ratios, and cluster analysis was carried out for silk samples. The experimental results show that the results of e-nose response are related to the headspace, sample quality, and headspace generation time etc.; as the response time increases, the relative standard deviation of each sensor turns to be smaller, and the response value is more stable. There are significant differences in the fluctuations between the ten metal sensors, and different samples have significant effects on the response of sensors No. 2, 7, and 9. The e-nose sensor has a higher prediction accuracy when identifying different ratios of silk/polyester yarns at the accuracy rate up to 90.286%.

1. Introduction

In recent years, silk products have become more and more popular among people, and their sales volume has been increasing year by year. The production and sales of silk quilts in the world have achieved unprecedented development. Silk products are ranked as the fastest growing products among Chinese textiles with an annual increase of 20% (Ghosh et al., 2016). Compared with cotton, down feather or chemical fibre products sold on the market, silk is made of natural silk fibre, which has the advantages such as excellent health care and improved sleep quality.

In 2017, China's General Administration of Quality Supervision, Inspection and Quarantine conducted a random inspection of 180 batches of silk products from 12 provinces, with the pass rate of sampling inspection less than 50%, because many manufacturers used tussah silk or polyester to impersonate mulberry silk, which seriously infringed consumers' rights (Wilson, 2012). Conventional silk detection methods include sensory identification, burning, chemical dissolution, microscopic observation, and spectroscopic analysis etc. (De et al., 2011, Hu et al., 2008). These detection methods are more affected by human factors, and it is difficult to distinguish correctly for products of the same variety, and organic solvents may be poisoned by chemical methods (Peris & Escuder-Gilabert, 2016, Kadri and Mouss, 2017, Zhang et al., 2018).

The e-nose is an olfactory instrument that mimics the olfactory function of the mammal's nose. The entire monitoring system generally includes the gas sensor (Hu, 2018) array, signal processing system, and pattern recognition system (Herrero et al., 2016; Borisova et al., 2017). The e-nose generates a response signal by adsorbing gas molecules through the gas sensor array during operation, thereby identifying and detecting gas components. E-nose technology has been widely used in food, chemical and medical fields (Wilson, 2012). In this paper, the data collection of silk/polyester yarn with different mixing ratios was obtained by e-nose technology, and cluster analysis was conducted for the silk samples, to explore the suitable e-nose test conditions beneficial to distinguish different proportions of silk/polyester silk samples.

2. Experimental materials and methods

The relevant data analysis of one complete e-nose sensor includes acquisition of response data, preliminary pre-processing of data, dimensionality reduction of data, modelling and decision analysis (Chen et al., 2013,
Wilson, 2013). To eliminate the influence of dimension or magnitude on the data analysis of e-nose sensors, sample data must be standardized (Jiang et al., 2015). The experimental materials were selected from the mulberry silk of Zhejiang Tongxiang and the polyester yarn produced by a chemical fibre company of Nantong. From the appearance, polyester yarn is similar to mulberry silk. To explore the influence of different influencing factors on the e-nose detection samples, 500ml, 800ml and 1,000ml-range beakers were used in the experiment. The sample rest time was 30min, 45min and 60min before the test; the sample quality was 2g, 3g and 4g respectively; the samples were those at the silk ratio 0%, 10%, 30%, 50%, 70%, 90%, and 100%, respectively. The mulberry silk from Jiangsu Nantong and Zhejiang Tongxiang was selected for studying the influence of mulberry silk of different producing areas on e-nose detection. Figure 1 shows the flow chart of the e-nose: firstly, make sampling by an odour sampler, then the odour signal is transmitted to the gas sensor, and finally, it enters the pattern recognition unit after the signal pre-processing.

Figure 1: Work flow chart of electronic nose

3. Optimization of e-nose detection test parameters for different proportions of silk/polyester yarn samples

3.1 Study on the influencing factors of e-nose response characteristics

Gas sensor is the core of the e-nose system, and the results of the e-nose response are related to headspace, sample quality (Miao, 2018), and headspace generation time (Wlodzimirow et al., 2014). The headspace refers to the space touched by the gas sensor. The larger the headspace, the lower the response concentration of the gas, and the higher the mass of the sample. Under the same headspace, the higher the response concentration of the gas, the longer the headspace generation time, and the greater the response concentration of the gas. Ten different metal oxide sensors were selected for this experiment. In the previous experiments, the ten metal oxide sensor experiments found that the response of the first few seconds was not much different. Due to the different volatile concentrations of the sample gas, with the response time prolonged, the response results were quite different. The response values at the three characteristic moments of the ten sensors: 15s, 45s and 75s were analysed, and the influence of the headspace on the response signals of the sensor S7 and S9 was significant. F-detection was also carried out for the response signals of the e-nose sensors in the condition of different headspaces, indicating significant differences.

Figure 2 shows the relative standard deviation of the sensor response values in different headspaces. It can be clearly seen that the relative standard values of 500ml vary greatly, and the signal values of the sensors are unstable; the relative standard deviation curves of 800ml and 1000ml breakers make no big difference; at the headspace 800ml, the response data of the sensor is more stable when the relative standard deviation is small. The main reason is that the gas concentration cannot reach the detection requirement when the headspace is too large, with great test error; when the headspace is small, the gas volume is greatly affected by external factors, and the response signal fluctuates greatly.

Figure 3 shows the relative standard deviation of the response values by the sensors under different sample quality. The fluctuation between the sensors is not significant at 15s, and the responses values of sensors No. 2, 7, and 9 to different sample amount differ greatly.
Figure 2: Relative standard deviation of sensor response values in different headspaces

Figure 3: Relative standard deviation of sensor response values under different sample qualities
3.2 E-nose detection parameter selection

The continuous sampling of the e-nose sensor is required. If the sampling time is too short, the response signal of the e-nose sensor will end before being stabilized. The e-nose detection parameters include the sampling time and the washing time. Figure 4 shows the relative standard deviation of the response values by the sensors under the conditions of three-time moments. As the response time increases, the relative standard deviation of each sensor is smaller, and the response value is more stable. The e-nose should be washed once test is completed. The washing time will also affect the sensitivity of the e-nose test. To meet the requirements of rapid detection, the washing time is finally determined to be 60s.

4. E-nose detection analysis of different proportions of silk/polyester yarn

4.1 E-nose sensor response curve of different proportions of silk/polyester yarns

Silk and polyester have similar appearances. It’s difficult for human eyes to distinguish when the polyester is incorporated into silk, but the odours exhibited by the two materials are different and can be identified by e-nose sensors (Wilson et al., 2013). Different silk proportions have different odour concentrations in the same headspace, and the feasibility of different proportions of silk/polyester yarn can be quickly detected by e-nose. Figure 5 is a typical response signal diagram of the e-nose. It can be seen from the figure that within 15s, the response value of each sensor changes greatly; after 15s, the response value of each sensor tends to be stable, and No. 2, 6, 7, 8 and 9 sensors rose first and then tended to be flat throughout the whole inspection process. In the detection analysis of different proportions of silk/polyester yarn samples, the characteristics should be extracted and selected first. Table 1 lists the principal component analysis and contribution rate under different eigenvalues: the cumulative contribution rates of the first principal components at 15s, 45s and 75s are 61.67%, 62.603% and 87.33% respectively, and those of the second principal components were 34.644%, 20.134% and 6.308%, respectively. The first two principal components explained the information about 96.314%, 82.737% and 93.638% of the original variables.

Figure 4: Relative standard deviation of response values of sensors at three times

Figure 5: Electronic nose typical response signal diagram
Table 1: Principal component analysis and contribution rate under different eigenvalues

<table>
<thead>
<tr>
<th>Main ingredient</th>
<th>Eigenvalues</th>
<th>Contribution rate/%</th>
<th>Cumulative contribution rate/%</th>
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<tr>
<td>15s</td>
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<td>6.168</td>
<td>61.670</td>
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<td></td>
<td>2</td>
<td>3.327</td>
<td>96.425</td>
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<tr>
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4.2 Detection analysis of different proportions of silk/polyester yarn samples

In this paper, 10 metal oxide sensors were used to respond to different proportions of silk samples. Fig.6 shows the sensor stability value “loadings” analysis. Figure 7 shows the bar graphs of different ratios of silk/polyester yarn sensors; No. 2, 6, 7, 8, and 9 have the highest contribution rate, the sensor array combination after optimization selection was made for factor analysis, and the cumulative variance contribution rate of the first two principal components exceeded 95%. It plays a major role in distinguishing silk/polyester yarns. Only the response data of sensors No. 2, 6, 7, 8, and 9 were extracted during sample detection. Table 2 lists the classification of the test samples by discriminant analysis. The response values of 21 parameters for the seven groups of samples at three time points were classified, at the correct rate of 90.286%, which has a high prediction accuracy.

Table 2: Discriminant analysis of the classification of training samples

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<tr>
<th>Species</th>
<th>0%</th>
<th>10%</th>
<th>30%</th>
<th>50%</th>
<th>70%</th>
<th>90%</th>
<th>100%</th>
<th>Subtotal</th>
<th>Correct rate</th>
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<tr>
<td>10%</td>
<td>3</td>
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<td>6</td>
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<tr>
<td>30%</td>
<td>0</td>
<td>2</td>
<td>18</td>
<td>5</td>
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</tr>
<tr>
<td>50%</td>
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<td>2</td>
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<tr>
<td>90%</td>
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<td>30</td>
<td>28</td>
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</table>

5. Conclusions

In this paper, the data collection of silk/polyester yarn with different mixing ratios was obtained by e-nose technology, and the cluster analysis was conducted for silk samples. The specific experimental conclusions are as follows:
(1) The larger the headspace, the lower the response concentration of the gas, and the higher the mass of the sample. Under the same headspace, the larger the response concentration of the gas, the longer the headspace generation time, and the greater the response concentration of the gas.

(2) The e-nose detection parameters include the sampling time and the washing time. With the extension of the response time, the relative standard deviation of each sensor becomes smaller, and the response value is more stable. To meet the requirements of rapid detection, the washing time is finally determined to be 60s.

(3) Through the detection analysis for different proportions of silk-polyester yarn samples, it is found that the e-nose sensor has a high prediction accuracy rate, at the correct rate of 90.286%.

Acknowledgments

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