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Evaluation of Quality Changes of Leisure Dried Tofu During Storage Based on Electronic Nose

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The objective of this study was to detect and distinguish quality changes in the process of storage of leisure dried tofu at room temperature based on an electronic nose (E-nose). Through analysis for multiple modes such as principal component analysis (PCA), linear discriminant analysis (LDA) and discriminant factorial analysis (DFA) of the E-nose, which can be indicated that LDA can clearly distinguish the leisure dried tofu under different storage times; while DFA can classified that dried tofu samples of different storage times with a recognition rate of 83.3%. The main component (protein, fat and carbohydrate) content of the dried tofu is in direct connection with the response values of sensors of the E-nose. The content of protein significantly correlated to S3, fat was significantly correlated with S2 and S6, carbohydrate significantly correlated to S1, and the correlation coefficients were 0.930, 0.984, 0.968, 0.965 ($P < 0.01$), respectively. The results of comprehensive sense organs, E-nose and main components of the dried tofu suggested that before 60 days the dried tofu presented a high grade and excellent quality, from 90 to 120 days the samples displayed reduced characteristic flavor and the contents of the dried tofu changed to a middle grade. After 150 days the changes in the dried tofu was obviously low in both flavor and contents categories.

Keywords: Leisure Dried Tofu, Storage, Electronic Nose, Quality Change.

1. INTRODUCTION

Among many biobased materials[1–14] and foods,[15–18] tofu is a traditional Chinese food that is favored by consumers.[19] In recent years, tofu is processed by baking, stewing and seasoning into leisure dried tofu which has unique flavor and rich nutrition, and has made a figure in leisure food market.[20] With the development of economy and improvement of consumption, the demand for quantity and quality of leisure dried tofu will grow constantly and, therefore, the market prospect for dried tofu is promising. However, with prolonging of circulation time, dried tofu within the shelf life may have some quality changes such as hardened texture, loss of elasticity and rancidity which are not caused by microorganism, and these changes have become the new bottleneck in the bean product industry. Hence, establishment of a rapid detection method for quality evaluation of leisure dried tofu can help an enterprise to guide production and circulation according to the actual quality of its products and enables consumers to eat high-quality bean products.

The existing food quality evaluation method mainly adopts physicochemical index analysis and sensory evaluation techniques. These methods are relatively mature and high in accuracy, but they also have the defects that the detection time is long, sample pretreatment is complicated, the cost for hiring a professional sensory valuator is high, a plenty of restrictions exist, the subjectivity of the method is strong and the demand for rapid assessment cannot be satisfied.[21–23] Recently, much attention has been paid to sensing technologies,[24–33] E-nose is an intelligent olfaction bionic system built with a sensor array which is formed by combination of multiple metal-oxide semiconductor sensors with different properties, combining a specific signal recording system and a recognition algorithm for the intelligent identification mode.[34,35] It can correctly and rapidly identify and classify samples by recognizing the difference of "flavor finger-printing data" between different samples to be tested.[36–38]

There are a plenty of reports about E-nose techniques in food quality test. The E-nose has been applied for the

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Table I. Sensor performance table.

Sensors	Performances	Reference material	Main application area
S1	Aromatic compounds	Benzaldehyde (almond flavour) and cinnamaldehyde (cinnamon flavour)	Spice, raw materials of Chinese medicine and spiceberry such as clove, cinnamon and vanilla
S2	Oxynitrides and low- molecular amines	Methylamine and dimethylamine	Processing and storage change of meat products and aquatic products, meat spoilage, dairy food spoilage and so on
S3	Sulfides	Mercaptan and thioether	Fragrance detection of vegetables such as garlic, radish, onion and shallot
S4	Organic acid ester and terpenes	Geraniol and citral	Maturity grade detection of fruit and melons and microbial fermented products (white spirit, beer, soy, vinegar, yoghurt, cheese and so on)
S5	Terpenes and esters	Tert-butyl perbenzoate	Dairy food, baked products, cooked food, aroma enhanced or adjusted products, enzymes-hydrolyzed products etc.
S6	Aliphatic hydrocarbon containing oxygen derivatives	Alicyclic alicyclic	Rancidity, cream and lower fatty acids
S7	Sterols and triterpenes	Lenthionine	Mushrooms and edible mushrooms
S8	Amines	Trimethylamine	Putrefaction products, environment detection and gas detection
S9	Hydrogen	Hydrogen	Environmental and gas detection

detection of the changes of volatile components of pork, chicken and shrimp under different storage temperatures and times, and successfully evaluated the freshness of the three kinds of meat.[39–41] E-nose also has proven to be a good method for distinguishing and predicting the storage times of fruits such as apple, honey peach and pear, and proved the potential of the E-nose to predict the shelf life of fruit correctly.[42–44] In the area of tea detection, it has reached a consensus that E-nose is a new technique to distinguish grades of tea quickly, conveniently and reliably.[45] There are far more applications of E-nose in food quality, including liquor,[46,47] grain and oil,[48–50] aquatic products,[51,52] dairy products[53] and so on. However, there is no report on application of E-nose in quality test of bean products.

The objectives of this research were
(1) to distinguish leisure dried tofu under different storage times on the basis of E-nose and multiple pattern recognition algorithms;
(2) to build models for different quality grades of the dried tofu and recognize the grades of dried tofu under different storage times; as well as
(3) to analyze the relationship between the main component (protein, fat and carbohydrate) that produced volatile compounds of the dried tofu and different sensors.

2. MATERIALS AND METHODS

2.1. Samples

The samples of leisure dried tofu were produced in WuGang, Hunan. Each sample weighing 25 g was in an independent vacuum package made of aluminum foil. The shelf life marked on the package is 180 days under the room temperature. The samples were placed in simulation home and avoided light for 0 to 180 days. The monthly mean temperature in chamber is 28, 19, 13, 8, 8, 9, 11 °C, respectively.

2.2. Electronic Nose

An iNose electronic nose includes nine metal oxide sensors and collocated with Smart Nose intelligent identification software system for datum analysis (Ruibin Intelligent Technology, Shanghai, China). Each sensor is sensitive to different volatile compounds, the main application of the nine sensors are listed in Table I.

2.3. Sensory Evaluation

The sensory evaluation group was composed of 20 persons including 10 males and 10 females. All of food science and technology panelists with professional skills of food sensory evaluation to classify the quality of different storage time of the samples of dried tofu. The sensory evaluation was carried out with respect to the three quality grades (high, middle and low) of dried tofu. Three attributes of dried tofu, including texture, taste and flavor listed in Table II. Then, the evaluation of the three grades of dried tofu was performed using five-scale categories according to the following standard: 1-love; 2-like; 3-neither like nor dislike; 4-dislike; 5-hate. The average scores of sensory given by 20 panelists were provided as evaluation results. The average scores of sensory given by 20 panelists were provided as evaluation results.

2.4. Electronic Nose Sampling Procedure

Placed 2.00 g (correct to 0.01 g) of sample into a 20 mL of headspace bottle for 10 minutes (headspace generation time) under the temperature of 25 °C while the headspace collected the volatiles form the samples. The measurement

Table II. Sensory evaluation table for quality classes of the dried tofu.

	Class		
Attribute	High grade	Middle grade	Low grade
Texture	Dense in texture, moderate in hardness and good in elasticity	Bad in hardness and normal in elasticity,	Loose in texture, too hard and non-elastic
Taste	Fine and chewy	Normal in chewiness	Crispy and bad in chewiness
Flavour	Pure in fragrances of bean and brine, long in aftertaste time	Tasteless in fragrances of bean and brine, normal in flavour	Taste abnormal such as slight acid and putrid odor, bad in flavour

Table III. Results of sensory evaluation.

	Sensory scores		
Storage time (days)	High grade	Middle grade	Low grade
0	1.60 ± 0.66^{a}		
30	1.55 ± 0.59^{a}		
60	1.50 ± 0.67^{a}		
90		-0.10 ± 0.62^{b}	
120		-0.15 ± 0.36^{b}	
150			-1.25 ± 0.62^{c}
180			-1.30 ± 0.46^{c}

Notes: The values were the average ± standard deviation of the total score of the 20 graduates. Mean in the same row followed by different letters (*a*, *b*, *c*) is statistically different for $P < 0.05$.

phase gas flow at a constant rate of 0.3 L/min and lasted for 120 s, which was long enough for the sensors to reach stable signal values. There was a cleaning phase for 120 s, and an automatic zero setting phase for 10 s prior to the next detection. In this experiment, the flat values were selected for analysis.

First, select samples of dried tofu which in different storage times, and conduct 9 parallel tests which are mainly used for PCA and LDA. Then, conduct DFA for modeling according to sensory evaluation divided 108 samples into three grades, and extract other 24 samples randomly for model verification.

2.5. Contents of Dried Tofu Analysis

The contents of protein, fat and carbohydrate in the samples were used by the mode of "Prepared foods" of Calory Answer (JWP, Japan). The result was used for correlation analysis between the contents in the dried tofu and the response value of the sensors.

2.6. Statistical Analysis

Data obtained from the sensors were analyzed by the Smart Nose software of the E-nose. Other data were analyzed by the data processing software SPSS20.0.

3. RESULTS AND DISCUSSION

3.1. Results of Sensory Evaluation

The results of sensory evaluation of the samples are presented in Table III. There were statistically significant differences for the three grades ($P < 0.05$), indicating that the taste and quality of the dried tofu are getting worse with duration of the storage time. Both taste and flavor remained in good to very good quality for up to 60 days as the high grade and with fragrant flavor of bean and brine, good elasticity, soft texture and moderate hardness. From 90 to 120 days the dried tofu was shown to present slightly reduced characteristic flavour and texture belonging to middle grade. After 150 days the changes in dried tofu during storage was demonstrated to make the tofu change significantly to low grade, i.e., a slight rancidity and lost of elasticity, loose texture and increased hardness, as well as bad taste, 62.5% personnel can accept but dislike the senses, 32.5% cannot accept.

3.2. Response Curves of Electronic Nose

Figure 1 is typical response of Electronic nose measurement of the dried tofu sample. Each curve represents the change of the ratio of conductance during measurement (G/G0, where G and G0 are the conductivities of the sensor when exposed to the sample gas and the zero gas, respectively). As observed in Figure 1, except S7, S8 and S9, the conductivity of the rest sensors gradually increased then remained stable. This is because the three sensors are sensitive to environmental gases detection, and these substances were not detected from the sample. One spider plot drawn to provide a graphic representation of the flavor profiles of the dried tofu for different storage times at room temperature is easily to be identified in Figure 2. Different sensors had different response values for volatile substances of the samples under different storage times. From 0 to 150 days, the intensity decreased continuously because the dried tofu characteristic flavour gradually and slightly reduced as time went on. After 150 days, the sensors S1∼S6 presented higher response values, especially S2 and S4. This indicated that there might be loss

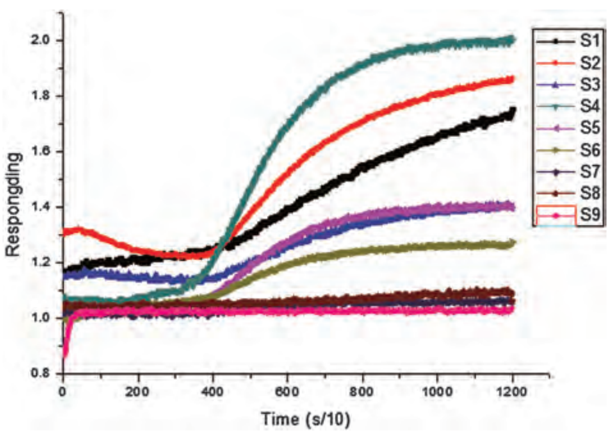

Fig. 1. Sensors response curves of a leisure dried tofu sample.

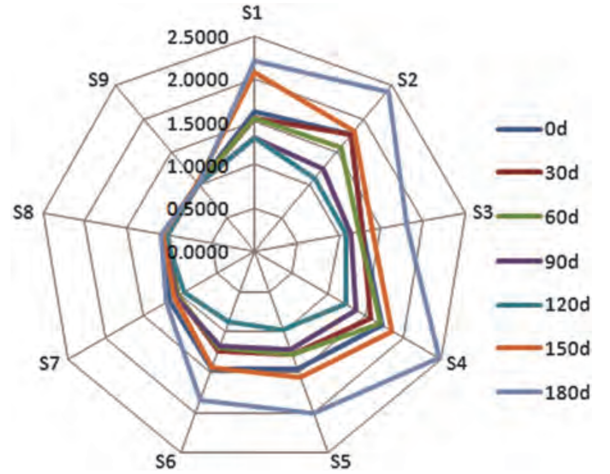

Fig. 2. Response values of different storage times of dried tofu by electronic nose.

of the quality of tofu, because S2 and S4 are sensitive to oxynitrides, low-molecular amines, organic acid ester and terpenes, these kinds of substances were produced by spoilage of food, which was consistent with its sensory evaluation in the period of storage. Considering very small changes in the response values of the S7–S9 sensors, S7–S9 were not within the subsequent study.

3.3. PCA and LDA of the Dried Tofu for Different Storage Times at Room Temperature

E-nose datum PCA of the dried tofu stored for 0–180 days is listed in Figure 3. The representativeness of the principal components increases with the contribution rate of the first principal component (PC1) and the second principal component (PC2) of the samples, and the total contribution rate of PC1 and PC2 shall exceed 70%–85%.[26,27] The contribution rate of PC1 was 94.2%, the contribution rate of PC2 was 2.5%, and the total contribution rate reached 96.7%, which can represent the overall information of the samples. The DI value was −35.6%, which is the key index in judging the distinguishing capacities of PCA model and LDA model. It refers that the distinguishing effect is better when

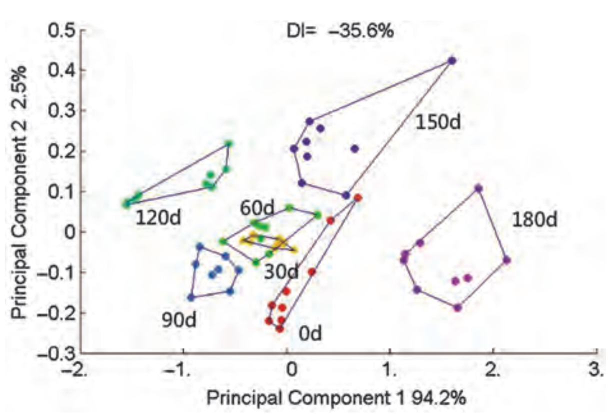

Fig. 3. PCA for the dried tofu for different storage times.

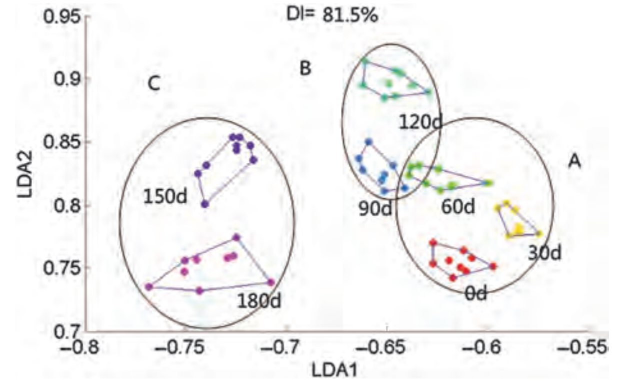

Fig. 4. LDA for the dried tofu for different storage times. A, High grade; B, middle grade; C, low grade.

the ID value is much closer to 100%, and the negative value means that the models cannot totally distinguish the samples to be tested.[28] Parts of the graphs of the dried tofu stored for 30 days and 60 days overlapped in the figure, and therefore the DI value was negative; the variation trend of the graph distribution was unobvious when the storage time changes, indicating that PCA was not the best method for distinguishing the dried tofu for different storage times. The area of graph range formed by parallel samples after stored for 150 days and 180 days increased more obviously than those of other groups, it can be reflected that the variation difference of flavor between the units in the two stages also increases.

Figure 4 shows datum result of the electronic nose for LDA of the same samples as above, the graphs can be totally distinguished, and the DI value was 81.5%. However, a certain trend appears along with the changes of storage times. The graphs for samples stored for 0, 30, 60, 90 and 120 days were basically the same, indicating that the main odor features of the dried tofu within 0–120 days were quite similar. The graphs of the 150 days and 180 days are far apart from other groups, which can indicate that the odor in those two stages differs greatly from that in earlier stage. Since the quality must have changed if the odor of the food has changed[29,30] and to some degree the sensory evaluation is in good agreement with the results of the E-nose, it can be inferred that quality of the dried tofu stored for 150–180 days has changed obviously. LDA model can exquisitely distinguish the odor difference between the samples. The model features are that the concentration ratio of the similar data is higher, and the distance between the groups is larger.[31,32] So LDA was more suitable than PCA for distinguishing the samples of dried tofu in different storage times.

3.4. Identification of Different Quality Grades Using DFA

DFA correctly classifies the sample collection based on PCA and LDA to build a model which is used to judge the category of the unknown samples.[33] The DFA results

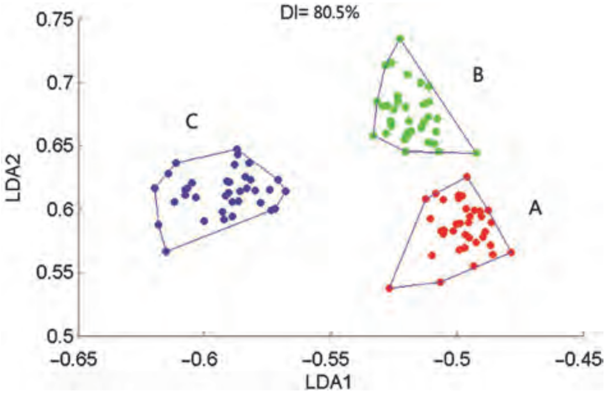

Fig. 5. DFA model of dried tofu in different quality grades. A, High grade; B, middle grade; C, low grade.

obtained for cluster air dried data set with four different air-dried licorice roots groups and a DFA recognition of 93.3%;[34] A spice category using the DFA model for recognizing 8 spices can reach 100%.[28] Naresh et al. have detected the milk which is not vaccinated and vaccinated with bacteria (*Pseudomonas*, *Pseudomonas fluorescens* and *Bacillus cereus*) and yeast (*Kluyveromyces lactis*), and the DFA model can clearly distinguish the unpolluted milk containing putrefying bacteria or yeast.[35]

DFA models for dried tofu in different classes are shown in Figure 5. The discrimination accuracy of the random-drawn 24 samples of the dried tofu for the three grades reached 83.3% by using DFA model which is listed in Table IV. Precision rate of class DFA model for dried tofu was not as high as the above since the pioneering studies mainly recognizes different categories and regions samples, reducing the difficulty of recognition. Therefore, quality discrimination model for the dried tofu was able to discriminate the quality classes of the dried tofu effectively.

Table IV. Results for recognizing quality of the dried tofu by DFA.

Samples: Storage time (d)	DFA analysis result[a]	Result of sensory evaluation	Samples: Storage time (d)	DFA analysis result	Result of sensory evaluation
0	1	1	84	2	2
13	3	1	92	2	2
24	1	1	99	2	2
35	1	1	104	2	2
38	1	1	105	2	2
45	1	1	109	3	2
47	1	1	133	1	3
58	2	1	140	3	3
61	1	1	151	3	3
62	2	2	151	3	3
76	2	2	180	3	3
81	2	2	187	3	3

Note: [a]1-High grade; 2-Middle grade; 3-Low grade.

Table V. Contents of principal components in high, middle and low grades of the dried tofu (%).

Quality	Protein	Fat	Carbohydrate
High grade	18.167 ± 0.424^{c}	14.544 ± 0.510^{ab}	8.544 ± 0.602^{a}
Middle grade	19.360 ± 0.327^{b}	14.047 ± 0.294^{b}	7.277 ± 0.741^{b}
Low grade	21.892 ± 0.796^{a}	14.925 ± 0.579^{a}	6.234 ± 0.563^{c}

Notes: The values are the average ± standard deviation with respect to 36 samples. Mean in the same column followed by different letters (*a*, *b*, *c*) is statistically different for $P < 0.05$.

3.5. Contents of the Dried Tofu and its Correlation with the Sensors Values

Some nutrition components of food may change with prolonging of storage time and generate different volatile organic compounds, which in turn highly influence its aroma and taste, and are very critical in determining its quality,[36,37] and cause that changes of response values of sensors of the E-nose may be generated. For example, carbohydrate can be decomposed into hydrocarbon, alcohols, ketones, aldehydes, carboxyl acid gases and so on; protein can be decomposed into ammonia, sulfuretted hydrogen, ethanethiol and so on; and fat can be decomposed into aldehydes, aldehyde acid and so on.[38,39] It is therefore very important to explore the relationships between these components of the dried tofu and the sensors.

Leisure dried tofu contains rich protein, fat and carbohydrate.[40] According to Table V, there were significant changes in the contents of protein and carbohydrate in the dried tofu for three quality grades ($P < 0.05$) during storage, while there was no significant different between the high and middle grade in the contents of fat in samples. The results suggested that protein and carbohydrate had influence on the quality of dried tofu than fat.

A correlation test was used to analyze the relationship between the volatile content and the sensors. According to Table VI, the content of protein was significantly correlated with S1∼S6 sensors, especially S3, and the correlation coefficient was 0.930 ($P < 0.01$), for S3 was sensitive to volatile sulfide, which was produced by protein. Fat had significant correlation with S2 and S6, and the correlation coefficients were 0.984 and 0.968, S2 and S6 which are respectively used for detecting primary products of spoilage of food and oil rancidity. Carbohydrate had the most significant relationship with S1 and the correlation coefficient was 0.965. It was demonstrated that the odor response of E-nose could reflect the inner quality of dried tofu to a certain degree.

Table VI. Correlation analysis of quality tofu and the sensors.

	S1	S2	S3	S4	S5	S6
Protein	0.741^{**}	0.737^{*}	0.930^{**}	0.879^{**}	0.895^{**}	0.787^{**}
Fat	0.610	0.984^{**}	0.856^{**}	0.912^{**}	0.897^{**}	0.968^{**}
Carbohydrate	-0.965^{**}	−0.539	-0.804^{**}	-0.727^{*}	-0.750^{*}	−0.602

Notes: *Refers to significant correlation in 0.05 level, **refers to significant correlation in 0.01 level.

From the results of comprehensive sense organs, E-nose and main components of the dried tofu, it was concluded that before 60 days dried tofu can be classified as high grade with excellent quality, while from 90 to 120 days the samples were of reduced characteristic flavour and the contents of dried tofu changed to middle grade. After 150 days the dried tofu changed to low grade obviously in both flavour and content categories.

Peter thought it was difficult or even impossible to applied E-nose in real routine use outside of scientific laboratories.[41] The present work was successfully applied to distinguish storage time and quality of the leisure dried tofu based on an electronic nose for the first time. At the same time, it also was applied to analyze the relationship between protein, fat and carbohydrate of the dried tofu and different sensors. The above results have indicated the potential of its application in enterprises. Although this model has selectivity, the benefit it brought can be once and for all. It is significant to explore more relations between the signal of the E-nose and the quality of the dried tofu, deepen quality changes of the dried tofu during shelf life theoretically, determine key indexes of quality changes, and predict reside shelf life of dried tofu according to partial least squares regression (PLS). Further complete quality class distinguishing model should be developed to continuously improve precision and accuracy of model detection. With development of sensing technology[41–48] and its application,[49–59] we can envision that the method presented here can be improved as a fast and effective quality evaluation technique for quality controls.

4. CONCLUSIONS

We could distinguish leisure dried tofu under different storage times on the basis of E-nose and multiple pattern recognition algorithms through statistical analysis results. Also we built models for different quality grades of the dried tofu and recognize the grades of dried tofu for different storage times, as well as to analyze the relationship between the main components (protein, fat and carbohydrate) that produced volatile compounds of the dried tofu and different sensors.

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