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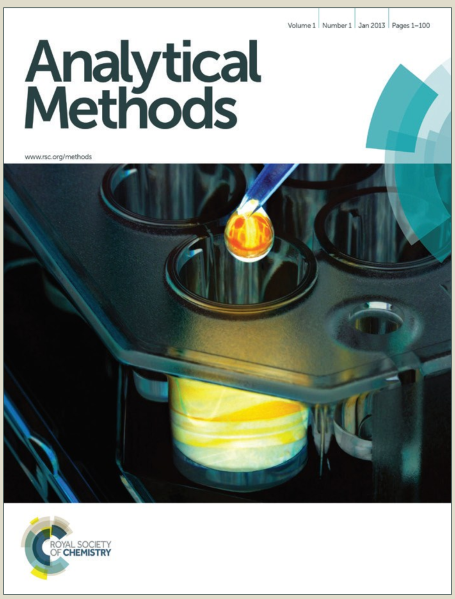

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Chinese Bayberry (*Myrica Rubra Sieb. Et Zucc.*) Quality Determination Based on Electronic Nose and Non-linear Dynamic Model

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Abstract:

In this paper, Chinese bayberry (*Myrica Rubra Sieb. Et Zucc.*) quality determination method using electronic nose (e-nose) and non-linear stochastic resonance (SR) technique has been studied. E-nose responses to bayberry samples stored at 4℃ for 7 days are measured. In order to characterize samples' quality, Physical-chemical indexes, such as human sensory evaluation (HSE), texture, color, pH, total soluble solids (TSS), and reducing sugar content (RSC), are examined. E-nose measurement data is processed by principal component analysis (PCA), SR and double-layered cascaded series stochastic resonance (DCSSR) methods. PCA can not totally discriminate all bayberry samples. Bayberry SNR maximum (SNR-Max) values calculated by SR and DCSSR increase with the increase of storage days. SNR-Max values successfully discriminate all bayberry samples. Multiple variable regression (MVR) between physical-chemical indexes (firmness, pH, color, TSS, and RSC) and SR/DCSSR SNR-Max values have been conducted. Results indicate that SR is more suitable for Chinese bayberry quality determination than DCSSR. Bayberry quality predicting model is developed based on linear fitting regression on SR eigen values. Validating experiments results demonstrate that the developed model

predicts bayberry quality with an accuracy of 95%. The proposed method takes advantages including easy operation, fast responses, high accuracy, good repeatability, and low cost.

Keywords: Chinese bayberry; quality; electronic nose; stochastic resonance; signal-to-noise ratio

1 Introduction

Chinese bayberry (*Myrica Rubra Sieb. Et Zucc.*) is one kind of bayberry fruits native to China with high commercial value. People are attracted by bayberry's red to purple color, sweet and sour taste, and nice flavor [1]. However, Chinese bayberry easily suffers from mechanical injury and microbiological decay due to its harvest season (from June to July). These factors reduce bayberry's postharvest life to a great deal [2,3]. Storage conditions, such low temperature (4℃) storage, bayberry's beneficial components still decline quickly, and have no more than 5 days' shelf life [4]. The intake of stale bayberry usually causes gastrointestinal diseases and does great harm to consumers' health [5]. Traditional fruit product quality analysis methods mainly include instrumental analysis, physical-chemical analysis methods, and human sensory analysis. Instrumental analysis methods, such as VIS/NIR spectroscopy, gas chromatography-mass spectrometry (GC-MS), and impedance spectroscopy, are usually utilized for fruit freshness detection under laboratory conditions. However, these methods are usually time-consuming, and high cost. Moreover, skilled operators are required to perform the instrumental analytical experiments. These disadvantages limit field applications of these traditional method. Physical-chemical analysis methods, such as conductivity/pH measurement, can determine fruit quality according to relative standards. However, these methods need complex experimental operations. The cost is also relatively high. Human sensory analysis evaluates some food characteristics, such as color, odor, and flavor. But the evaluating results are short of stability and reproducibility. So there is a strong demand for an effective, accurate, and low cost method for Chinese bayberry quality rapid determination.

The human nose can distinguish over 10,000 different smells using 350 receptors. Smell molecules, known as an odorants, interact with the receptors to create an overall responses that is recognized by the brain [6,7]. E-nose technique develops quickly in recent years [8,9]. Its system usually consists of an array of several chemical sensors with partial specificity. Appropriate patterns or fingerprints from known odors are employed to construct a database and train a pattern recognition system so that unknown odors can subsequently be classified and identified. In the past decade, e-nose technique has been employed in recognition and quality analysis of various food and agro-products [9-15]. At the same time, various promising results have been also reported with e-nose for quality screening of fresh and processed fruit juices [16,17].

In this paper, a convenient and rapid way for Chinese bayberry quality discrimination by using e-nose and non-linear dynamic SR was proposed. Bayberry samples are stored at 4℃ for continuous 7 days. E-nose sensor array responses to bayberry samples have been measured. The physical-chemical indexes including HSE, texture, color, pH, TSS, and RSC, are examined to characterize samples' quality. SR and DCSSR methods are used to extract eigen values of e-nose measurement data. Bayberry quality predicting model is proposed through linear fitting regression of SR eigen values.

2. Materials and methods

2.1 Materials

2.1.1 Samples

Fresh Chinese bayberry samples are purchased from Hangzhou Gouzhuang fruit market. Samples nearly in the same size, weight, and maturity were selected. All samples were without any ripening pretreatment and free of diseases. Upon arrival to the laboratory, the samples have the same weight (20.0 ± 4.0g) and curvature radius approximately. The samples are placed in non-hermetic box and stored at 4℃ in a cooler. Each day, 20 bayberries are taken randomly out of the box. 20 samples are used in e-nose measurement. Firmness, pH, soluble sugar content, color, and reducing sugar content are respectively examined using 10 samples. The samples are taken randomly. The experiments are conducted for 7 successive days. Another 70 bayberries are used in model validating experiments and stored at 4℃. 21 samples are randomly-taken and used for e-nose measurement every day.

2.1.2 Chemicals

NaOH (Tianjin Damao Chemical Reagent Co., Ltd, China), HCl (Quzhou Juhua Chemical Reagent Co., Ltd, China), H_3BO_3 (Dongguan Dongjiang Chemical Reagent Co., Ltd, China), MgO (Beijing Tongguang Chemical Reagent Co., Ltd, China), Methyl red (Tianjin Jizhun Chemical Reagent Co., Ltd, China), Methylthionine chloride (Shanghai Sanai Chemical Reagent Co., Ltd, China), Ethanol (Sinopharm Group Chemical Reagent Co., Ltd, China). Deionized water. All chemicals were of analytically pure grade or better quality.

2.1.3 E-nose system

The bayberry samples were monitored by a self-developed portable e-nose [21-23]. Its structure is shown in Fig.1(a). It consists of three main parts: data acquisition unit (U1); gas sensor array unit (U2); power supply unit (U3). The pump is FML201 (Xinweicheng Technology Co., Ltd, Chengdu, China). The valve is WTA-2K (Takasago Electrical Co., Ltd, Tokyo, Japan). The data acquisition unit is developed by laboratory. The sensor array unit has 8 metal oxide semiconductor (M.O.S) sensors with various chemical compositions and thickness to provide selectivity towards different gases. The selectivity toward volatile compound classes of M.O.S sensors is indicated by the manufacturer: sensor

S1 (TGS-825, sulfide), sensor S2 (TGS-821, flammable gases), sensor S3 (TGS-826, ammonia gas), sensor S4 (TGS-822, ethanol, aromatic hydrocarbons), sensor S5 (TGS-842, hydrocarbon component gas, sensor S6 (TGS-813, methane, propane, butane), sensor S7 (TGS-2610, propane, butane), sensor S8 (TGS-2201, nitrogen oxides). E-nose has a gas sensor chamber, which contains eight M.O.S gas sensors (see Fig. 1(b)). The sensor has the gas inlet hole at the hole. The gas sensing membrane is located within the sensor. The detecting mechanism is displayed in Fig. 1(c). The sensing membrane is heated by the resistance wire, and V_H is the heating voltage. V_C is the supplying voltage. V_{RL} is sampling voltage. So E-nose response is measured as sampling voltage (V). The M.O.S sensors rely on changes in conductivity induced by the adsorption of molecules in the gas phase and on subsequent surface reactions. They consist of ceramic substrate coated by M.O.S film, and heated by wire resistor. Due to the high temperature (250-500℃), the volatiles transferred to the surface of the sensors are totally combusted to carbon dioxide and water, leading to a change in the resistance. The high temperature avoids water interference and provides M.O.S fast response and rapid recovery time. Polytetrafluorethylene (PTFE) material is used to fabricate gas sensor chamber. We design a rounded separated sensor chamber, and its schematic diagram is shown in Fig. 1(d). We distribute 8 gas sensors along a circle. The sampling gases are blown from the center of the circle into the separated chamber for the reaction with the sensors. The distance between the circle center and the chamber entrance is the same. At the chamber entrance, the foam is placed to disperse the gas flow, which ensures that the flow influence on the sensor is reduced to the lowest. This method actually reduces the cross-influence and flow influence on sensors in the chamber.

(The preferred position of Fig.1)

2.2 Methods

2.2.1 HSE

HSE experiments are conducted according to Table 1. The bayberry samples are evaluated from the following aspects: (a) Vision observation on bayberry's color, and skin wetness. (b) Touch estimation on bayberry's firmness and resilience by hand. (c) Taste estimation on bayberry. (d) Odor estimation on bayberry's smell by human nose [18].

(The preferred position of Table 1)

The HSE of Chinese bayberry samples is evaluated by 10 experienced panelists (ranged in age from 28 to 48 years), and voting number is set at k, $k \in (1,10)$ [18]. Bayberry quality is divided into m levels, and the score of a specific level is set at h_j, $j \in (1,m)$. Bayberry attributes are divided into n elements, and a specific element is set at u_i, $i \in (1,n)$. The contributory weight is determined by pairwise comparison of contribution weight of attributes is set at x_i ($\sum x_i = 1$). If there is a specific relationship between two objects of h_j and u_i, the relation set (matrix) of f is calculated as follows:

$$F = \begin{vmatrix} f_{11}/k & f_{11}/k & \dots & f_{1m}/k \\ f_{21}/k & f_{22}/k & \dots & f_{2m}/k \\ \dots & \dots & \dots & \dots \\ f_{n1}/k & f_{n1}/k & \dots & f_{nm}/k \end{vmatrix} \quad (1)$$

Thus, the overall acceptability of bayberry is calculated by the weight grade method as follows:

$$Z = \sum_{i=1}^{n} xi \bullet \sum_{m=1}^{m} \frac{fij}{k} hj \quad (2)$$

2.2.2 Texture

TA.XT2i Texture Analyzer (Stable Micro Systems, UK) is used to conduct texture measurement. Flat cylindrical probe p/5 (5mm in diameter) was used. In texture analyzer software setting, TPA mode was selected for instrument control. Speed in pre-measurement and after-measurement was 3mm/s.

Measurement speed was 1mm/s. Compression degree was 50%. Residence time interval was 5s. Load probe type was Auto-0.2g. Data collection rage was 200.

2.2.3 Color

The surface fresh colour of jumbo squid is measured using a chromatic meter (TES-135, Taiwan Taishi Electronic Technology Co., Ltd.) and reported as $L*$, $a*$, and $b*$ as CIELab coordinates. Parameters of $L*$, $a*$, and $b*$ indicate the lightness (the scale range of 0-100 points from black to white), red (+) or green (-), and yellow (+) or blue (-), respectively. Because of differences from the data measured in different positions of a specific sample, we cut meat nearly in the same area to detect. Every group has three coordinate samples and each sample's color detection was repeated eight times. Calculate the mean value and standard deviation as color value.

Changes in the external color are monitored by measuring L*, a*, and b*. According to Carrno group's method, the hue angle (H) and the chroma (C) are defined in this research.

$H = \arctan(b^* / a^*)$, and $C = [(a^*)^2 + (b^*)^2]$ [19]. Hue angle can be distributed in the four quadrants of the $a^* b^*$ plane, and chroma will be higher the further it is from the origin of the coordinates. We use the color index for red grapes (*CIRG*) proposed by Carrno et al to characterize color changes of bayberries. $CIRG = (180 - H) / (L^* + C)$.

2.2.4 pH

In pH measurement, 10g bayberry is smashed by a muller. A little purified water is added into the smashed bayberry to fully dissolve chemical substances. Bayberry sample and water were poured into conical flask. The muller is washed by purified water, and the washing liquid was also poured into conical flask. The mixture in the conical flask was finally diluted to 100 mL, and left to stand for a while. Then the mixture was filtered and measured by PHS-3C pH meter.

2.2.5 TSS

Bayberry is ground in a mortar and squeezed with a hand press for juice. The juice was used for TSS measurement. The measurement was conducted utilizing refractometer (WZ113/ATC, China) at 25℃ temperature.

2.2.6 RSC

RSC index is examined according to China standard protocols GB/T 5009.7-2008: Determination of reducing sugar in foods [20].

2.3 E-nose measurement

Each sample is placed into a 50mL air-tight vial, and sealed with sealing membrane. The vials are equilibrated for 30 min at 20□. Turn on e-nose power, then start washing pump and valve 2. The sampling pump and valve 1 remain off. The air is filtered by active carbon to obtain zero gas. Sensor array are recovered by zero gas. When sensors' responses return to the baseline, washing pump and valve 2 are shut off. Then sampling pump and valve 1 are turned on. The gases in sample's headspace are pumped into gas sensor chamber by sampling pump at a flux speed of 400 mL/min for 40s. E-nose measurement interval is 0.05 s. E-nose real-time responses to bayberry samples are recorded. When measurement finishes, gas sensors are recovered by zero gas at a flux speed of 1000 mL/min for 600s, waiting for the next measurement.

2.4 Data treatment

2.4.1 PCA

PCA is one of the most commonly used methods in E-nose data processing. It is a method to analysis some principal components (PCs) from experimental data. It can transform some related variates into unrelated variates. In the mathematical transformation, we should make sure the total variance of all variates remains unchanged, and the variate that has maximum variance becomes the first variate (or the first PC). Similarly, the variate which variance just behind the maximum becomes the second variate (or the second PC), and make sure it has no relativity with the first variate and other variates. And by this analogy, the number of the variates is same as PCs, and there are no relativity between all variates. So, PCA method is a useful tool for reduced data.

2.4.2 SR

SR was proposed by Italian scientist Benzi to give an explanation for Earth climate periodical changes [24-26]. SR model can be described as follows:

$$\frac{dx}{dt} = -\frac{dV(x)}{dx} + MI(t) + C\xi(t) \quad (3)$$

Where x is the position of the Brownian particle, t is the time, M and C are adjustable parameters, $I(t) = S(t) + N(t)$ is an input signal $S(t)$ with an intrinsic noise $N(t)$, $\xi(t)$ is the external noise, and $V(x)$ is the simplest double-well potential with the constants a and b.

$$V(x) = -\frac{1}{2}ax^2 + \frac{1}{4}bx^4 \quad (4)$$

Eq. (3) can be transformed as

$$\frac{dx}{dt} = ax - bx^3 + MI(t) + C\xi(t) \quad (5)$$

The minima of $V(x)$ are located at $\pm x_m$, where $x_m = (a/b)^{1/2}$. A potential barrier separates the minima with the height given by $\Delta U = a^2/4b$. The barrier top is located at $x_b = 0$. When three elements of SR interact coherently, the potential barrier can be reduced and the Brownian particle may surmount the energy barrier and enter another potential well. The intensity of signals will increase, which makes it possible that the weak signal can be detected from noise background.

Suppose the input signal is $I(t) = A\sin(2\pi ft + \varphi)$, where A is signal intensity, f is signal frequency. D is external noise intensity. SNR is the common quantifier for SR and it can be approximately described as:

$$SNR = \sqrt{2}\Delta U (A/D)^2 e^{-\Delta U/D} \qquad (6)$$

Noise intensity is a parameter in SR model. This model is used for e-nose data analysis. $I(t) = A\sin(2\pi ft + \varphi) + EN(t) + N(t)$ denotes an input matrix. It has a sinusoid signal $A\sin(2\pi ft + \varphi)$, electronic nose response data $EN(t)$, and intrinsic noise $N(t)$. SNR between the output and input is calculated. This model has been successfully used in food analytical applications [21,22].

2.4.3 DCSSR

Under adiabatic elimination condition, supposing signal amplitude is much smaller (A<<1), the Brownian particle is in one of potential wells because no enough driving energy is provided by the bistable system to drive the particle to jump from one potential well to another one in the absence of external noise. The signal period is longer than some characteristic intrawell relaxation time for the system. The existence of periodic forcing inclines the potential function, and forms the Brownian particle's transfer from one potential well to another one. So the potential function $V(x)$ changes with the input signal and becomes

$$V(x,t) = -\frac{1}{2}ax^2 + \frac{1}{4}bx^4 + Ax\sin(2\pi ft + \varphi) + EN(t)x + N(t)x \qquad (7)$$

Equation (7) indicates the potential function gets time dependence. Equation (8) displays the first-order and second-order derivation of $V(x,t)$ with respect to x, and let the equations equal to zero:

$$\begin{cases} \partial V(x,t)/\partial x = -ax + bx^3 + A\sin(2\pi ft + \varphi) + EN(t) + N(t) = 0 \\ \partial V^2(x,t)/\partial x^2 = -a + 3bx^2 = 0 \end{cases} \qquad (8)$$

Setting noise intensity $D=0$ and $\sin(2\pi ft+\varphi)=1$, the critical amplitude value of the periodic signal can be obtained: $A_c=\sqrt{4a^3/27b}$. If $A<A_c$, the particle hovers around its original stable state and can not jump from one potential well to another one. However, the particle can jump from one potential well to another one with the help of external noise even if $A<A_c$, which means the occurrence of SR phenomenon. We use fourth-order Runge-Kutta numerical algorithm to solve Equation (3):

$$x_{n+1}=x_n+\tfrac{1}{6}[k_1+(2-\sqrt{2})k_2+(2+\sqrt{2})k_3+k_4],\quad n=0,1,\cdots,N-1 \tag{9}$$

$$k_1=h(ax_n-bx_n^3+sn_n) \tag{10}$$

$$k_2=h[a(x_n+\frac{k_1}{2})-b(x_n+\frac{k_1}{2})^3+sn_n] \tag{11}$$

$$k_3=h[a(x_n+\frac{k_2}{2})-b(x_n+\frac{\sqrt{2}-1}{2}k_1+\frac{2-\sqrt{2}}{2}k_2)^3+sn_{n+1}] \tag{12}$$

$$k_4=h[a(x_n+k_3)-b(x_n-\frac{\sqrt{2}}{2}k_2+\frac{2+\sqrt{2}}{2}k_3)^3+sn_{n+1}] \tag{13}$$

x_n is the *n*th numerical value of $x(t)$, and sn_n is the *n*th numerical value of $S_n(t)$. h is the computation step. Much progress has been achieved on the applications of SR in the past few decades.

Single SR system connected in series forms the cascaded SR to obtain DCSSR (see Fig. S1 (b)). According to Equation (3), the relative Langevin equations of the cascaded bistable systems can be respectively written as:

$$\begin{cases} dx_1/dt=ax_1-bx_1^3+M[A\sin(2\pi ft+\varphi)+EN(t)+N(t)] \\ dx_2/dt=ax_2-bx_2^3+x_1(t) \\ \cdots \\ dx_n/dt=ax_n-bx_n^3+x_{n-1}(t) \end{cases} \tag{14}$$

In practical engineering measurement, measured data usually consists of signal and intrinsic noise. If an aimed weak signal is submerged in strong noise, we are not able to detect it. With the help of SR, the energy of intrinsic noise is lowered, and the embedded weak signal is amplified effectively so that the signal can be caught for measurement characterization. In some special measurement, the weak signal is so weak that the aimed signals are still embedded in the noise. DCSSR is designed to solve this problem. For a DCSSR system, several bistable systems are in serial. If the measurement signals are still embedded in the noise, the output of the former bistable system is taken as the latter bistable system. If the aimed signals are successfully obtained, the current number of bistable systems is the suitable for the information measurement. The schematic diagram of double-layered cascaded series stochastic resonance is showed as Fig. 1(e).

2.5 Statistical analysis

HSE and physical-chemical data were subjected to one-way analysis of variance (ANOVA). Mean separations were performed by Tukey's multiple range test (SPSS version 17.0). Differences at $P < 0.05$ were considered significant.

3 Results and discussion

3.1 HSE results

Sensory attributes of Chinese bayberry are divided into 5 elements, whose preference levels are scored from 1 to 5 (see Table 1). Here, a sensory score of 3 is set as the minimal value for acceptable bayberry quality. Changes in preference scores of Chinese bayberry stored at 4℃ are displayed in Fig. 2. The initiative preference score is 5 in day 0, and it presents a decrease trend during storage ($P < 0.05$). After 4 days of storage, bayberry's preference score is 2.6, which is less than 3 and therefore considered as unacceptable. HSE results demonstrate that the quality of Chinese bayberry decreases sharply during cold storage. Changes in sensory attributes of bayberry can be attributed to biochemical reactions occurred in fruit tissues under the effect of relevant enzymes.

(The preferred position of Fig. 2)

3.2 Texture measurement results

Texture measurement results of Chinese bayberry stored at 4℃ are displayed in Fig. 3. As shown in Fig. 3(a), bayberry firmness is 240 g in day 0 and during the following 6 days of storage, it decreases swiftly. At the end of storage, the firmness value approximately decreases to 125 g, nearly losing 48% with respective to the initial firmness value. Changes in resilience and cohesiveness of Chinese bayberry during storage are similar (Fig. 3(b) and (c)). The initiative resilience and cohesiveness of bayberry are 1.43 and 0.187, respectively. During the following storage days, bayberries suffer sharp decreases in resilience and cohesiveness, and it decreases to 0.6 for resilience and 0.133 for cohesiveness after 6 days. Unlike other fruit, Chinese bayberries have no firm peel and thereby are easily susceptible to losing moisture, which to some extent contributes to the texture decay. On the other hand, the degradation of cell-wall polysaccharides and other materials due to the effect of pecinase and other catalysis enzymes largely accelerates this process [27,28]. Similar changes in texture profiles are also reported in plum [29] and kiwifruit [30].

(The preferred position of Fig. 3)

3.3 Color measurement results

It is widely accepted that the most important parameter in determining fruit and vegetable acceptability by consumers is color. Color indexes including L^*, a*, and b* are determined and then H, C, and CIRG are calculated during cold storage for 6 days, as displayed in Table 2. The *CIRG* value of bayberry is 8.10 in day 0, with 12.63 of L^*, -0.86 of H, and 9.73 of C. After 1 day of storage, significant changes in H and C are presented ($P < 0.05$). But in the following 5 days, both of the two values fluctuate within a small range. By contrast, significant differences in L^* and *CIRG* are presented in bayberries nearly for the whole storage period (P < 0.05). The $L*$ and $CIRG$ of bayberry are 24.99 and 3.90 in day 6. Similar changes in $L*$, H, and C are also reported by Ali et al. in papaya [27]. Changes in color suggest that Chinese bayberries get ripe with the increase of storage time. These results are supported by Zheng et al. [31] and agree well with the HSE results.

(The preferred position of Table 2)

3.4 pH measurement results

Acidity is an indispensible chemical index that reflects the taste of fruit. Changes in pH of Chinese bayberry during cold storage are displayed in Fig. 4. Bayberries show a pH value of about 2.30 in day 0 and they have a minor increase in pH with the first 2 days. Afterwards, the pH values of bayberry increase rapidly, and significant differences are presented (P < 0.05). Bayberry's pH value is recorded as 2.752 after 6 days of storage. The main organic acids existing in Chinese bayberries are malic acid and citric acid [32]. During postharvest storage, living cells still conduct normal respiratory metabolism by consuming organic acids and other materials [29,33], which is the main cause for the increase in pH. On the other hand, oxidation resulted from oxygen also contributes to this change. This finding is in consistent with the report of Zheng et al. [31].

(The preferred position of Fig. 4)

3.5 TSS measurement results

TSS is one of the most important chemical indexes utilized to evaluate internal quality of fruit. Fig. 5 shows the variation of TSS of Chinese bayberry during 6 days of cold storage. The TSS of Chinese bayberry is 8.8% in day 0 and a minor increase in TSS is observed after 1 day of storage. In the following 5 days, the TSS in bayberries decreases significantly (P < 0.05). The TSS retention is merely 6.6% at the end of storage period. The decrease in TSS of Chinese bayberry obtained in this research agrees well with previous reports [31,33]. This change may be explained by the decomposition of relevant components due to strong respiratory metabolism. Although the solubilization of cell-wall polyuronides and hemicelluloses can improve TSS content [34], this effect is relatively weak and therefore cannot make up for the considerable loss of TSS. Similar findings are also reported by Wang et al. in apricot [35].

(The preferred position of Fig. 5)

3.6 RSC examination results

RSC is an essential indicator that has been widely used for determining the reducing capacity of fruit. Changes in RSC of Chinese bayberry during cold storage for 6 days are displayed in Fig. 6. The RSC value of bayberry is 2.31 g/100g in day 0, followed by a significant increase with the first 3 days ($P < 0.05$). Afterwards, the RSC of bayberries presents a decrease trend as the storage time advanced, and the final RSC value is recorded as 2.82 g/100g. A possible explanation for the increase in RSC in the early stage lies in the degradation of polysaccharides due to enzymatic catalysis [33]. However, the occurrence of respiratory consumption and oxidation decomposition becomes dominant when fruit enters into late maturity period and thereby, induces the significant decline of RSC [31,33].

(The preferred position of Fig. 6)

3.7 E-nose measurement results and bayberry quality forecasting model

3.7.1 E-nose original responses and PCA results

In order to evaluate the time drift of the sensors included in the e-nose, e-nose measurements of blank samples are conducted. Blank sample is the zero gas. The air is filtered by active carbon to obtain zero gas. Every day, e-nose responses to blank sample are measured to evaluate the sensors' time drift. The measurements last for eight days. Fig. 7(a) displays the sensors' responses to blank sample as function of measurement time on day0. The results indicate that the responses of sensors to zero gas are stable during measurement. Fig. 7(b) displays the e-nose sensors' saturated values to blank sample as function storage time from day0 to day8. Each day, the standard deviations of eight sensors are small enough. For each sensor, the saturated values are almost the same. Results indicate that the sensors' time drift during nine days' measurement has no influence to e-nose detection.

In order to evaluate the sample drift of sensors, e-nose measurements on samples stored for different storage time from day0 to day8 have been conducted. Fig. 7(c) shows the maximal errors of sensor responses during measurement from day0 to day8. Each day, the maximal errors of eight sensors are tolerable relative to the absolute values of sensors' responses. For different sensors, the maximal errors are all less than 0.5%, which indicate that the sample drift can be neglected in measurement.

So, these two groups' experiments demonstrate that the e-nose instrument was stable during the period or perform such measurements.

E-nose original responses to bayberry samples are displayed in Fig. 7(d) in day0. The volatile gases existing in the headspace of samples are inhaled into e-nose gas chamber and sensed by the functional materials settled in gas sensors. The specific absorption of function materials for specific gas species induces materials' changes in their electrical characteristics. So signals induced by electrical changes can be used to characterize gas concentrations. Sensor S1 presents the maximal responses, which indicates that there is amount of volatile gas containing sulfide in sample headspace. Sensor S7, S3, S6, and S5 also have sufficient responses, which suggest that there are some alkane, ammonia, hydrocarbon, and other reducing gases within the sample headspace. Sensor S4, S8, and S2 present weak responses, suggesting that there are little ethanol, nitrogen oxides, or flammable gases in the sample headspace. Eight gas sensors have different responses due to their different sensing abilities for specific gas species. The M.O.S sensors rely on changes in conductivity induced by the adsorption of molecules in the gas phase and on subsequent surface reactions. There is no equivalent circuit or impedance spectrum in e-nose system. So there is no Nyquist plot in this study [6,8,9]. So e-nose sensor array forms different responding pattern for bayberries under different storage days.

All sensors' initiative responses are close to zero. Sensors' responses gradually increase and finally reach their stable values. Sensor S1 presents the maximal stable value (about 1.7V). S7's stable value is about 1.3V. S3, S6, and S5 are similar, and their stable values are much lower than S1 and S7's. While the rest three sensors (S4, S8, and S2) present weak responses to samples.

PCA analysis results of bayberries under different storage days at 4℃ are displayed in Fig. 7(e). The first principal components (PC1) and the second principal components (PC2) capture 92.11% of data variance. The first two principal components of bayberries fluctuate during storage. Although bayberries stored for 0, 3, and 4 days have obvious discrimination borders, those stored for 1, 2, 5, and 6 days get together and cannot be well discriminated from each other. Therefore, bayberries stored under different storage days cannot be qualitatively or quantitatively discriminated by the two-dimensional PCA.

(The preferred position of Fig. 7)

3.7.2 SR and DCSSR SNR spectrum

Bayberry e-nose measurement data SNR spectrum calculated by SR as function of external noise intensity is displayed in Fig. 8(a). Derivative vales arise before the formation of eigen peaks for bayberry samples under different storage days. After that, SNR value increases gradually with the increase of white noise intensity. Eigen peak appears at noise intensity of 208. The SNR-Max values range between -65 and -50 dB. SNR-Max values increase with the increase of storage time. Bayberries under different storage days can be discriminated using SNR-Max values.

Bayberry e-nose measurement data SNR spectrum calculated by DCSSR method as function of storage time is displayed in Fig. 8(b). Obvious derivative vales appear at noise intensity of 83. Then SNR value increases gradually with the increase of noise intensity. Eigen peaks appear at noise intensity of 134. The SNR-Max values range between -56 and -45 dB. SNR eigen values increase with the increase of storage days. Bayberries of different quality can be discriminated according to SNR-Max values.

(The preferred position of Fig. 8)

3.7.3 MVR between physical-chemical indexes and SR/DCSSR eigen values

Quality is a general description for fruits. The physical-chemical indexes including firmness, pH, *CIRG*, TSS, and RSC have significant changes during storage. These indexes reflect internal quality of Chinese bayberries, while flavor is an external quality signal. If the relationship between bayberry internal quality and e-nose responses can be quantitatively modeled, it will provide a novel strategy for bayberry quality prediction. MVR results between physical-chemical indexes and SR SNR-Max values are displayed as Equation (15) and Table 3. $R^2 = 0.9992$ demonstrates that SR eigen values have good linearity with physical-chemical indexes. P= 0.01789 <0.05 and F=1800.95668, which indicate that SR SNR-Max values present significant linearity relation with physical-chemical indexes. SR can be used to characterize bayberries' quality.

$$\begin{aligned} SR &= 91.1595 - 0.3134 \times Firmness + 1.6226 \times CIRG \\ &-19.5706 \times pH - 680.8790 \times TSS - 129.053 \times RSC \end{aligned} \quad (R^2 = 0.9992) \qquad (15)$$

(The preferred position of Table 3)

MVR results between physical-chemical indexes and DCSSR SNR-Max values are displayed as Equation (16) and Table 4. $R^2 = 0.9944$ demonstrates that DCSSR eigen values have better linearity with bayberry quality indexes. P= 0.12819 >0.05 and F=34.67745, which show that DCSSR eigen values have no significant linearity relation with physical-chemical indexes. DCSSR SNR-Max values are not suitable for bayberry quality characterization.

$$\begin{aligned} DCSSR &= -23.6630 - 0.0461 \times Firmness + 0.1550 \times CIRG \\ &+ 0.0476 \times pH - 181.9790 \times TSS - 154.7910 \times RSC \end{aligned} \quad (R^2 = 0.9944) \quad (16)$$

(The preferred position of Table 4)

In section 2.5.3, we discuss DCSSR model. If two bistable SR systems are cascaded in serial, the intrinsic noise reducing efficiency can be improved to some extent. However, in Chinese bayberry quality analysis occasions, SR SNR spectrum eigen values have more significant linearity relation with fruit's physical-chemical indexes than DCSSR SNR eigen values. A possible explanation lies in bayberry quality feature information loss induced by DCSSR noise reduction procedure. When e-nose measurement data passes through 1st SR system, most of intrinsic noise has been restrained, and its energy has been transferred into feature information, which effectively amplifies the feature information. When the output signal of 1st SR system passes through 2nd SR system, the remained weak intrinsic noise is further reduced, and its energy is also transferred into feature information. However, partial feature information is also restrained by 2nd SR system. Finally, the effectiveness of 2nd SR system for intrinsic noise reduction relies on the dynamic balance between intrinsic noise reduction and partial feature information inhibition. If the former procedure holds superiority, 2nd SR presents its advantage in noise reduction. If the latter procedure holds superiority, 2nd SR presents its disadvantage. In this research, it is obvious that the latter procedure is on the ascendant in Chinese bayberry quality analysis. So SR is more suitable for bayberry quality analysis.

3.7.4 Chinese bayberry quality forecasting model

Fig. 9 shows the individual SNR-Max value of samples under different storage time. Bayberry quality predicting model is established by linear fitting regression of SNR-Max values. The fitting results are displayed as Eq. (17), and the regression coefficients R= 0.98644. After one-step's transform, Eq. (18) is used as bayberry quality forecasting model. The input is e-nose measurement data SR processed SNR-Max values, and the output is the bayberries' storage time. According to Equation (18) and physical-chemical examination results, the approximate quality of the measured samples can be obtained. This method is suitable for most of samples following the bayberry quality changing discipline. It can not be used as quality forecasting for special ones.

$$y = -64.27206 + 1.73297x \text{, } R = 0.98644 \tag{17}$$

$$Quality_{bayberry} = \frac{SNRMax + 64.27206}{1.73297} \tag{18}$$

(The preferred position of Fig. 9)

3.7.5 Validation experiment results

Validating experiments are carried out to examine forecasting accuracy of the developed model. Eq. (18) is used to examine the freshness of bayberries. Another 70 samples are prepared, and 21 samples are randomly taken out for e-nose measurement. The results are displayed in Table. 5. The forecasting accuracy of this model is 95%. Validating experiment results demonstrate that the developed predicting model detects the samples' quality with good accuracy and repeatability.

(The preferred position of Table 5)

4. Conclusions

Chinese bayberry (*Myrica Rubra Sieb. Et Zucc.*) quality rapid determination method using e-nose and non-linear SR technique has been investigated in this paper. Bayberry samples are stored at 4℃

temperature for continuous 7 days. E-nose responses to bayberries stored at 4℃ for different storage days are measured. Meanwhile, some physical-chemical indexes including HSE, texture, color, pH, TSS, and RSC, are examined to characterize samples' quality. E-nose measurement data is processed by PCA, SR and DCSSR methods. PCA only gives a qualitative discrimination for bayberry samples. SNR spectrum calculated by SR and DCSSR discriminates samples successfully. SNR eigen peak values increase with the increase of storage days. MVR between physical-chemical indexes (firmness, pH, *CIRG*, TSS, and RSC) and SR/DCSSR output SNR-Max values have been conducted. Regression results indicate that SR eigen values have more significant relation with physical-chemical indexes. SR is more suitable for Chinese bayberry quality characterization than DCSSR. Bayberry quality predicting model $Quality_{bayberry} = \frac{SNRMax + 64.27206}{1.73297}$ (*R=0.98644*) is developed via linear fitting regression on SR SNR-Max values. Validating experiments results demonstrate that the developed model presents a predicting accuracy of 95% for Chinese bayberry quality.

Acknowledgements

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Conflict of interest

Ying Xiaoguo declares that he has no conflict of interest. Liu Wei declares that he has no conflict of interest. Cai Yanping declares that he has no conflict of interest. Jin Jiaojiao declares that he has no conflict of interest. Hui Guohua declares that he has no conflict of interest.

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Collected Table and Figure captions in sequence:

Table 1 HSE scheme for evaluating the Chinese bayberry during storage

Table 2 Color measurement and CIRG calculation of Chinese bayberries during storage at 4 □[a,b]

Table 3 Multiple variable regression between SR eigen values and physical-chemical indexes (F= 246.9722, P=0.0483, regression of SS (sum of squares) =397.2905, regression of MS (mean square) =79.4581, residual of SS= residual of MS=0.3217)

Table 4 Multiple variable regression between DCSSR eigen values and physical-chemical indexes (F= 35.6078, P=0.1265, regression of SS (sum of squares) =27.8097, regression of MS (mean square) =5.5619, residual of SS= residual of MS=0.1562)

Table 5 Bayberry quality predicting results (√ right; × wrong; / not calculated)

Fig. 1 Schematic diagram of detecting system: (a) e-nose; (b) M.O.S gas sensor; (c) M.O.S gas sensor detecting mechanism; (d) e-nose chamber design; (e) DCSSR

Fig. 2 HSE results. Each data point is the mean of five replicates. Vertical bars represent standard deviation of means. Different letters on the same sampling day indicate significances ($p < 0.05$).

Fig. 3 Texture measurement results: (a) firmness; (b) resilience; (c) cohesiveness. Each data point is the mean of five replicates. Vertical bars represent standard deviation of means. Different letters on the same sampling day indicate significances ($p < 0.05$).

Fig. 4 pH measurement results. Each data point is the mean of five replicates. Vertical bars represent standard deviation of means. Different letters on the same sampling day indicate significances ($p < 0.05$).

Fig. 5 TSS measurement results during storage. Each data point is the mean of five replicates. Vertical bars represent standard deviation of means. Different letters on the same sampling day indicate significances ($p < 0.05$).

Fig. 6 Reducing sugar measurement results. Each data point is the mean of five replicates. Vertical bars represent standard deviation of means. Different letters on the same sampling day indicate significances ($p < 0.05$).

Fig. 7 E-nose original responses and PCA results: (a) e-nose to blank samples; (b) e-nose saturated values to blank sample; (c) e-nose sensor array saturated value to samples; (d) e-nose original responses; (e) e-nose PCA results.

Fig. 8 SNR spectrum calculated by (a) SR and (b) DCSSR respectively.

Fig. 9 Chinese bayberry quality predicting model using SR SNR-Max values.

Table 1 HSE scheme for evaluating the Chinese bayberry during storage

Attributes	Attribute degree				
	5	4	3	2	1
Color	Red	Slight purple	Purple	Dark purple	Purple brown
Skin wetness	Very dry	Dry	Slight wet	Wet	Very wet
Touch	Very hard	Hard	Slight soft	Soft	Very soft
Taste	Delicious	No off-flavor	Slight off-flavor	Off-flavor	Strong off-flavor
Odor	Fruity	Silight fruity	Slight vinosity	Vinosity	Strong vinosity
Maturation	Strong crudeness	Crudeness	Slight maturation	Maturation	Strong maturation

Table 2

Storage time (d)	L^*	H	C	$CIRG$
0	12.63±0.28 a	-0.86±0.41 a	9.73±0.70 a	8.10±0.32 d
1	16.17±0.16 b	0.18±0.03 c	23.25±1.30 b	4.57±0.15 c
2	17.35±0.43 c	0.18±0.02 bc	22.61±2.74 b	4.51±0.26 c
3	19.39±0.25 d	0.25±0.01 d	23.60±3.36 b	4.20±0.33 ab
4	20.33±0.13 e	0.14±0.03 b	22.49±1.88 b	4.21±0.19 b
5	22.00±0.67 f	0.12±0.03 b	20.86±1.46 b	4.20±0.13 b
6	24.99±0.38 g	0.16±0.10 bc	21.20±1.56 b	3.90±0.11 a

[a] Mean of five replications ± standard deviation.

[b] Means in same row with different letters are significantly different ($p < 0.05$).

Table 3

Index	Value	Standard error	T Stat	P
Y-Intercept	91.1595	48.8990	1.8642	0.3134
Firmness	-0.3134	0.0739	-4.2422	0.1474
CIRG	1.6226	0.8105	2.0020	0.2949
pH	-19.5706	12.4436	-1.5728	0.3605
TSS	-680.8790	153.9114	-4.4238	0.1415
RSC	-129.0530	70.5743	-1.8286	0.3186

Table 4

Index	Value	Standard error	T Stat	P
Y-Intercept	-23.6630	34.0718	-0.6945	0.6136
Firmness	-0.0461	0.0515	-0.8957	0.5350
CIRG	0.1550	0.5647	0.2744	0.8295
pH	0.0476	8.6704	0.0055	0.9965
TSS	-181.979	107.2423	-1.6969	0.3390
RSC	-154.7910	49.1747	-3.1478	0.1958

Table 5 Bayberry quality predicting results (√ right; X wrong; / not calculated)

Sample No.	SNR-Max	Predicting value	Storage days	Error (%)	Results
evad-1	-62.5893	0.971	1	2.9	√
evad-2	-55.2364	5.214	5	4.28	√
evad-3	-59.1095	2.979	3	0.7%	√
evad-4	-64.0554	0.125	0	/	X
evad-5	-60.9673	1.907	2	4.65	√
evad-6	-53.6368	6.137	6	2.28	√
evad-7	-57.16	4.104	4	2.6	√
evad-8	-62.4576	1.047	1	4.7	√
evad- 9	-59.2516	2.897	3	3.43	√
evad-10	-55.2121	5.228	5	4.56	√
evad-11	-54.2798	5.766	6	3.9	√
evad-12	-58.8843	3.109	3	3.63	√
evad-13	-60.7056	2.058	2	2.9	√
evad-14	-64.2495	0.013	0	/	√
evad-15	-54.1671	5.831	6	2.82	√
evad-16	-55.9555	4.799	5	4.02	√
evad-17	-58.8739	3.115	3	3.83	√
evad-18	-57.6417	3.826	4	4,35	√
evad-19	-53.3925	6.278	6	4.63	√
evad-20	-55.2883	5.184	5	3.68	√
evad-21	-58.8496	3.129	3	4.3	√

Fig. 1(a)

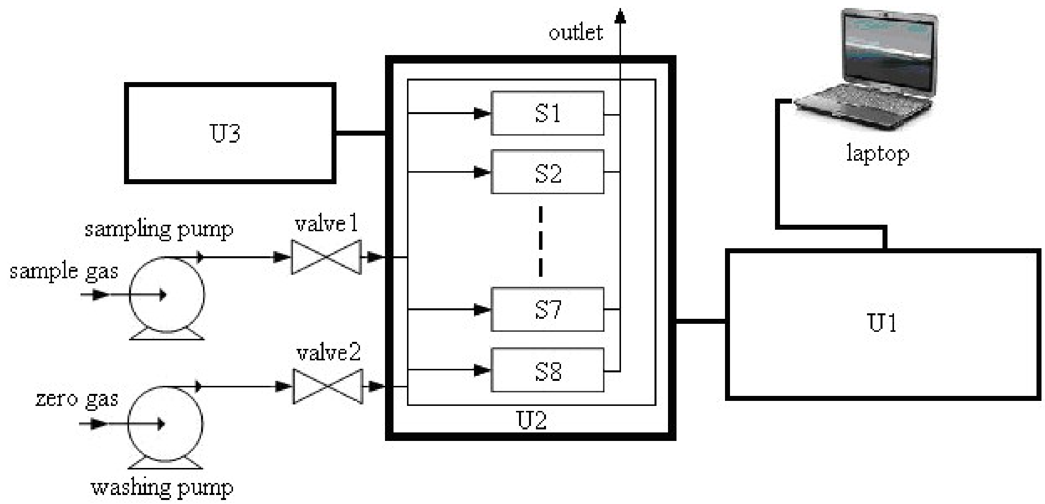


Fig. 1(b)

Fig. 1(c)

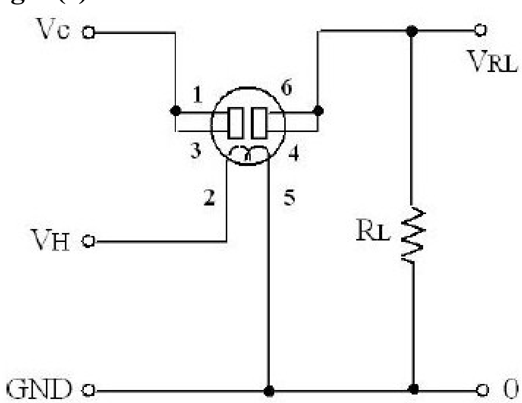


Fig. 1(d)

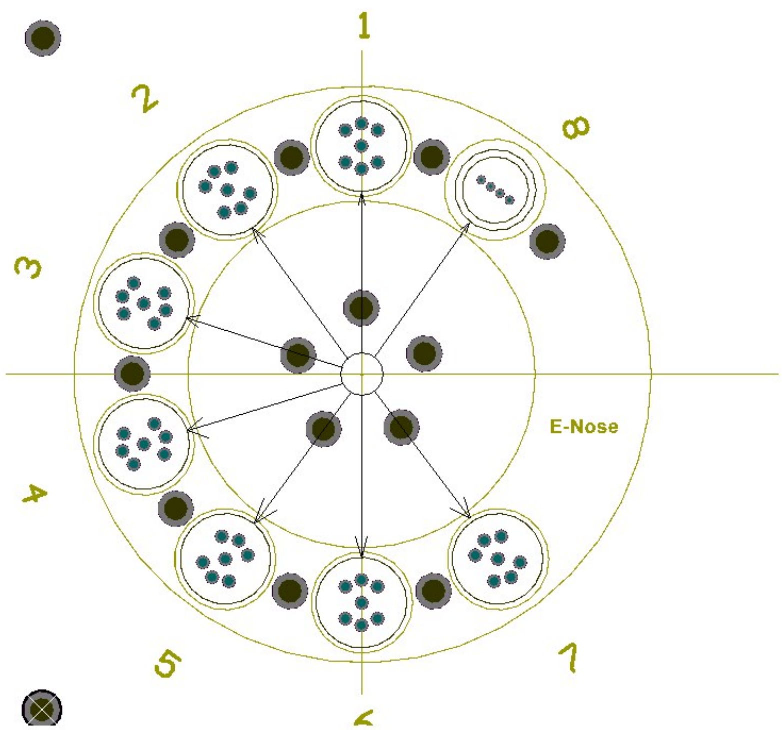

1
2
8
3
E-Nose
4
5
7

Fig. 1(e)

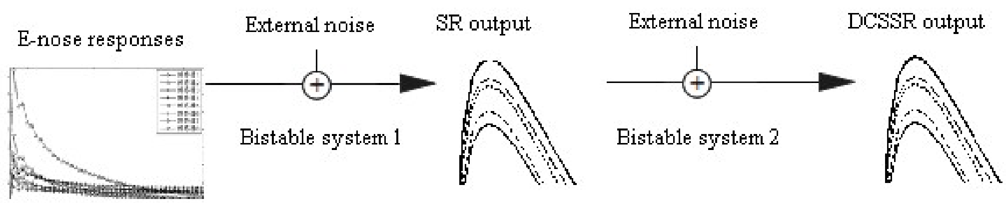

E-nose responses
External noise
SR output
External noise
DCSSR output
Bistable system 1
Bistable system 2

Fig. 2

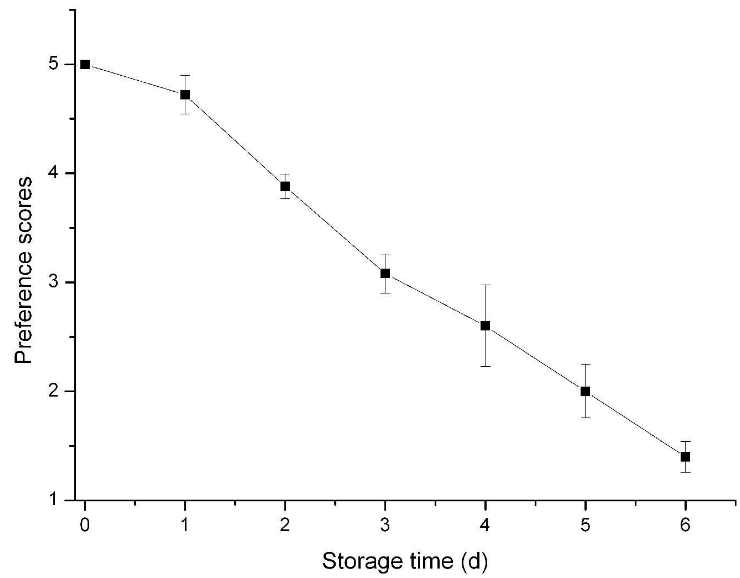

Preference scores
Storage time (d)
1
2
3
4
5
0
1
2
3
4
5
6

Fig. 3(a)

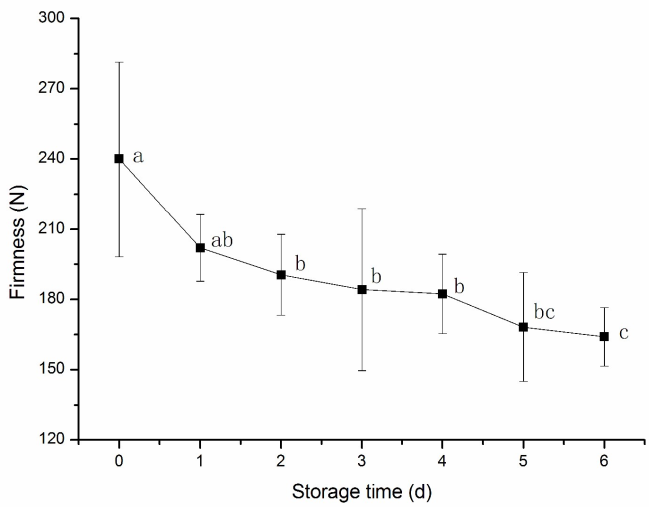

Firmness (N)
Storage time (d)
300
270
240
210
180
150
120
0
1
2
3
4
5
6
a
ab
b
b
b
bc
c

Fig. 3(b)

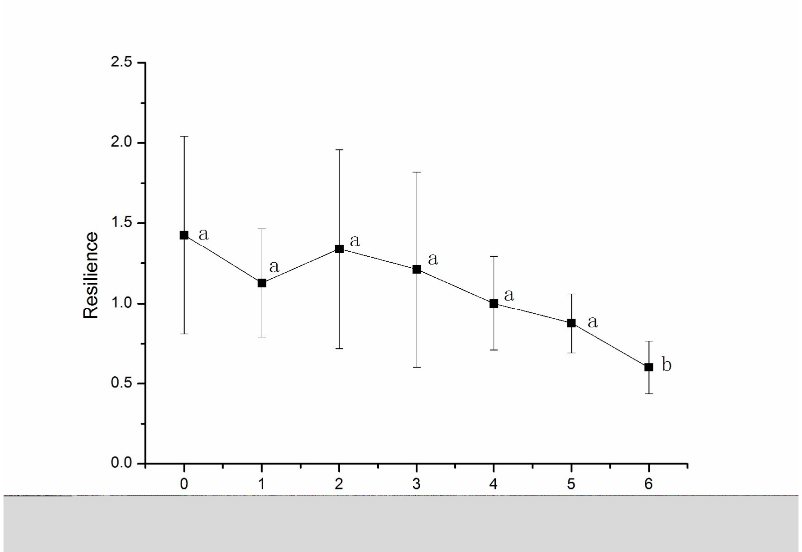

Resilience
2.5
2.0
1.5
1.0
0.5
0.0
0
1
2
3
4
5
6
a
a
a
a
a
a
b

Fig. 3(c)

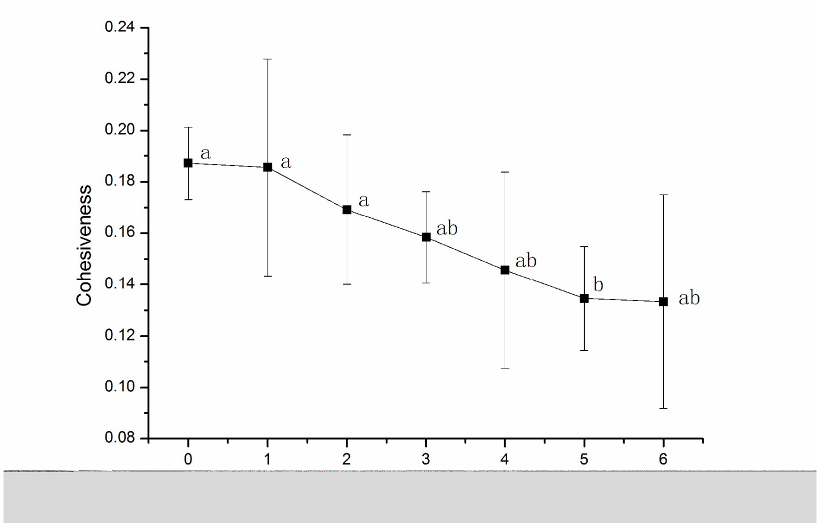

Cohesiveness
0.24
0.22
0.20
0.18
0.16
0.14
0.12
0.10
0.08
0
1
2
3
4
5
6
a
a
a
ab
ab
b
ab

Fig. 4

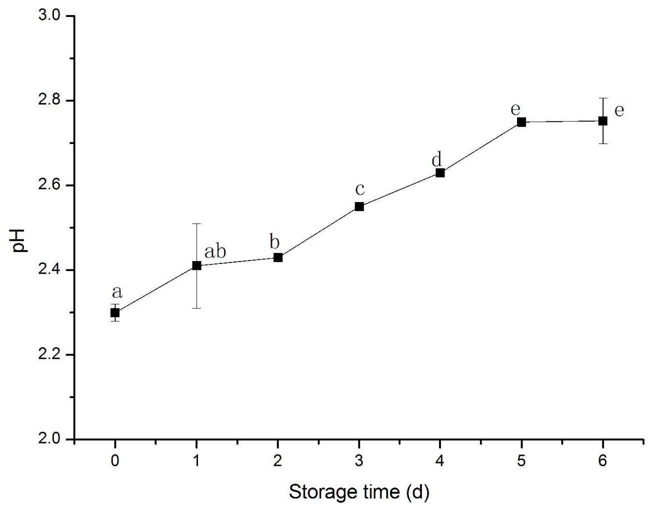

pH
3.0
2.8
2.6
2.4
2.2
2.0
0 1 2 3 4 5 6
Storage time (d)
a
ab
b
c
d
e
e

Fig. 5

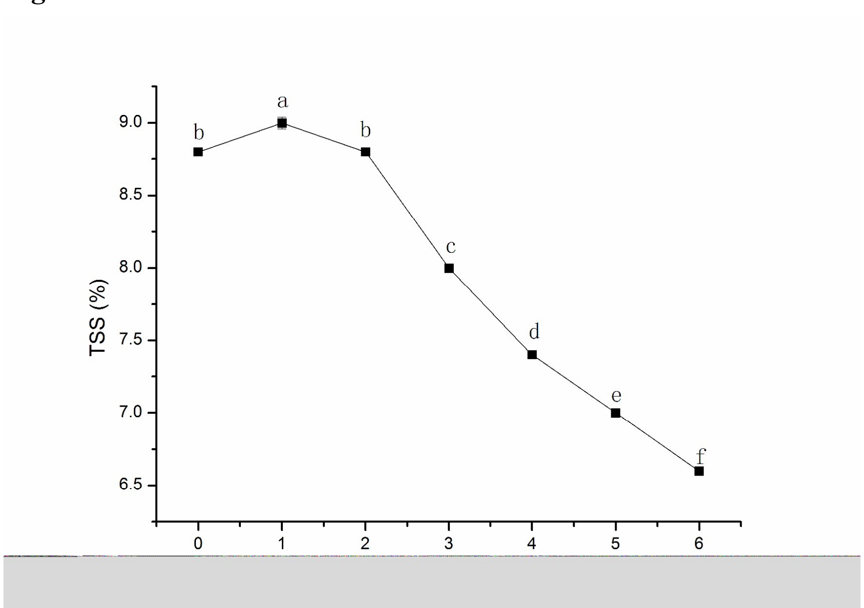

TSS (%)
9.0
8.5
8.0
7.5
7.0
6.5
0 1 2 3 4 5 6
b
a
b
c
d
e
f

Fig. 6

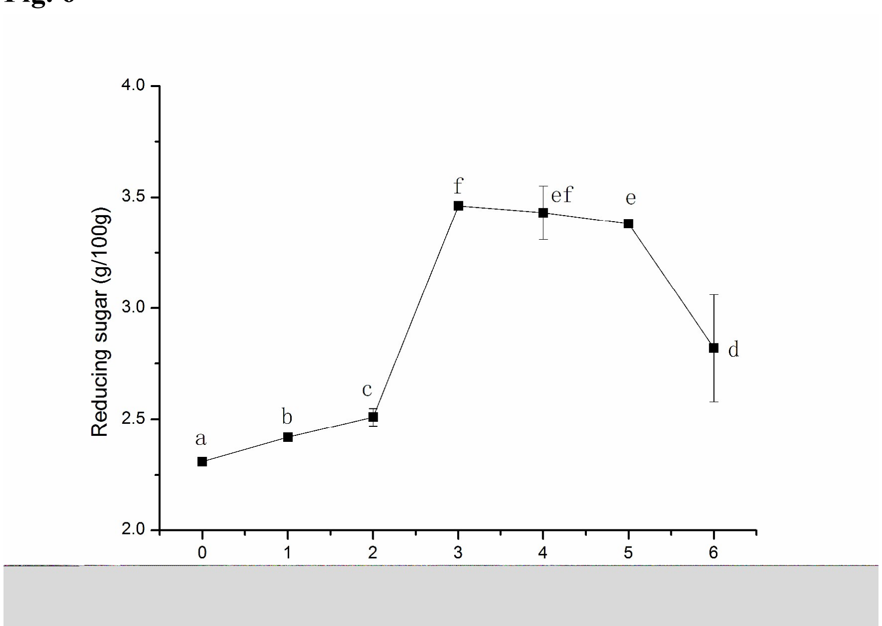

Reducing sugar (g/100g)
4.0
3.5
3.0
2.5
2.0
0
1
2
3
4
5
6
a
b
c
f
ef
e
d

Fig. 7(a)

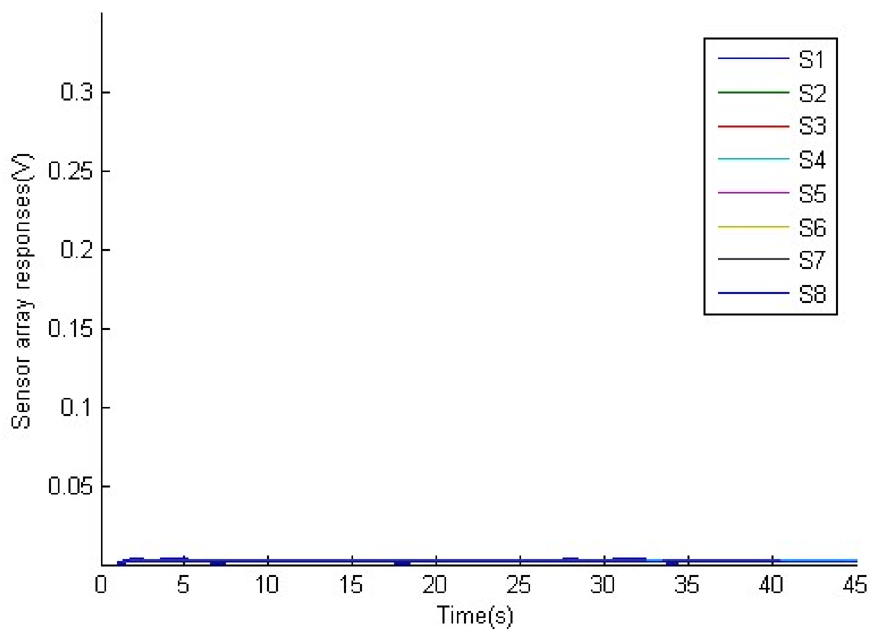

S1
S2
S3
S4
S5
S6
S7
S8
Sensor array responses(V)
0.3
0.25
0.2
0.15
0.1
0.05
0
5
10
15
20
25
30
35
40
45
Time(s)

Fig. 7(b)

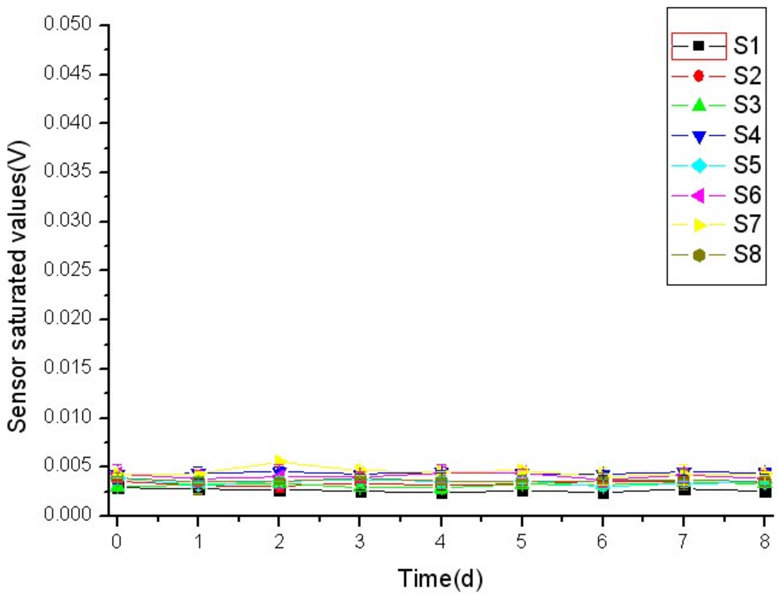

S1
S2
S3
S4
S5
S6
S7
S8
Sensor saturated values(V)
Time(d)

Fig. 7(c)

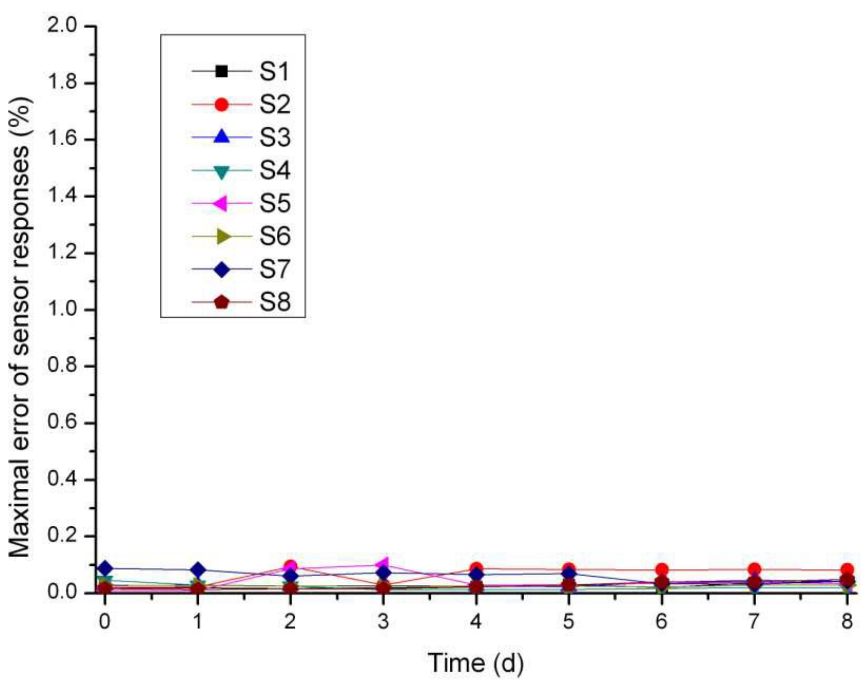

S1
S2
S3
S4
S5
S6
S7
S8
Maximal error of sensor responses (%)
Time (d)

Fig. 7(d)

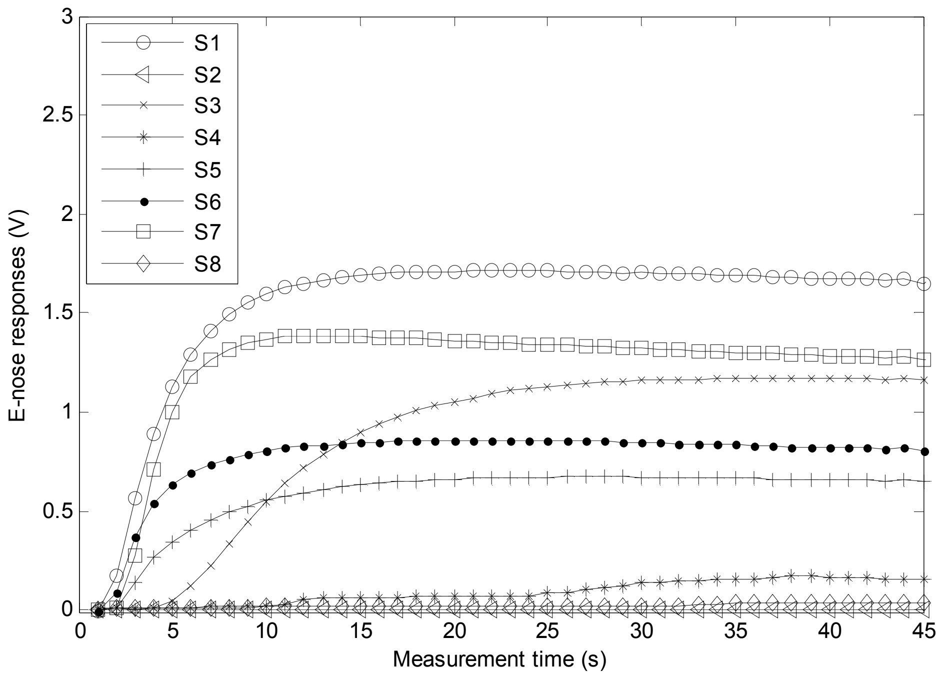

S1
S2
S3
S4
S5
S6
S7
S8
E-nose responses (V)
Measurement time (s)
0
0.5
1
1.5
2
2.5
3
0
5
10
15
20
25
30
35
40
45

Fig. 7(e)

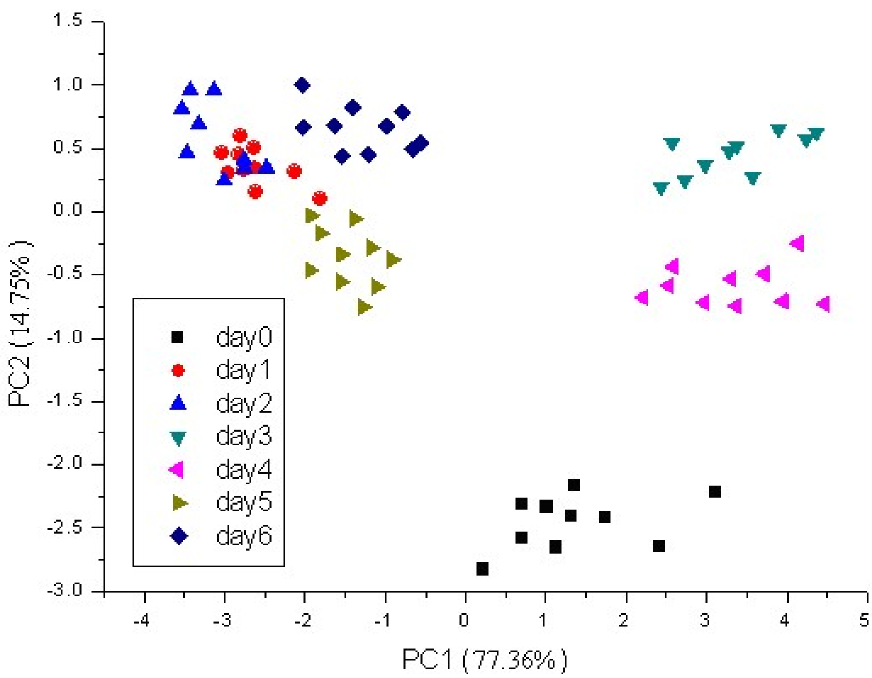

day0
day1
day2
day3
day4
day5
day6
PC2 (14.75%)
PC1 (77.36%)
1.5
1.0
0.5
0.0
-0.5
-1.0
-1.5
-2.0
-2.5
-3.0
-4
-3
-2
-1
0
1
2
3
4
5

Fig. 8(a)

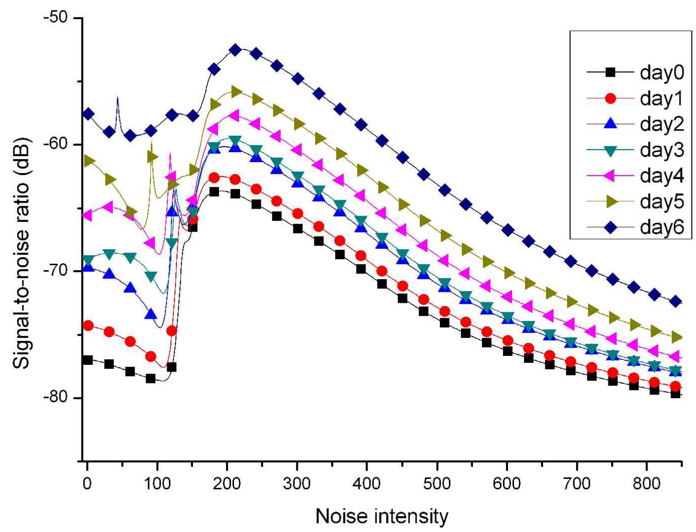

Signal-to-noise ratio (dB)
Noise intensity
-50
-60
-70
-80
0
100
200
300
400
500
600
700
800
day0
day1
day2
day3
day4
day5
day6

Fig. 8(b)

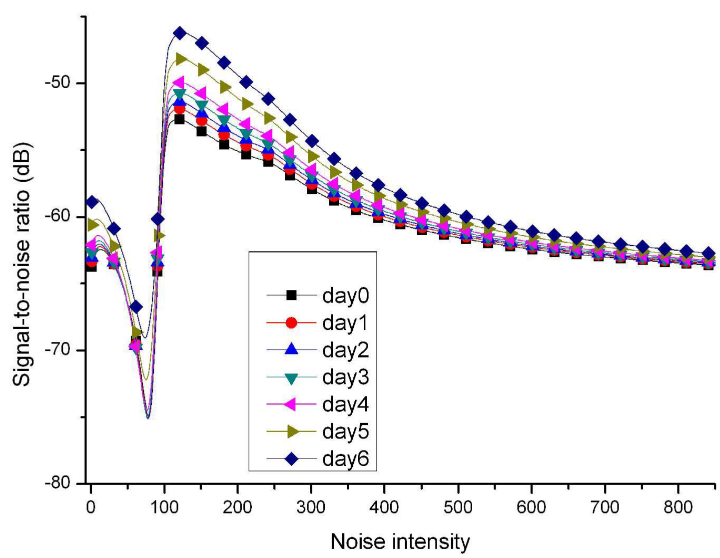
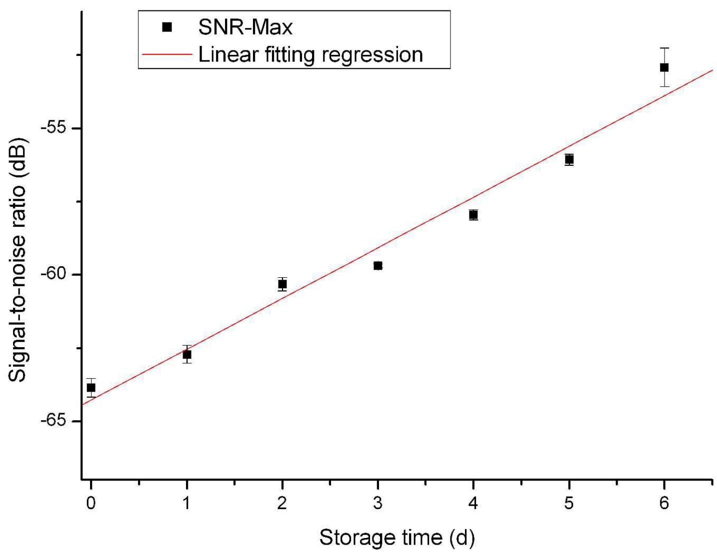
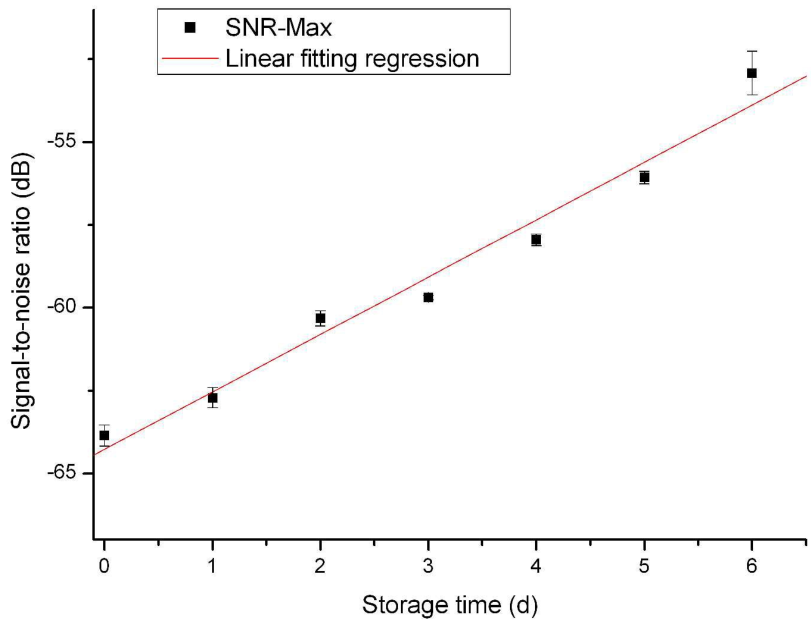

Signal-to-noise ratio (dB)
Noise intensity
-50
-60
-70
-80
0
100
200
300
400
500
600
700
800
day0
day1
day2
day3
day4
day5
day6

Fig. 9


SNR-Max
Linear fitting regression
Signal-to-noise ratio (dB)
-55
-60
-65
0
1
2
3
4
5
6
Storage time (d)



Bayberry quality predicting model $Quality_{bayberry} = \frac{SNRMax + 64.27206}{1.73297}$ (*R=0.98644*) is developed via linear fitting regression on SR SNR-Max values. Validating experiments results demonstrate that the developed model presents a predicting accuracy of 95% for Chinese bayberry quality.