Kiwi fruit (Actinidia chinensis) quality determination based on surface acoustic wave resonator combined with electronic nose

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In this study, electronic nose (EN) combined with a 433 MHz surface acoustic wave resonator (SAWR) was used to determine Kiwi fruit quality under 12-day storage. EN responses to Kiwi samples were measured and analyzed by principal component analysis (PCA) and stochastic resonance (SR) methods. SAWR frequency eigen values were also measured to predict freshness. Kiwi fruit sample's weight loss index and human sensory evaluation were examined to characteristic its quality and freshness. Kiwi fruit's quality predictive models based on EN, SAWR, and EN combined with SAWR were developed, respectively. Weight loss and human sensory evaluation results demonstrated that Kiwi fruit’s quality decline and overall acceptance decrease during the storage. Experiment result indicated that the PCA method could qualitatively discriminate all Kiwi fruit samples with different storage time. Both SR and SAWR frequency analysis methods could successfully discriminate samples with high regression coefficients (R = 0.98093 and R = 0.99014, respectively). The validation experiment results showed that the mixed predictive model developed using EN combined with SAWR present higher quality prediction accuracy than the model developed either by EN or by SAWR. This method exhibits some advantages including high accuracy, non-destructive, low cost, etc. It provides an effective way for fruit quality rapid analysis.

Introduction

Kiwi fruit (Actinidia chinensis) is a valuable source of vitamins,1-2 fats, proteins, amino acids, dietary fibers and rich minerals (such as calcium, iron, pectin, etc). It is widely cultured in south Asia and Southeast Asia and China is its main producing area. Apart from its edible and medicinal values, the emodin extracted from its root has broad applications in medicine, health care, and other fields.3 The leaching solution extracted from its branches is a good source of adhesive colloid.4-6 Moreover, due to its unique ability for human to regulate emotion and enhance appetite, Kiwi fruit is very suitable for patients suffering from gastric, hypertension and other diseases.

Ripe Kiwi and other fruits are more vulnerable to many factors from both environment and itself. As a result, fruit quality decline or even rot occurs during the storage.7,8 Human sensory evaluation method can directly discriminate Kiwi fruits with different qualities. However, its result is often affected by various factors, such as individual preference, health situation, physiological age, etc. Physical/chemical examining method, such as firmness, microbial, etc, effectively reveals fruit's quality condition. Nevertheless, these measurements cover some drawbacks including fuzzy operation, low repeatability, high cost, and too much time consuming, etc, which makes it unsuitable for rapid quality analysis.9-11 Instrument analysis method, such as gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS), presents food quality with precise analysis abilities. However, some disadvantages exist in these methods, such as high cost, time consuming, etc. In addition, only skilled operators are required to perform the instrumental analytical experiments.12 So, there is an urgent demand for developing a rapid quality analysis method with quick response, high accuracy, and low cost in fruit area.

Surface acoustic wave (SAW) is first proposed in 1970s and it provides the basis for making highly integrated devices with small size and high sensitivity.13-18 So far, there are many reports about detection applications about SAW in many fields especially in food analysis, such as monitoring of the growth of bacteria, detection of pancreatic lipase, and biomedical analysis, etc.19-22 Currently, more and more researches focus on the chemical/biological modification about surface acoustic wave resonator (SAWR). If specific reaction (such as antigen-antibody, receptor-ligand interaction, etc) occurs during the processing, some information about wave (velocity, for example) will change accordingly when SAW passes the piezoelectric substrate resonator, which realizes the characterization about test sample's species and concentration. However, there are also some defects within this technique, to illustrate, SAW...
devices are often used only one time after modifying, which results in high testing cost.\textsuperscript{23-25}

EN technique, simulating human’s olfactory system, is a method regarding odor fingerprint detection.\textsuperscript{26} A traditional EN system consists of 3 main functional parts: gas sensor array, signal preprocessing and pattern recognition. It chooses gas as analysis object and obtains characteristic signal. It could real-time capture and examine aromatic substances from specific positions, so it is called EN figuratively. Because of its particular functions, it has applied to food, makeup, petrochemicals, packaging material, environmental inspection, clinic, chemistry, etc.\textsuperscript{27-29}

In this study, EN combined with a 433 MHz SAWR was utilized to examine the responding signals of Kiwi fruit with different storage time. Human sensory evaluation and weight loss examinations were performed to characteristic Kiwi fruit’s freshness. The PCA method could qualitatively discriminate Kiwi samples with different storage days. Both SR and SAWR frequency analysis methods could discriminate all samples with high regression coefficients. The validation experiment result demonstrated that the built predictive model based on EN combined with SAWR present higher prediction accuracy than the model built based on EN or SAWR. The proposed method in this research takes some unique advantages including fast response, non-destruction, high accuracy, etc. The proposed method is promising in fruit quality analysis.

**Results and Discussion**

**Human sensory evaluation**

Kiwi fruit’s human evaluation result is shown in Figure 1a. Kiwi fruit sample’s initial score is set at score of 5 and score of 3 is regarded as the limit of overall acceptance. There is no obvious change observed in Kiwi fruit samples within the first 4 days. After that, it exhibits a significant quality decline trend in the following days. In day 8, the preference score is 2.98 ± 0.13, which indicates that Kiwi fruit is severely spoiled and it loses commercial and edible values. Owing to the influences of microbial infection and self-physiological metabolism, Kiwi fruit’s quality changes significantly with the increase of storage time, including increasing losses of moisture, color, roughness and glossiness, softer touch, severer rot degree and much more cracks. Therefore, human sensory evaluation can classify Kiwi fruits with different qualities.

**Weight loss**

Kiwi fruit’s weight loss result is shown in Figure 1b. There is no significant change can be seen within the first 2 days. After that, it shows a continuous increase trend and reaches approximate 5% in day 12. During the storage, living cells in Kiwi fruit still precedes strong respiration along with some internal physical/chemical reactions. As a result, considerable moisture loses in Kiwi fruits, which results in Kiwi fruit’s weight loss increase with the increase of storage days. Some researches have also reported similar results in other fruits.\textsuperscript{30-32}

**Freshness predictive model based on SAWR measurement result**

SAWR frequency measurement is performed by following processing: first, connect Kiwi sample with SAWR system, then use frequency meter to collect its frequency value, next, transfer the data to a computer through a RS-232 communication interface. The data can be read real-time by self-PC software. The SAWR frequency detecting result is shown in Figure 2a. With the increase of storage time, Kiwi fruit’s SAWR frequency increases continuously. The initial frequency value is about 260 MHz, while it reaches about 440 MHz in day 9. Different frequency values are obtained, corresponding to different storage days. The result shows that the SAWR detecting system has high sensitivity toward Kiwi fruit samples with different storage time.

Due to the different dielectric characteristics of Kiwi fruits with different qualities, samples would significantly influence SAWR current frequency when it is in serial with SAWR circuit. According to equation (1), SAWR’s frequency value finally rises due to the significant increase of $R e$ and decrease of conductivity...
Although the dynamic capacitor parameter \(C_e\) also changes during the whole processing, it has weak impact on SAWR frequency responses than \(R_e\). So it can be neglected.

According to the result shown in Figure 2a, the relationship between output frequency \(Freq\) and storage time \(Time\) is obtained after linear-fitting and shown as equation (1):

\[
Freq = 264.70909 + 21.30909 \times Time \quad (R^2 = 0.98093) \quad (1)
\]

After one-time conversion to equation (1), Kiwi fruit’s freshness predictive model is acquired based on SAWR and expressed as equation (2):

\[
Time_{SAWR} = \frac{Freq - 264.70909}{21.30909} \quad (2)
\]

With the help of equation (2), it can realize the prediction about Kiwi fruit’s storage time based on SAWR system.

To validate the robustness of the predictive model, a batch of Kiwi fruit samples with unknown storage time was examined using SAWR system and the detected frequency value was set as true value. The predicting values were obtained by inputting the detected values into equation (2). The linear fitting result between predicting value and true value is shown in Figure 2b with regression coefficient \(R^2 = 0.865\), which demonstrates that SAWR cannot efficiently discriminate Kiwi fruit samples and partial samples bring about major errors.

The PCA result, freshness predictive model, and the validation experiment results based on EN

EN original responses to Kiwi fruit samples are shown in Figure 3a. The volatile gases existing in the headspace of samples are inhaled into EN gas chambers 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. And the responses rise accordingly with the growth of gas concentration. So signals induced by electrical changes can be used to characterize gas concentrations. What’s more, 8 gas sensors give different responses due to their different sensing abilities for specific gas species. So EN sensor array forms different responding pattern for Kiwi fruits with different storage days. EN system’s unique functions have been confirmed such as analysis of aroma compounds of commercial cider vinegars,\(^{33}\) composition of commercial truffle flavored oils,\(^{34}\) detection of adulteration in cherry tomato juices,\(^{35}\) etc.

All sensors’ initiative responses to Kiwi fruit are close to zero. All sensors’ response values increase gradually and finally reach individual stable value. Sensor S4 presents the maximal stable value (about 0.095 V). The final stable value of S1, S5, S7 and S6 is about 0.058, 0.028, 0.020 and 0.010 V, respectively, while the rest 3 sensors (S3, S8, and S2) present weak responses to all samples.

Kiwi fruit’s PCA result is shown in Figure 3b. The first principal components (PC1) and the second principal components (PC2) capture 91.06 % of data variance. Five sensors’ response values (S1, S4, S5, S6 and S7) to Kiwi fruit are chosen as sample’s whole freshness eigen values. Kiwi fruit samples with different storage days can be well distinguished by the PCA method. However, this method is not suitable for quantitative discrimination.\(^{36,37}\)

Kiwi fruit’s SNR spectrum calculated by SR as function of external noise intensity is shown in Figure 3c. Derivative values arise before the formation of eigen peaks for Kiwi fruit samples with different storage days. After that, SNR value increases gradually with the increase of stimulating noise intensity. Each eigen peak appears at noise intensity of 208 or so. Sample’ SNR-Max
value increases with the increase of storage time and ranges between $-80.00$ and $-64.00$ dB during the experiment. So, Kiwi fruit samples with different storage days can be successfully discriminated using SNR-Max eigen values.

For the further research, SNR-Max values are chosen as eigen values and equation (3) is obtained by linear-fitting. The result is shown in Figure 3d. Thus, predicting value of Kiwi fruit’s storage day ($Time_{e - n0se}$) can be calculated by inputting the SNR data.
into equation (4).

\[ y = -77.605 + 1.29768x \] \hspace{1cm} (R^2 = 0.99014) \hspace{1cm} (3)

\[ \text{Time}_{e-nose} = \frac{\text{MaxSNR} + 77.605}{1.29768} \] \hspace{1cm} (4)

Furthermore, a batch of Kiwi samples with unknown storage time was examined using EN system. Kiwi fruit’s SNR spectrum calculated by SR was input into equation (4) and the predicting values were calculated. The result is shown in Figure 3e with regression coefficient \( R^2 = 0.939 \), which indicates that the SNR spectrum can achieve the goal of freshness prediction about Kiwi fruits, but partial samples present low predicting accuracy.

**Freshness predictive model and the validation experiment results based on EN combined with SAWR**

Based on above 2 model’s properties, a new predictive model was proposed to predict Kiwi fruit’s storage time combining EN with SAWR system. Two confidence coefficients \( (P_1 \text{ and } P_2) \) were preset. By inputting \( Freq, SNR \) and \( Time \) values into equation (5), the result is that \( P_1 = 0.5153, P_2 = 0.4726 \). By inputting \( P_1 \) and \( P_2 \) values into equation (5), a mixed predictive model based on EN combined with SAWR is built and the result is shown in equation (6).

\[ \text{Time}(Freq, SNR) = P_1 \times \frac{Freq - 204.49196}{26.65642} + P_2 \times \frac{MaxSNR + 77.605}{1.29768} \hspace{1cm} (5) \]

\[ \text{Time}(Freq, SNR) = 0.5153 \times \frac{Freq - 204.49196}{26.65642} + 0.4726 \times \frac{MaxSNR + 77.605}{1.29768} \hspace{1cm} (6) \]

Next, a batch of Kiwi fruit samples with unknown storage time was examined using EN and SAWR system. A series of SNR and SAWR frequency eigen values were recorded and input into equation (6). The linear fitting relationship between predicting value and true value is shown in Figure 4. The regression coefficient \( R = 0.998 \), which suggests that EN in combination with SAWR system could better predict Kiwi fruit’s quality and freshness. In addition, the combination of EN and SAWR technique applied in many other areas also exhibits some significant benefits.

From the aspect of non-destructive detection, 3 predictive models were built to predict Kiwi fruit’s quality. SAWR detection result reflects Kiwi fruit’s internal information, while EN analysis result reveals Kiwi fruit’s external message, thus this combination could better deliver Kiwi fruit sample’s whole change during storage. This technique is promisingly used to guide fruit’s optimal harvest time, which is of great use to reduce economic loss due to fruit’s nutrition decreases. What’s more, we decide to conduct further research work in the near future and apply this technique for fruit’s best picking period judgment.

**Materials and Methods**

**Kiwi fruit samples**

Kiwi fruit samples were purchased from Gouzhuang wholesale fruit market (China, Hangzhou province). The samples were nearly in the same level of quality (such as size, weight, and

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**Table 1. Kiwi fruit’s sensory evaluation method**

| Attributes              | 5                  | 4                  | 3                  | 2                  | 1                  |
|-------------------------|--------------------|--------------------|--------------------|--------------------|--------------------|
| Outward integrity       | Firm               | Integral           | General integral   | Slight crack       | Much crack         |
| Touch                   | Very hard          | Hard               | Slight soft        | Soft               | Very soft          |
| Color                   | Dark green         | Green              | Slight green       | Slight yellow      | Yellow             |
| Moisture content        | Very abundant      | Abundant           | Modest             | Minor              | Less               |
| Glossiness              | Very bright        | Bright             | Slight dull        | Dull               | Very dull          |
| Roughness               | Very rough         | Rough              | Slight rough       | Smooth             | Very smooth        |
| Rot degree              | No rot             | Little rot         | Partly rot         | Most rot           | Badly rot          |
maturity) without any ripening pretreatment. All samples were stored at 4°C temperature. The experiments were conducted under the condition of normal temperature and atmospheric pressure.

Methods

Human sensory evaluation

As the method previously described,40 Kiwi fruit sensory evaluation was evaluated by 6 experienced panelists in our lab. Voting number is set at \( k, k \in (1,10) \). Kiwi fruit’s quality is divided into \( m \) levels, and the score of a specific level is set at \( h, j \in (1, m) \). Kiwi fruit attributes are divided into \( n \) elements, and a specific element is set at \( u, i \in (1,n) \). The contributory weight is determined by pairwise comparison of contribution weight of attributes is set at \( \sum x_i = 1 \). If there is a specific relationship between 2 objects of hand \( u \), the relation set (matrix) of \( f \) is calculated as follows:

\[
F = \begin{bmatrix}
    f_{11}/k & f_{12}/k & \cdots & f_{1m}/k \\
    f_{21}/k & f_{22}/k & \cdots & f_{2m}/k \\
    \vdots & \vdots & \ddots & \vdots \\
    f_{n1}/k & f_{n2}/k & \cdots & f_{nm}/k
\end{bmatrix}
\] (7)

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

\[
Z = \sum_{i=1}^{n} x_i \cdot \sum_{m=1}^{m} \frac{f_{ij}}{k} h_j
\] (8)

Kiwi fruit’s sensory evaluation method is shown in Table 1.

SAWR series of testing device

SAWR system and its load circuit were self-developed in our lab. As it shown in Figure 5a, ST cut type quartz was taken as the piezoelectric base material. And precision photolithographic process was conducted to make a 433 MHz high-frequency and single-ended SAWR. It’s outer size was 4.5mm × 11mm. Vacuum pack was adopted to package SAWR. Figure 5b is a schematic diagram of detecting system, which consists of SAWR and its load circuit, stabilized power supply (DF1741SB3A, Ningbo CSI Electronics Co., Ltd), and universal counter (EE 3386, Jiangsu New Union Technology Co., Ltd). The experiment was performed as following process: connecting Kiwi samples to SAWR circuit, putting them into a metal shield box, then capture the load frequency from SAWR by the universal counter and transfer it to the computer via RS232 communication interface for subsequent analysis.
Equivalent circuit model of SAWR in serial with Kiwi fruit samples is shown in Figure 5c, where $C_0$ is a static capacitor, $L_s, C_s$ and $R_e$ represent dynamic inductance, capacitance and resistance of SAWR, respectively. When Kiwi frit sample connect to SAWR, $C_e$ is an equivalent dynamic capacitor and $R_e$ is an equivalent dynamic resistance of sample. The frequency of SAWR loaded with Kiwi fruit sample can be calculated by following formula:

$$F = F_0 \left[ \frac{\pi F_0 C_s(2\pi F_0 C_s - Y e_e)}{G_e - \pi F_0^2 C_0 G_e Y + 4\pi^2 F_0^2 C_s(C_0 + C_e)} - \pi F_0 C_e R_s \right]$$

(9)

In equation (9), SAWR’s unloaded frequency $F_0 = 1/2\pi\sqrt{L_s C_0}$. $Y$ is amplification circuit’s phase parameter, analytic’s conductivity $G_e = 1/R_e$, electrode capacitance $C_e = \kappa_e + C_p$, where $\kappa$ is a permittivity, and $C_p$ is parasitic capacitance between wires. These parameters keep highly stable, so $R_e$, $C_e$ and $\kappa$ become decisive factors to oscillation frequency. So, if Kiwi fruit samples with different quality are connected to the circuit, the changeable parameters (including $R_e$, $C_e$ and $\kappa$) will directly lead to the differences of SAWR’s working condition and frequency.

With the rapid development of SAW technique, it has been widely employed including the distinct detection of ammonia, hydrogen detection, etc.

**EN detection system**

In recent years, EN detection technique has aroused increasing research interest, especially in food, such as vinegars, cherry tomato juices, Chinese green tea, and flavored oils.

As it shown in Figure 6, EN system’s structure consists of 3 main components: signal control and its collection (U1), sensory arrays (U2) and gas supply device (U3). The experiment was performed as following processing: first, open clean pump and valve 2 to absorb clean air for washing all sensors. Next, close the clean pump and valve 2 when all sensors’ responses stabilize at baseline. Then put Kiwi fruit sample into a clean vial and seal them with parafilm. After standing for 30 min, sampling probe and pneumatic balancer were simultaneously inserted. Then turning on EN system, the sampling time lasted for 45 s. Pneumatic balancer insulated impurity gas and absorbed clean gas into the vial via active carbon, which realized pressure balance. Eight metal oxide semiconductor (M.O.S) gas sensors were adopted to constitute array units. And their detailed parameters are shown in Table 2.

**Table 2. Constitution of gas sensor array**

| Sensor no. | Sensor type | Sensing species                  |
|------------|-------------|----------------------------------|
| S1         | TGS-825     | Sulfide                          |
| S2         | TGS-821     | Flammable gases                  |
| S3         | TGS-826     | Ammonia gases                    |
| S4         | TGS-822     | Ethanol, aromatic hydrocarbons etc|
| S5         | TGS-842     | Hydrocarbon component gas        |
| S6         | TGS-813     | Methane, propane, butane         |
| S7         | TGS-2610    | Propane, butane                  |
| S8         | TGS-2201    | Nitrogen oxides                  |

**Methods**

**PCA**

Principal component analysis (PCA), as a pattern recognition technique, has proved to be effective for discriminating between the responses of e-nose to complex gases. So PCA method was used to analyze the EN data.

**SR**

Stochastic resonance (SR) is a non-intuitive phenomenon, which provides enhancement in a nonlinear noise system and attracts more and more attention in the field of signal processing. SNR from the output signal is used to describe SR usually. There are 3 factors existing in SR system: a bistable system, an input signal and an extra noise source. Currently, overdamped Brownian motion particle driven by cycle power in a bistable potential well is used to represent the characteristics of the system.

$$dx/dt = -dV(x)/dx + Asin(2\pi f_0 t + \varphi) + \sqrt{2D}\xi(t)$$

(10)

Where $V(x)$ is a nonlinear symmetric potential function, $\xi(t)$ is a gauss white noise, its autocorrelation function is $E[\xi(t)\xi(0)] = 2D\delta(t)$, $A$ is the intensity of input signal, $f_0$ is the frequency of modulating signal, $D$ is noise intensity, and $\mu$ is a
real parameter,

$$V(x) = 0.25ax^4 - 0.5bx^2$$

(11)

Thus, equation (10) can be rewritten as:

$$dx/dt = bx - ax^3 + Asin(2\pi f_0 t + \varphi) + \sqrt{2D_\xi(t)}$$

(12)

Nowadays, what can reflect SR’s characteristics most commonly is SNR, and here we define SNR as:

$$SNR = \frac{\lim_{\Delta \omega \rightarrow 0} \int \Omega + \Delta \omega \Omega + \Delta \omega S(\omega)d\omega}{\Omega - \Delta \omega} / S_N(\Omega)$$

(13)

$$S(\omega)$$ and $$S_N(\Omega)$$ represent signal spectrum’s density and noise intensity within the extent of signal frequency, respectively.

Conclusions

A rapid freshness predictive model for forecasting Kiwi fruit’s storage comes up in this study. Kiwi fruit’s weight loss percentage increases with the increase of storage time, which indicates moisture loss in samples is significant. Human sensory evaluation also demonstrates that Kiwi fruit’s overall acceptance declines significantly during the whole experiment. Three freshness predictive models about Kiwi fruit based on SAWR, EN, and EN combined with SAWR, correspond to

$$Time_{SAWR} = 264.7099 + \frac{21.30909}{\text{MaxSNR} + 77.605}$$

($$R^2=0.865$$), $$Time_{EN} = \frac{\text{MaxSNR} + 77.605}{20.4936} + 0.4726 \times \frac{26.50642}{1.29708}$$

($$R^2=0.939$$), $$Time_{EN\text{combined}} = \frac{\text{MaxSNR} + 77.605}{26.50642}$$

($$R^2=0.998$$), respectively. Compared with 3 models’ prediction accuracy, it is clear that the mixed predictive model presents higher prediction accuracy than the model developed based on EN or SAWR and the validation experiments also validate this fact.

Furthermore, the proposed technique lowers the detection cost for SAWR. The SAWR detection method proposed in this study has following advantages: test sample works as a SAWR load, while SAWR device works as a stable frequency supply, which reduces one-time use waste. From another aspect, the variations of working frequencies must exist in most SAWR devices even produced in the same bath. Therefore, this method eliminates some basic errors due to the replacement of SAWR, which contributes to improving experiment accuracy. SAWR detection result reflects Kiwi fruit’s internal information, while EN analysis result reveals sample’s external message, so this combination could real-time monitor and deliver Kiwi fruit real change during storage. This method is promising for judging fruit’s best harvest time including rapid response, good repeatability, low cost, etc.

Disclosure of Potential Conflicts of Interest

No potential conflicts of interest were disclosed.

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