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Prediction of fatty acids in rice storage based on odor characteristics by gas chromatography-ion mobility spectrometry

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Abstract

In order to develop a quick method for predicting fatty acid in rice storage, gas chromatography-ion mobility spectrometry (GC-IMS) was applied to detect and analyze volatile organic compounds (VOCs) at different rice storage stages, and partial least squares regression (PLSR) algorithm was used to establish a linear regression model between fatty acid values and characteristic VOCs. The results showed that rice fatty acid values increased gradually with extension of storage time. Odor components of rice mainly included alcohols and aldehydes. Except for 1-octene-3-alcohol, the content of other VOCs showed an overall downward trend during the storage period. After variable optimization using two different algorithms, the correlation coefficient of the PLSR cross validation model could reach 0.9544, and the corresponding root mean square error was 2.4093. In conclusion, fatty acid values of rice with different storage periods could be accurately predicted by using characteristic VOCs variables and chemometric tools, which would provide a rapid and nondestructive detection method for rice quality during storage based on odor information.

Keywords: rice; odor characteristics; fatty acids; partial squares regression; gas chromatography-ion mobility spectrometry.

Practical Application: Odor components could be used to predict rice fatty acids.

1 Introduction

Grain reservations are primarily based on raw grains in China, which is also supplemented by processed grains. In recent years, processed grain storage has become a new direction for the development of grain reservation, among which rice reservation account for the largest proportion. In addition, rice reservation has the advantage of small storage capacity, and savings in manpower and material resources, which could also quickly and efficiently meet market demand, and has great flexibility and timeliness (Munarko et al., 2022). However, rice nutrients are exposed due to loss of husk protection during storage, which makes it easy to be affected by internal and external factors. Common quality and safety problems with rice storage include biological pollution caused by insects and rats (Müller et al., 2022), aging problems caused by self-respiration (Griglione et al., 2015) and mildew pollution caused by the metabolism of different molds (Gao et al., 2021). Therefore, how to achieve rapid detection of rice quality during storage and minimize grain loss is worthy of further research.

Apart from being rich in starch and protein, rice also contains a small amount of lipids. The lipid components of rice generally undergo biochemical reactions in the following two ways. The first is that lipids are decomposed into free fatty acids, among which unsaturated fatty acids can be further oxidized to form peroxides due to the existence of double bonds. Then, these substances can be further decomposed to form volatile organic compounds (VOCs), like aldehydes, ketones and acids. Another way is that lipids are hydrolyzed by lipolytic enzymes synthesized by themselves or moulds to produce fatty acids and glycerol (Li et al., 2022). As known, fatty acids are difficult for micro-organisms to use and will accumulate in the rice body. In this way, the content of rice fatty acids will gradually increase during the storage and further produce unpleasant odors. Therefore, fatty acid content has been considered as one of the key indicators that could reflect rice storage quality.

In fact, fatty acids are intermediate products in the series of chemical reactions of rice lipids. The metabolic pathway of rice lipids has shown that partial unsaturated fatty acids would eventually decompose into VOCs (Hu et al., 2020). At the same time, rice self-respiration can also lead to changes of odor components (Routray & Rayaguru, 2018). In addition, many studies (Ma et al., 2019; Prodhan & Qingyao, 2020; Setyaningsih et al., 2019; Wei et al., 2021; Zhao et al., 2020a) have also demonstrated that odor component changes in rice have a certain correlation with the quality of rice. Therefore, fatty acid content changes in rice are closely related to its odor components, and odor components may be used to characterize rice storage quality (Zheng et al., 2017), which provides a possibility for detecting content of rice fatty acids based on odor components.

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At present, rice quality detection and evaluation methods have been frequently reported based on flavor information (Verma & Srivastav, 2020). At the same time, relevant odor analysis technologies, including solid-phase micro-extraction technology combined with gas chromatography-mass spectrometry (Darmasiwi et al., 2022), gas chromatography-ion mobility spectrometry (GC-IMS) and electric nose technology (Cui et al., 2018) have also been applied in the fields of key odor component screening of fragrant rice (Zhao et al., 2020b), rice variety distinction (Sung et al., 2014), microbial toxin infection detection (Gu et al., 2020), rice geographical origin identification (Lim et al., 2018) and adulteration analysis (Fan & Yang, 2022; Timsorn et al., 2017). In addition, previous studies have also demonstrated that partial VOCs (like aldehydes, ketones, and furans) would change in high temperature storage, which was an important reason for decline of rice quality during storage (Shi et al., 2021). These above studies showed that odor analysis technology could achieve the purpose of rice quality detection, which had great reference value in terms of flavor chemistry applications. However, these studies mainly focused on analysis of the overall odor components of rice and the development of application methods based on different characteristic flavor components. Little is known on the relationship between VOCs and rice quality, especially the correlation and internal relationship between VOCs and rice fatty acid contents during storage.

GC-IMS is an analytical technique with highly sensitivity and selectivity, which has been widely used in the analysis of volatile components in food and agricultural products (Gu et al., 2021). In this study, the changes in fatty acids and VOCs were respectively detected by chemical procedures and GC-IMS technology. In addition, chemometric tools were applied to establish a linear regression model to achieve rapid and non-destructive detection of rice fatty acids, which would also provide a method for the prediction and evaluation of rice quality based on odor components.

2 Materials and methods

2.1 Samples and reagents

Rice samples (Shanglin brand) used in this experiment were bought from local markets (Liuzhou city, Guangxi province). Anhydrous ethanol, potassium hydroxide, potassium hydrogen phthalate and other analytical reagents and phenolphthalein reagents were all purchased from Sinopharm Chemical Reagent Co., Ltd. Petri dishes (60 mm) were purchased from Wande Guanglian Experimental Material Factory (Taizhou city, Jiangsu province).

2.2 Sample preparation

Our previous research had shown that rice samples stored in a constant temperature and relative humidity incubator (35 °C, RH=90%) initially had no obvious mildew phenomenon. After that, significant moldy characteristics began to appear gradually in the next 2 months. Therefore, a strategy of gradual sample preparation in the initial stage and centralized sampling detection in the final stage was adopted based on the estimated time required for rice mildew growth during storage. When the initially placed rice samples had sensory mildew in the incubator, the repetitive sample preparation procedure would be stopped, and all prepared samples were then subjected to GC-IMS analysis. In this manner, sample preparation process could be simplified, and the total time of sample preparation could be saved. The specific preparation process for rice samples was as follows. Firstly, each rice sample (100 g) was weighted accurately, put into a petri dish with a marked date, and the remaining rice was sealed and stored in a refrigerator (-5 °C) to avoid quality changes. Then, the above mentioned step was repeated once every day for the initial 20 d. The interval times for later sample preparation were 2, 3, 5, 7 and 12 d respectively, and each time interval was also repeated twice. Next, the sample preparation would be stopped when the earliest prepared rice sample had moldy appearances, including surface yellowing, embryo color changing and brightness dimming. In addition, the initial rice stored in the refrigerator was used as the reference sample (fresh rice). Finally, a total of 31 rice samples with different storage times were prepared by using the method as described above.

2.3 Analytical methods

Fatty acid detection

The fatty acid value of each rice sample was determined according to the Chinese National Standard GB/T 20569-2006. Each sample was measured three times and the average value was taken as the final measured value.

GC-IMS analysis

VOCs from each rice sample were detected by using a commercial GC-IMS instrument (FlavorSpec®, Gesellschaft Für Analytische Sensorsysteme mbH, Germany) based on an existing method (Chen et al., 2021). First, a 5 g rice sample was weighted and transferred into a 20 mL sealed headspace vial. The bottle was subsequently incubated at 60 °C for 10 min with an oscillation speed of 500 rpm. Then, a 500 μL volume of VOCs was drawn from the top of the vial and then automatically injected into the injector at 80 °C via a heated syringe (80 °C, splitless injection). Next, the VOCs were primarily separated by a MXT-5 capillary column (15 m × 0.53 mm × 1 μm, Restek, USA) with a temperature of 40°C. The carrier gas was nitrogen (purity ≥ 99.999%), which used the following programmed flow: 2 mL/min for 2 min, increased to 15 mL/min within 8 min, raised to 100 mL/min within 10 min, and finally reached to 150 mL/min within 10 min. After that, the VOCs were separated again in the IMS tube after ionization (45 °C). The total analysis time was 30 min. The length of drift tube was 9.8 cm, which was also filled with a constant reverse drift gas flow (nitrogen, purity ≥ 99.999%) under the flow rate of 150 mL/min. The retention index (RI) was calibrated by a mixture of n-ketones (C4-C9) under the same detection conditions, which would be used to identify VOCs by combining RI with ion mobility database.

Data analysis

GC × IMS library search (version 1.0.3) software was used to perform the qualitative analysis of VOCs. LAV software (version 2.0.1) with gallery report plug-in was applied to implement the

visual analysis of GC-IMS maps. MATLAB (version R2016b, Mathworks Inc.) software was used to realize characteristic variable screening and partial least squares regression (PLSR) analysis.

3 Results and discussion

3.1 Fatty acid analysis

Changes in rice fatty acids from different storage periods are shown in Figure 1. Fatty acid levels from fresh rice samples were 17.14 mg/100 g, then reached 57.44 mg/100 g (maximum value) at the end of storage. Fatty acid values between the initial period and the later mildew stage changed significantly during the entire storage process ($P < 0.05$). As can be seen, fatty acid values gradually increased with the extension of storage time. The main reasons for this phenomenon were that the lipids in rice samples were hydrolyzed via action of related lipases to form fatty acids, which cannot be consumed by microorganisms. In this situation, the fatty acids would continually accumulate in the rice body. According to the rule from GB/T 20569-2006, the fatty acid content of rice suitable for storage should be less than or equal to 25.0 mg/100g. In addition, when this value was greater than 35.0 mg/100 g, it indicated that the rice was no longer suitable for storage. Combined with our previous research (Chen et al., 2021), the rice storage process could be divided into four stages, i.e. fresh stage, early mildew stage, middle mildew stage, and late mildew stage (see different mark ranges in Figure 1). As seen from Figure 1, the fatty acid contents of rice samples from the fresh stage were within the proper range of storage. During the initial mildew stage, the fatty acid contents of partial rice samples was greater than the suitable storage value. Moreover, this value from rice samples in the middle and late mildew periods was far beyond the storage threshold. Therefore, it is considered that changes in fatty acid content are closely related to rice storage period, which means that fatty acid indicator could be used to judge the storage quality of rice.

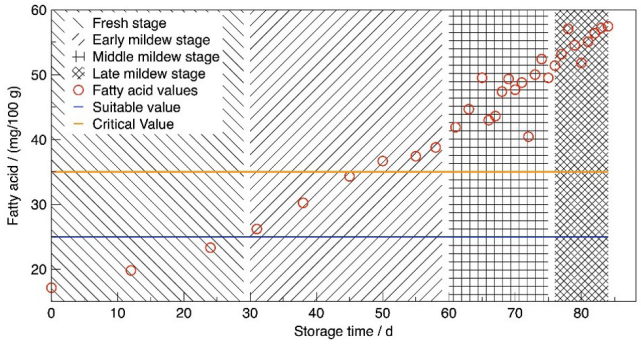

Figure 1. Fatty acid changes in rice samples from different storage periods.

3.2 VOCs analysis

In order to compare rice VOCs differences from different storage periods, fresh rice was selected as the reference, and rice with other storage times were operated by deducting the reference and visualized for comparison. As shown in Figure 2B and 2C, the original background color faded from blue (Figure 2A) to yellow after reference deduction. A red point or line in the map showed that the content of a volatile component is lower than the reference, while a green point or line indicated that the content of a volatile component is higher than the reference. As can be seen, the contents of partial volatile components increased significantly with the storage time while others decreased significantly. This phenomenon demonstrated that odor fingerprints of rice changed significantly during the storage process, and the storage time had different degrees of influence on different rice VOCs. Based on VOCs changes in GC-IMS map during rice storage process, a total of 21 characteristic regions were finally selected from two-dimensional maps of 31 rice samples, and a gallery report of these selected peaks for comparison was shown in Figure 3. In Figure 3, X axis indicates the number label of characteristic peaks, Y axis indicates the storage time of rice samples, and the color of each cell represents the signal intensity, which reflected the concentration of each odor substance. The identification results for these corresponding peaks were shown in Table 1. Due to the capacity limitations

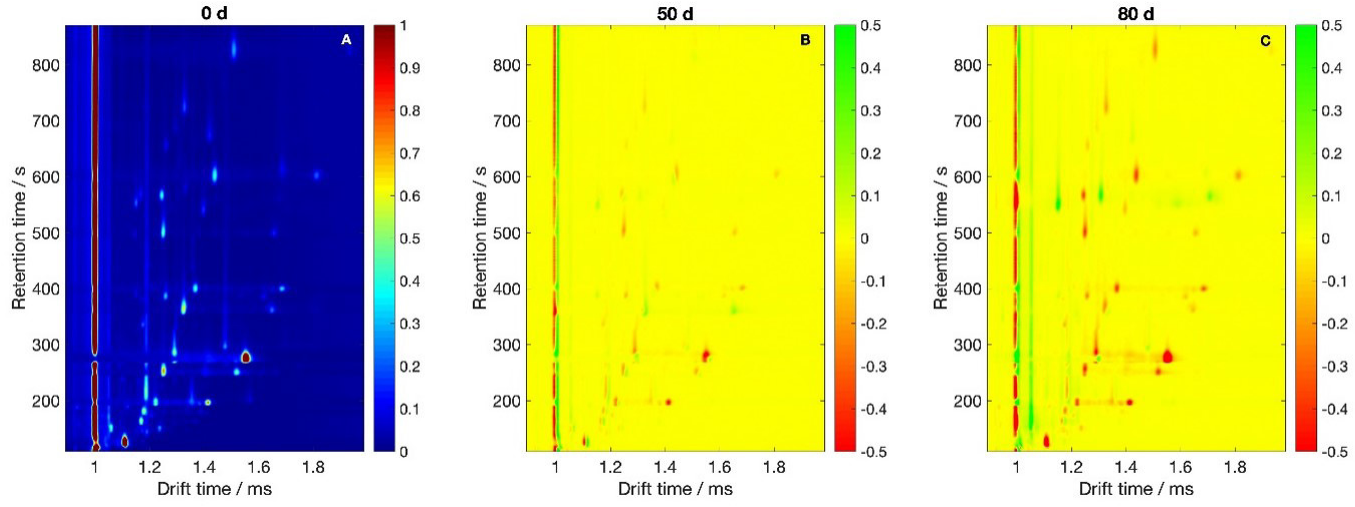

Figure 2. Topographic plots of VOCs comparison between fresh rice (A), rice stored for 50 d (B) and 80 d (C).

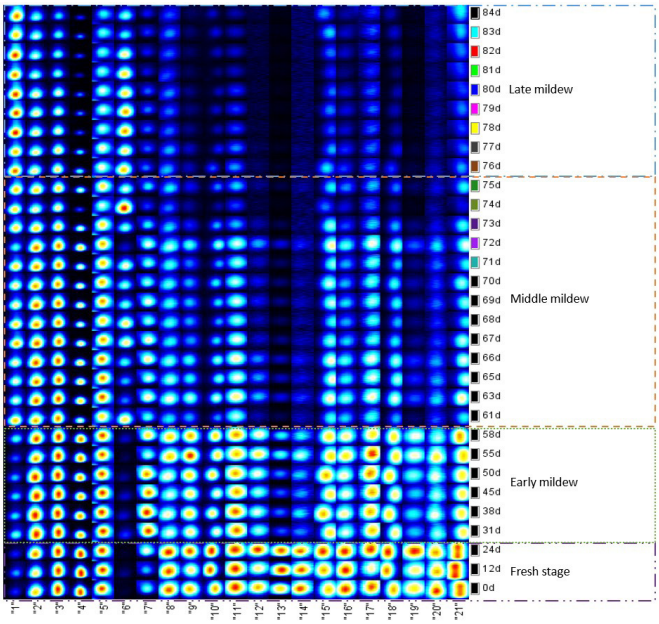

Figure 3. Gallery report of selected characteristic peak changes in rice samples from different storage periods

Table 1 Partial compound information corresponding to selected VOCs.

Characteristic peak number	Compound	Retention time / s	Drift time after normalization / ms
1	1-octen-3-ol	553.343	1.1523
2	hexanol	364.138	1.3269
3	1-pentanol	250.764	1.2537
4	1-hexanal	274.347	1.5573
5	1-butanol	180.660	1.1824
6	hexanoic acid	574.247	1.3091
7	3-hexen-1-ol	359.957	1.6527
8	2-heptanone	384.909	1.2612
9	2-pentylfuran	564.841	1.2447
10	2-furanmethanol	398.078	1.3660
11	1-pentanol	248.881	1.5201
12	octanal	600.848	1.8154
13	pentanal	195.253	1.4167
14	α-pinene	497.940	1.6584
15	phenylethyl alcohol	829.977	1.5098

of ion mobility database provided by G.A.S company, only 15 compounds were eventually identified. It could be seen that compounds corresponding to these selected characteristic peaks were mainly alcohols and aldehydes, with a few ketones and furans. 1-Pentanol (No. 3 and 11) contained both the monomer (the former) and dimer (the latter) forms for their corresponding substances in the map. With the extension of storage time, the concentration of most volatile compounds gradually decreased or even disappeared, including 1-pentanol, 1-hexanal, 1-butanol, 2-heptanone, 2-pentylfuran, 2-furanmethanol, 1-pentanol, octanal and phenylethyl alcohol. As a freshness marker of rice samples, the changes of octanal concentration were consistent with the conclusions from previous research (Zhao et al., 2020b). Unlike other volatile substances, the concentration of 1-octene-3-alcohol (No. 1) increased with the extension of time. The main reason for this phenomenon was that this substance might be produced by the physiological metabolism of moldy microorganisms. Other residual volatile compounds had no obvious changes. For example, hexanoic acid (No. 6) was initially not found in the fresh and early mildew stages. However, its concentration increased and then decreased between the middle and late mildew stages, which could have an important role in quality characterization in the latter storage period. The above results show that the composition and content of VOCs during rice storage are complex and changeable. Therefore, it is necessary to further analyze the correlation between VOCs and fatty acid indexes by using chemometric tools.

3.3 Correlation analysis between fatty acid and VOCs

Taking the intensity (peak height) of each selected characteristic peak as a variable, 21 characteristic peaks selected from 31 rice samples could be formed into a 31 × 21 matrix, which was used to build a linear regression model between VOCs variables and fatty acids by using PLSR algorithm. Because of the limitation of sample sizes, the sample set was not divided into the training set and the prediction set. Instead, all rice samples were used as the correction set to preliminary analysis of the correlation between VOCs and fatty acids (Figure 4). As can be seen, the 5-fold cross-validation results showed that the correlation coefficient (R^2) between measured values and predicted values could reach 0.9201, and the root mean square error (RMSE) was 3.4638. This indicated that the fatty acid index of rice could be accurately predicted based on the concentrations of its VOCs. However, the number of samples still needed to be expanded to form a prediction set for further verification and optimization in the late stage. In order to further analyze the importance of different VOCs related to fatty acids, these selected characteristic peaks were screened by using two different variable optimization methods, i.e. the monte carlo uninformative variable elimination (MCUVE) algorithm and competitive adaptive reweighted sampling (CARS) algorithm (Li et al., 2018). During MCUVE optimization process, the max principal component number for cross-validation was set to 10, and the number of monte carlo simulations was set to 500. The reliability index was used to evaluate the importance of characteristic peaks and the results were shown in Figure 5. The higher the wave peak, the greater the selection potential for the characteristic peak. Taking 2 as the threshold value, a total of 10 characteristic peaks were screened according to their importance, and their corresponding number labels were 8, 20, 11, 16, 14, 18, 13, 12, 1 and 10 respectively. On the other hand, the results of CARS variable selection were illustrated in Figure 6. The process of variable selection was shown in Figure 6A. As can be seen, the number of selected peak variables decreased gradually, and the downward trend moved from fast to slow while the variable selection process continued, which demonstrated that the change was in the way as peak variables were screened. In addition, Figure 6B showed the changes in the cross-validation error rate during variable selection. The curve showed that the error rate of cross validation decreased during the initial selection, and then increased with variable selection. The error rate of cross validation reached

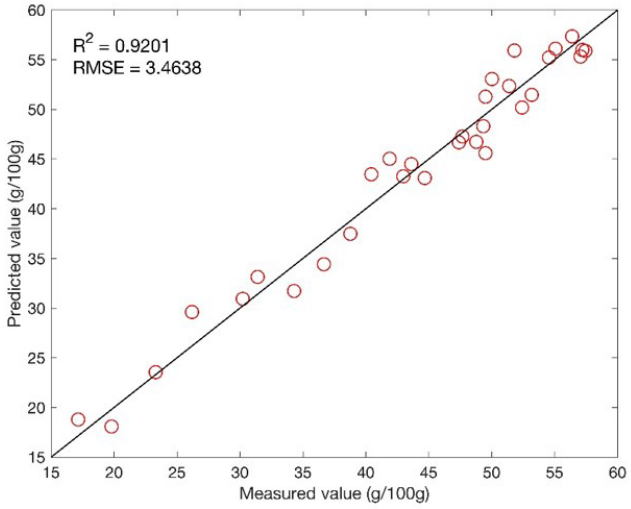

Figure 4. Cross validation results of rice fatty acids by using PLSR.

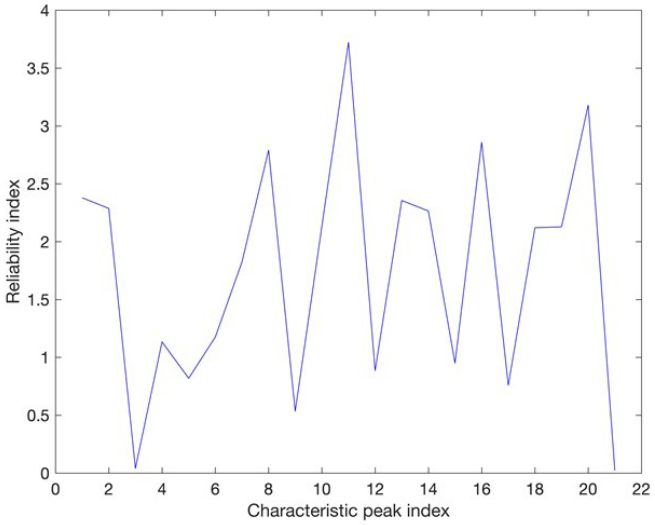

Figure 5. Optimization results of characteristic variables by monte carlo uninformative variable elimination.

the minimum when the number of sampling runs were in the range of 20-22. Figure 6C indicated the change in regression coefficient paths with the increase of sampling runs. When the number of sampling runs was 20, the error of cross validation and the number of selected variables all reached minimum values. Finally, a total of former 10 peaks were retained for comparison, which were 11, 20, 16, 8, 1, 13, 2, 14, 10 and 19. These filtered characteristic variables from two different variables selection methods were then used for regression analysis respectively, and the results were shown in Table 2. As seen in the table, the R^2 and *RMSE* indexes from MCUVE filtering algorithm were 0.9544 and 2.4093, which had better performance than the CARS method. In addition, it could be seen that the performance of each model was all improved by characteristic peak selection compared with no pre-treatment. From these selected variables by using two different variable screening methods, it could be found that the shared and important VOCs were mainly

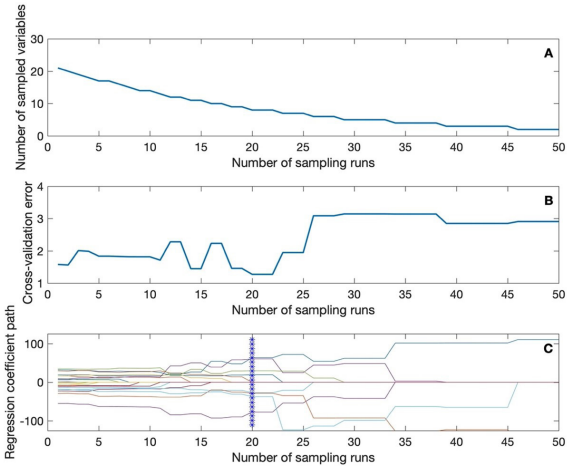

Figure 6. Optimization process of characteristic variables by using competitive adaptive reweighted sampling. (A) The changing trend of the number of sampled variables with the increasing of sampling runs; (B) standard deviation of cross validation; (C) the regression coefficient path of each variable.

Table 2. PLSR cross validation results from different variable optimization methods.

Methods	Variable size	R^2	*RMSE*
—	21	0.9201	3.4638
MCUVE	10	0.9544	2.4093
CARS	10	0.94627	3.0518

Note: "—": no variable extraction pre-treatment; MCUVE: monte carlo uninformative variable elimination; CARS: competitive adaptive reweighted sampling; R^2: correlation coefficient; *RMSE*: root mean square error.

alcohols (e.g., 1-octen-3-ol), aldehydes (e.g., 1-pentanol) and ketones (e.g., 2-heptanone). Among the alcohols, 1-octen-3-ol might be produced by both lipid oxidation and metabolism of microorganisms. Its concentration increased with the prolongation of storage time, while the concentration of other alcohols tended to decrease slowly over time, which might be from the rice itself and be related to its freshness. The production of aldehydes was partly due to decomposition of fatty acids, which could further form aldehydes and ketones in the rice body. Other characteristic odor components could also be from the rice itself. Previous papers had reported that the content of pentanal gradually increased with the expansion of storage time during the rice aging period (Saikrishna et al., 2018), which was consistent with our observation (see No.13 in Figure 3) in the early mildew stage. In addition, the concentration changes of octanal and 2-heptanal were also similar to the pentanal. Therefore, it is scientific and feasible to predict the fatty acid value of rice with the help of odor fingerprints.

4 Conclusion

In this study, changes in fatty acids during rice storage were analyzed, and GC-IMS technology combined with chemometric tools was conducted to predict the content of rice fatty acid based on odor fingerprints. The results showed that there was a high correlation between the screened characteristic odor

components and fatty acid values. After variable selection by MCUVE algorithm, the R^2 value from cross-validation of PLSR model was 0.9544 and the *RMSE* value was 2.4093. The characteristic peak optimization results showed that alcohols and aldehydes were closely related to the quality changes in rice storage. In conclusion, the proposed method could achieve the rapid and non-destructive detection of rice quality based on odor fingerprints. This method enriches detection means and application areas of flavor chemistry, providing a good example for technical improvement, method development and application expansion of flavor detection technology.

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