

Vintage identification of sauce-flavor liquor based on fluorescence spectroscopy

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RESEARCH ARTICLE

Abstract

The vintage of sauce-flavor liquor represents its collection value and grade, so its identification is essential for collectors and connoisseurs. In this research, a vintage identification model for sauce-flavor liquors was proposed based on the fluorescence spectroscopy method. A fluorescence spectrophotometer was used to measure the fluorescence spectrum of sauce-flavor liquors of different years, and the optimum emission peaks of the spectrum were obtained. By analyzing these optimum emission peaks, it was determined that intensity of the spectrum increased with the vintage of sauce-flavor liquor. A prediction model was established to identify quantitatively the vintage of sauce-flavor liquor according to spectrum intensity, and its coefficient of determination (R^2) was 0.995. The prediction model was verified with an average error of 0.06 years. Moreover, an online testing platform was designed to realize the real-time, rapid, and quantitative vintage identification of sauce-flavor liquors. The model and platform should be helpful for evaluating sauce-flavor liquor.

Keywords: vintage identification; sauce-flavor liquor; fluorescence spectroscopy; online testing platform

Introduction

People favor sauce-flavor liquor because of its high collection value and taste, and its age detection is essential for collectors and sommeliers. Sauce-flavor liquor must be cellared generally for more than 3 years to improve its quality (Dan Qin *et al.*, 2022). The selling price of liquor is closely related to the year (He *et al.*, 2023). Moreover, it is generally believed that the longer the year, the better the quality of liquor (Burns *et al.*, 2021). There is a lot of substandard and fake blended liquor on the market, which seriously harms the rights and interests of consumers (Gu *et al.*, 2019). The traditional methods of identifying aged liquor mainly include sensation and precise instruments. The sensory method must be performed by professional liquor taster, and this method is relatively

subjective. Some accurate instrumental detection methods are available, including main chromatography (Chen, Sha, Qian, & Xu 2017; Niu, Chen, Xiao, Ma, & Zhu 2017; D. Qin *et al.*, 2023; Sun *et al.*, 2018; P. P. Wang, Li, Qi, Li, & Pan 2015; Wu *et al.*, 2022) and spectroscopy (Su, Wang, Yu, & Zheng, 2022). These methods are time-consuming due to the complex pretreatment steps. Therefore, a real-time and rapid identification method is urgently needed to meet the market demand.

Fluorescence spectroscopy has been widely used in the food testing industry (Clément, Bacon, Sirois, & Dorais 2015; Guan *et al.*, 2022; Tan & Li 2021). Fluorescence spectroscopy is a non-destructive and non-contact analytical technique. This method is inexpensive and easy to operate (Azcarate *et al.*, 2015). In addition, it has a low

detection limit and can detect polycyclic aromatic substances and some heterocyclic substances (He *et al.*, 2023). The fluorescence spectrum of Chinese liquor is produced by various fluorescent substances present in the liquor (W. H. Wang *et al.*, 2021). During liquor's aging process, the content of fluorescent substances changes because of physical and chemical reactions. Furthermore, vintage liquor has a different fluorescence spectrum (Qiao, Zhang, & Wang, 2013; Su *et al.*, 2022). Therefore, fluorescence spectroscopy can be used to identify vintage of the liquor. Gu *et al.* (2018) defined a prediction model based on three-dimensional (3D) fluorescence spectral distance, which predicted the vintage with an average error of 0.3 years, and aroma-type classification of 16 brands of liquor was achieved with success. Ma *et al.* (2017) used a combination of fluorescence spectroscopy and chemometrics to analyze the 325–435-nm region of emission spectrum at an excitation wavelength of 300 nm. The liquor's origin and main aroma components were distinguished. Zhu *et al.* (2017) studied the 3D fluorescence spectra of liquor and combined it with a simulated annealing algorithm to develop a high-precision vintage prediction model in the 200–230-nm excitation wavelength range. A review of the above literature demonstrated that fluorescence spectra contained information required for liquor analysis. However, the study of the information on the vintage of liquor is limited to the excitation wavelength range below 300 nm. In addition, light sources below 320 nm have a significant harmful effect on humans (Hockberger, 2002). Therefore, it is very imperative to study the fluorescence spectral information of liquor for longer wavelengths.

This research used fluorescence spectroscopy to analyze differences of the aged Chinese liquor of the same brand. Fluorescence spectra of liquor were tested at 10-nm intervals from 320 nm to 390 nm. Moreover, using Matrix Laboratory (MATLAB), five prediction models were established to fit accurately the relationship between sauce-flavor liquor vintage and spectrum's intensity with optimum excitation peak. The best prediction model with a quadratic polynomial was chosen to identify quantitatively the vintage of sauce-flavor liquor. In addition, an online testing platform was successfully designed using Python 3.9.13 and PyQt v5.15.9 to realize the real-time, rapid, and quantitative vintage identification of liquors.

Materials and Methods

Sample

The samples used in the experiment were provided by Tianbang Weiye Brewing Ltd., Renhuai City, Guizhou Province. All samples are Tianbang series sauce-flavor

aged liquor, and 5-, 10-, 13-, 15-, and 30-year liquor samples were used for establishing a mathematical model to identify the year of liquor. Furthermore, 1- and 6-year samples of the same brand were used to verify the feasibility of the model.

Methods

We used FS5 fluorescence spectrometer (Edinburgh Instruments Ltd., Germany) to measure the fluorescence spectrum of sauce-flavor liquor. The tested liquor samples were placed in a 1×1×4-cm sealable quartz colorimeter.

We washed quartz cuvette with a small amount of deionized water before each test and rinsed the same with a small amount of the liquid sample to be tested. Then 5 mL of the liquid sample was aspirated with a pipette gun. The tested wine sample was placed into a 1×1×4-cm sealable quartz cuvette. The cuvette's lid was closed and placed into a fluorescence spectrometer; the measurements were taken according to the following steps:

Step 1: The emission slit (Em) and the excitation slit (Ex) were set to 2 nm and 3 nm, respectively, and the step size was 1. The excitation spectrum of 270–430 nm was obtained for five vintages of sauce-flavor liquor, with an emission wavelength of 430 nm.

Step 2: The experimental conditions were the same as mentioned in Step 1. The excitation wavelength was 320–390 nm, and the emission spectrum was tested every 10 nm. The fluorescence emission spectrum of five sauce-flavor liquors was measured. Three comparison experiments were carried out for each sample to obtain the average of fluorescence spectrum.

Data analysis

Fluorescence spectra were drawn using Origin 2021 (Origin Lab Ltd., Northampton, MA, USA). The emission spectrum intensity and vintage prediction model of sauce-flavor liquor were simulated and verified by MATLAB R2022b (Math Works Ltd., USA). Python 3.9.13 (Organization of Python Software Foundation, USA) and PyQt v5.15.9 (Riverbank Computing Ltd., UK) were used to design vintage of the Tianbang series Chinese liquor detection platform.

Results and discussions

Previous studies discussed the light scattering of liquid colloidal particles under light irradiation (Liu *et al.*, 2022). The results showed that scattering and absorption

in liquor were the main factors for detection of liquor quality. Moreover, the brightness of the light column is closely related to the fluorescence phenomenon of liquor. Paths of light with different brightness could be used to classify the vintage of Chinese liquor. Fluorescence spectroscopy tests were conducted to investigate the relationship between fluorescence intensity and the vintage of liquor.

Excitation spectrum and emission spectrum

Fluorescence spectroscopy is only related to excitation and emission characteristics of fluorescent substances. Also, the overall fluorescence in a mixed substance shows different peaks because of different fluorescence characteristics; therefore, fluorescence spectroscopy is more suitable for analyzing and detecting mixed substances (Albani 2011). Fluorescence spectroscopy has a high detection accuracy, and even a concentration of one trillionth of a fluorescent molecule could be tested accurately (Rye, Dabora, Quesada, Mathies, & Glazer, 1993). Although infrared spectroscopy, Raman spectroscopy, and near-infrared spectroscopy have a high degree of chemical specificity and could search for compound functional groups present in a substance at specific wavelengths, the complex mechanism of the item to be tested usually prevents the spectroscopic results from being analyzed in a direct manner in actual experimental process (Beć, Grabska, Bonn, Popp, & Huck, 2020). In addition, the sensitivity of fluorescence spectroscopy is one to three orders of magnitude higher than that of UV-visible spectroscopy (Burns *et al.*, 2021). Fluorescence spectroscopy is non-destructive, does not cause changes in the chemical state of the sample, and the sample can be recovered for multiple measurements, an advantage that chromatographic techniques have always lacked (Facci, Cezário, de Gois, Luna, & Pacheco, 2021). Chinese liquor contains a large number of compounds, and different liquors contain different compounds. The fluorescence of Chinese liquor is formed by the superposition of the fluorescence of various single fluorescent substances. Therefore, the fluorescence spectra of different Chinese liquors are also different. Use of fluorescence characteristics of Chinese liquor could reliably identify their quality (He *et al.*, 2023).

Figure 1 shows the photoluminescence excitation spectrum (PLE) of five types of liquors. As shown in the figure, regardless of the liquor vintage, the excitation curve starts to rise from 300 nm, reaches a peak near 370 nm, and then begins to decline. The general trend is a bell-shaped curve (the rising trend near 450 nm is due to the frequency doubling peak caused by the excitation light source close to the listening band). In addition, the more the age of liquor, the more pronounced the upward trend.

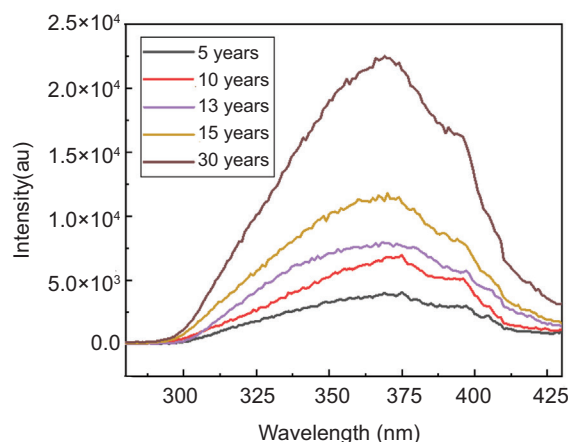


Figure 1. Excitation spectrum of five liquor samples.

On the other hand, ethanol, acids, aldehydes, phenols, and other substances in liquor are stored for some time only, and intermolecular association forms macromolecular groups. Alcohol and water molecules were synthesized under the action of hydrogen bond association groups. Most of the free single-molecule trace substances (ethanol, acids, esters, aldehydes, phenols, and other aromatic compounds) in liquor were replaced by hydrogen bonds instead of lone pair of electron bonds.

In addition to the excitation spectrum, the photoluminescence emission spectrum (PL) of these five liquors was also tested from 320 nm to 390 nm (Figure 2). According to the observation of PL of these five liquors, the overall PL shows a red shift trend with increased excitation source band, and the excitation intensity gradually rises with vintage. As shown in Figure 3, the PL of these five liquors could be divided into two ranges. There are two peaks in the younger aged group and single peak in the older aged group. The optimal excitation wavelength is 350 nm for shorter vintages and 370 nm for longer vintages.

Further analysis showed two peaks in the PL from 330 nm to 370 nm for 5-, 10-, and 13-year-old liquors, which were around 405 nm and 428 nm. An incomplete oxidation reaction, with increase in excitation wavelength, was the main reason for two peaks. Many materials, such as ethanol and aldehydes, resulted in the formation of two peaks. However, liquor aged more than 13 years had a long storage time and thorough oxidation reaction.

The optimal excitation wavelengths of the five samples were summed together. It was observed that the vintage of most liquors could be distinguished, and the degree of differentiation increased with increase of year gradient. With increase of liquor vintage, the peak width of liquor increased gradually at 428 nm and the number of peak values decreased gradually. It was speculated that with

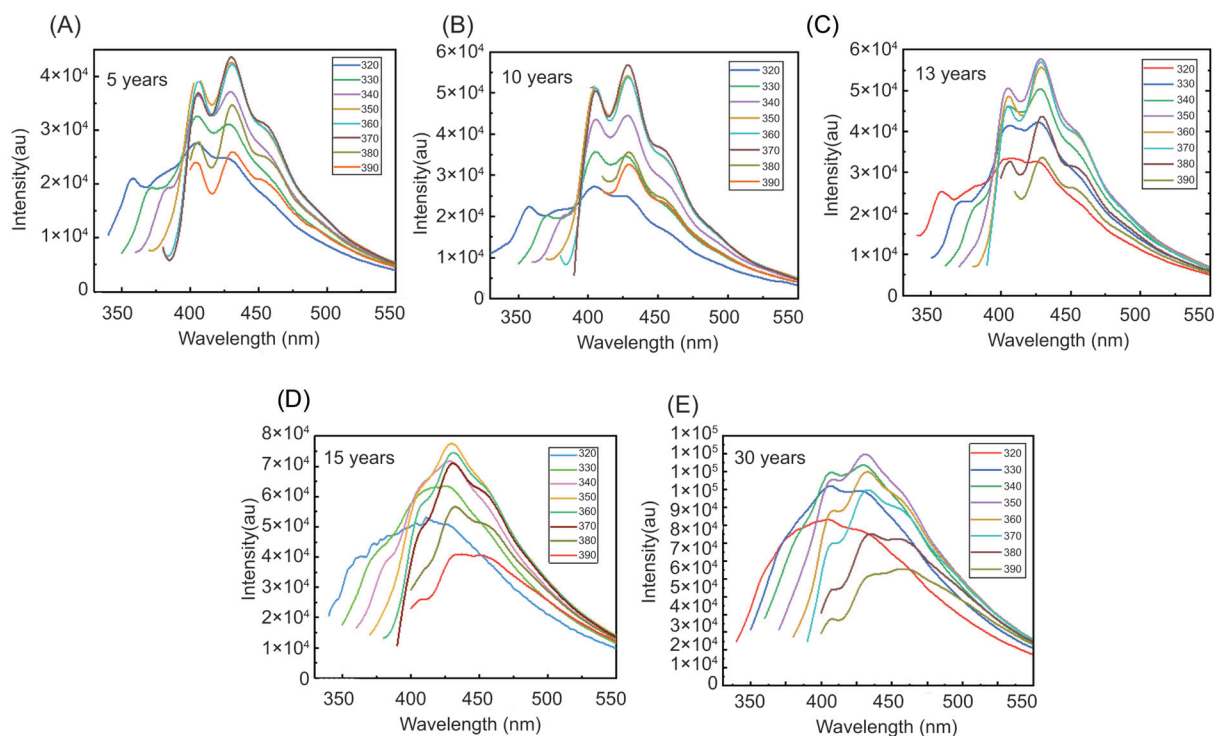


Figure 2. Emission spectrum (PL) of five samples, aged (A) 5 years, (B) 10 years, (C) 13 years, (D) 15 years, and (E) 30 years.

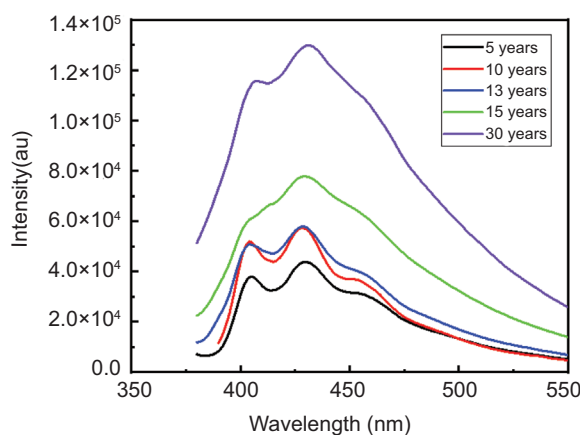


Figure 3. Summary of optimal emission spectrum wavelength of five liquors.

increase in liquor vintage, most reactions in liquor were gradually concentrated at 428 nm, and the reaction degree became more and more stable.

With the increasing liquor vintage, the peak intensity at 405 nm also increased gradually, but due to its small peak width, the growth rate was slower than that at 428 nm. Macroscopically, the peak strength at 405 nm was weak. It was speculated that reaction occurred between the monomers present in the liquor at a higher peak intensity of 405 nm with increase in the content of some of the monomers; this to a certain extent reflected vintage of the liquor, that is, the larger the peak intensity, the larger the vintage of liquor.

At 428 nm, a linear relationship was observed between peak intensity and vintage of the liquor aged less than 10 years. The overall strength increased between 10 and 15 years, but the degree of liquor reaction was more significant at 405 nm and more monomer substances were formed at this stage. The growth rate of liquor at this stage was low, and between 15 and 30 years, it was consistent with the predicted year at 405 nm, with constant growth.

Year forecast model

In order to explore the relationship between fluorescence intensity and vintage of the liquor, the peak data were fitted with five functions using the MATLAB-R2022b software. The curve fitting toolbox in MATLAB can use the least squares method to calculate relationship coefficients between response data and predicted data to model a relationship between fluorescence intensity and vintage of the liquor (Wen, Ma, Zhang, & Ma, 2012). MATLAB has been widely used to find a correlation relationship between different objects (Ke, Chiu, & Wu, 2016; Zhang, Ding, Wang, & Cao, 2021).

Figures 4A–E show images of five function fits. The ordinate of fluorescence spectrum represents relative intensity. The relative intensities were scaled down by a factor of 1,000 when entering the data for ease of data fitting. The abscissa represents vintage of the liquor. In addition, the spectrum data of 1- and 6-year-old liquor of the same brand were used to verify accuracy of the fitting model.

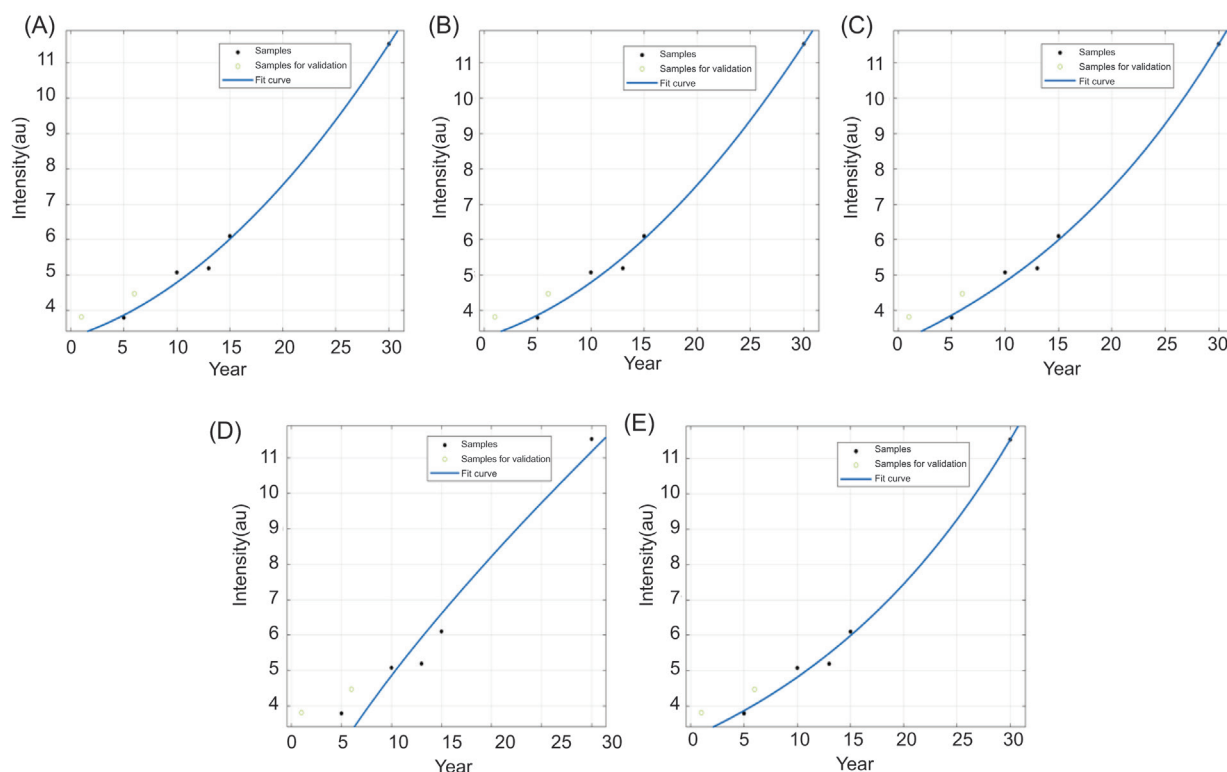


Figure 4. Function simulation images. (A) quadratic multi-idol function simulation; (B) Fourier function simulation; (C) Gaussian function simulation; (D) power function simulation; and (E) exponential function simulation.

Table 1. Simulation results of five functions.

Fitting name	Fitting function	R^2	SSE	RMSE	Verified SSE	Verified RMSE
Polynomial function fitting	$f(x) = p_1x^2 + p_2x + p_3$	0.9950	0.1826	0.3022	0.4197	0.4581
Fourier function fitting	$f(x) = a_0 + a_1 \cos(\omega x) + b_1 \sin(\omega x)$	0.9950	0.1826	0.4274	0.4197	0.4581
Gaussian function fitting	$f(x) = a_1 e^{-\left[\frac{-(x-b_1)}{c_1}\right]^2}$	0.9952	0.1742	0.2952	0.5212	0.5104
Power function fitting	$f(x) = ax^b$	0.9500	1.8180	0.7785	10.2147	2.2600
Exponential function fitting	$f(x) = ae^{bx}$	0.9952	0.1743	0.2410	0.5038	0.5019

Five functions successfully fitted the corresponding curves. Vintage of the liquor and relative fluorescence intensity of the best emission peak in its fluorescence spectrum showed a nonlinear relationship.

Table 1 shows the results of five fitting models. The coefficient of determination (R^2) characterizes the quality of a fit through change in data. The closer the R^2 value to 1, the better the fitting effect. The sum of squares due to error (SSE) is the sum of squared errors between the fitted data and the corresponding points of original data (Karabulut, Alkan, & Yilmaz, 2008). The closer the value of SSE to 0, the better the selection fit of the model and the more successful the data prediction. Root mean

square error (RMSE) is the square root of the mean of sum of squares of corresponding point errors of predicted and original data. The closer the RMSE value to 0, the closer the predicted value to the actual value and the better the prediction accuracy (Ozer, 1985). Gaussian function fitting and power function fitting had the highest R^2 value of 0.9952. Function fitting had the lowest R^2 value of only 0.9500. In addition, all goodness-of-fit data showed that power function fitted the worst, as observed from the SSE and RMSE data of validation set. Compared to Gaussian and exponential function fittings with higher R^2 values, the verification effect was better for both quadratic polynomial function and Fourier function fittings. However, the quadratic polynomial function fitting was

similar to the Fourier function fitting, except different RMSE values between the two. The RMSE value of the quadratic polynomial function fitting was closer to 0, indicating that the predicted value of the overall fitting was more consistent with the actual value. In summary, we identified the best model for liquor vintage prediction using polynomial function fitting. The obtained year prediction function is:

$$f(x) = 0.0061x^2 + 0.0940x + 3.246, \quad (1)$$

$f(x)$ is the relative fluorescence intensity, which is to be multiplied by 10^{-3} , and x is the year of Chinese liquor (x takes a positive value).

The predicted values were calculated for 5-, 10-, 15-, and 30-year-old liquors, and the results are shown in Table 2. Analyzing the relationship between predicted and actual values of five samples showed that the average error of the prediction model was about 0.06 years. Previous studies proposed that a simulated annealing algorithm was used to process the fluorescence spectral information of Chinese liquor, and the accuracy of the predicted vintage model obtained was 0.96 years (Weihua *et al.*, 2017). In contrast, the average difference was 0.3 years between the year prediction methods using fluorescence spectral distances (J. Gu, 2018). A degree of improvement in the accuracy of vintage prediction was observed compared to the previous work.

Online year detection platform

An online testing platform was designed with reference to previously reported studies (He *et al.*, 2023). Figure 5 shows the online year testing platform for the Tianbang series of liquor, which is based on the year prediction model. The test platform contains five main parts. After logging in, users are to input the spectrum data of Chinese liquor into the system. Then, the system analyzes the input spectrum data. Users can choose different functions according to their requirements. The vintage verification function verifies the labeled year of the liquor purchased from the market. The year prediction function is used to detect Chinese liquor products of unknown years. After the function selection is completed, users are

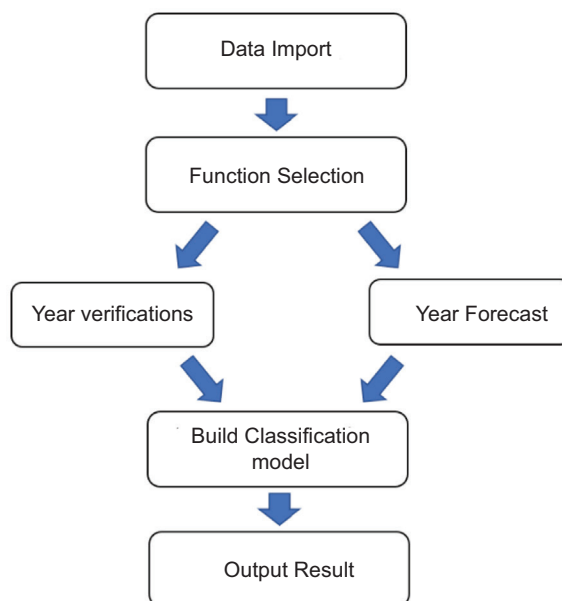


Figure 5. Flow chart of online year detection platform.

required to select the year prediction model for Chinese liquor. We provide five functions for the detection model, which users can adjust according to their specific requirements. The default detection model is the quadratic polynomial function model. Based on the model selected by the user, the platform automatically analyzes the processed data and generates results of the detection year. Users can download analysis report from the result output screen. Each detection interface is shown in Figure 6.

Conclusions

The fluorescence spectrum of sauce-flavor liquor was studied to summarize the rules of year identification of liquor in terms of peak intensity, fixed-point peak intensity, and state of substance composition. The results showed that peak intensity of the excitation spectrum of liquor increased with the year, and fixed-point peak intensity of the emission spectrum presented a quantitative trend with the year. According to the fitting results of MATLAB, the best relationship between year and emission peak intensity was determined by a quadratic polynomial model. The R^2 value of the model was 0.995. The prediction model was verified with an average error of 0.06 years. In addition, we successfully built an online year-testing platform for the Tianbang series of liquor using Python 3.9.13 and PyQt v5.15.9 for users to perform real-time testing. In conclusion, the above results successfully implemented the year testing of the Tianbang series of liquor and combined spectrum information with modern digital processing software and computer programs, providing consumers and regulators with a new method to quickly and efficiently check the year of liquor. In the future studies, the spectrum data of more brands

Table 2. Predicted results.

Actual value	Predicted value	Error value
5	4.51	-0.49
10	11.25	1.25
13	11.25	-1.25
15	15.26	0.26
30	29.94	-0.06

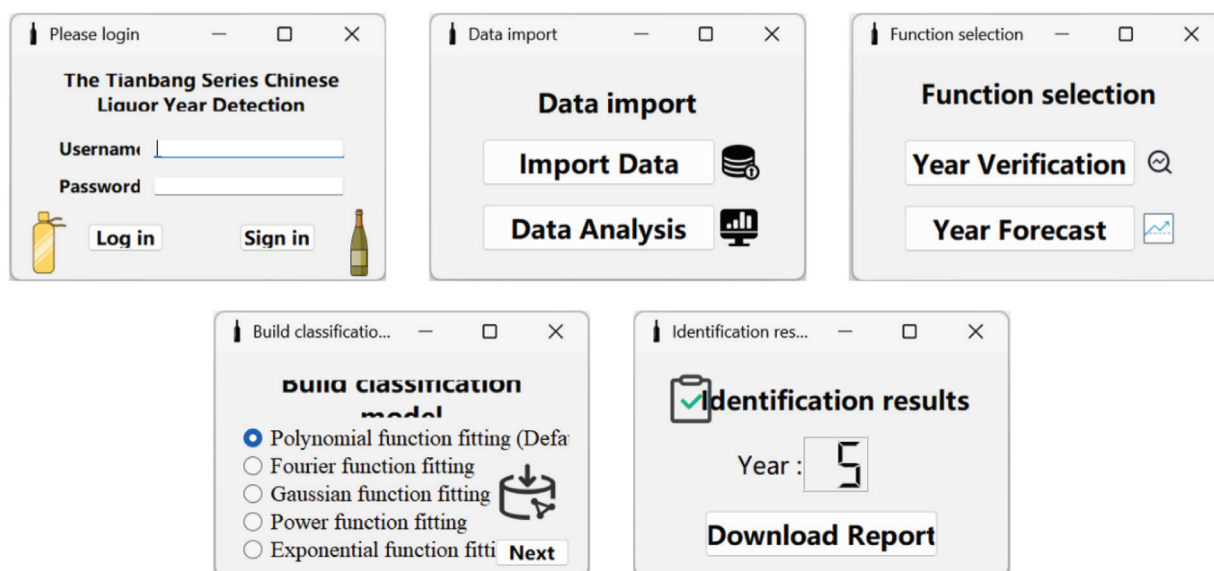


Figure 6. Online year detection platform program interface diagram.

of series year liquor must be measured to build up an extensive database containing enough samples to identify liquor year quickly.

Author Contributions

Analysis, data collection, investigation, editing, writing and original drafting were done by Rizeng Tao and Huizi Liu. Supervision, reviewing and editing were done by Chunfeng Guo. Resources and conceptions were done by Jun Zou. Sample collection was done by Qifei Zhu and Yifan Yang. Bobo Yang did methodology, supervision and reviewing. Lihua Chen did conceptions and reviewing. All authors read and approved the final manuscript.

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Conflicts of Interest

The authors declare no conflict of interest.

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