Development of a portable NIR rice wine analyzer

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A portable near-infrared (NIR) spectroscopy analyzer is developed for rice wine analysis. The analyzer is composed of a lamphouse, a temperature-controlled sample chamber, a mini-spectrometer, and a control unit. The mini-spectrometer is based on the crossed Czerny-Turner structure grating platform and a linear InGaAs array detector with spectrum range of 1100−2500 nm and spectral resolution of 1.5−12 nm full-width at half-maximum (FWHM). The control software written in VC++ consists of three parts: the spectra acquisition, the model establishment, and the sample analysis. The calibration models of determination of alcohol and nonsugar solids in rice wine are established with 54 samples. After calibration, the portable analyzer is used for real-time analysis of the rice wine sample.

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Rice wine, which has plenty of amino acids and vitamins, has more than 4000 years of history in China[1]. It is famous for its nutritional value and special flavor. The established methods of determination of rice wine components are generally based on either colorimetric or chromatographic techniques, such as ultraviolet-visible (UV-vis) spectroscopy and high-performance liquid chromatography (HPLC). These techniques are usually suitable for laboratory performance.

Near-infrared (NIR) spectroscopy technology is a rapid, nondestructive analysis technique, which generally requires little sample processing before analysis[2]. The NIR spectroscopy method has been reported to predict concentrations of amino acid, nitrogen[3], alcohol[4], and sugar[5] in rice wine. However, these reported methods are based on the Fourier transform NIR spectrophotometer, which is heavy, complex, and expensive.

In this letter, a portable NIR analyzer is developed based on a mini-spectrometer. The calibration models for the rapid quantitative determination of alcohol and nonsugar solid contents in rice wine are established. The advantages and limitations of this method compared with the traditional methods are also discussed.

The portable NIR rice wine analyzer consisted of a lamphouse, a sample chamber, an optical fiber, a mini-spectrometer, and a control unit. Figure 1 shows the optical path of the analyzer. The polychromatic light from a tungsten halogen lamp was collimated and directed through the sample chamber. The outlet light was transmitted into the mini-spectrometer through an optical fiber with an SMA905 adaptor. The transmission light was then split into monochromatic spectrum by grating and then received by an InGaAs array detector. The light signals were converted into analog electronic signals and were then converted into digital signals by an analog-to-digital (A/D) converter. Through the universal serial bus (USB) interface, the digital signals were transmitted to the control unit (embedded system or computer) for processing. The analyzer was compact, lightweight, and robust with no movable elements.

The lamphouse consisted of a tungsten halogen lamp, a constant-current source, and a heat dissipation system. The lamp was a fiber-coupled, constant current-operated 20-W tungsten light source. It had a stable light output with a wavelength range of 390–2900 nm using a high-precision driving current control and a gas-filled bulb. The heat dissipation system and slow ramp start were built into the source to ensure stable and long operational life.

A temperature-controlled sample chamber was developed in order to improve the stability and sensitivity of the analyzer. This sample chamber was controlled by a semiconductor thermal energy converter (TEC). The control core of the system was controlled by the chip PIC18F2455. The temperature display and setting board were controlled by a chip ZLG7290. By comparing the temperature of the sample chamber with the preset temperature, a PWM voltage signal was transmitted to a MAX1968 chip driver in the TEC to heat/cool the sample chamber. The light path of the sample chamber (1, 5, and 10 mm) can be adjusted by a stop plate.

The spectrometer included an SMA905 optical fiber, a mini optic grating platform, and an InGaAs array detector. The signal light was collimated through a spherical lens and then split into monochromatic spectrum by a plane grating. The monochromatic spectrum was then reflected into a linear detector array by a spherical mirror. The important elements of the spectrometer
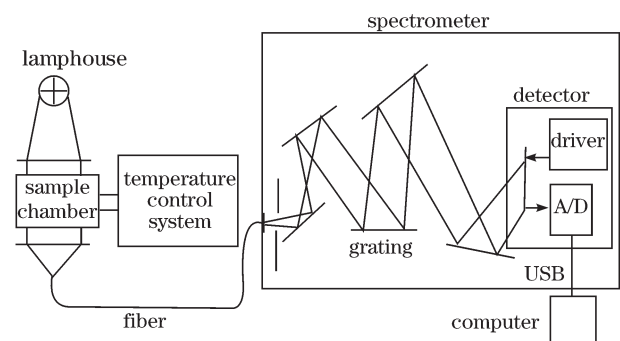

Fig. 1. Optical path of the portable NIR analyzer.

Table 1. Specifications of the Spectrometer

Performance	Index
Detector	256-Pixel InGaAs Array
Wavelength Range	1100−2150 nm
Platform	Crossed Czerny−Turner Structure
Grating	600 lines/mm
Spectral Resolution	1.5−12 nm FWHM
A/D Converter	16 bit
Integration Time	1 ms (min)−65.5 s (max)
Wavelength Accuracy	±0.5 nm

Table 2. Calibration Results of Alcohol and Nonsugar Solids

Component	Alcohol	Nonsugar Solids
Calibration Samples	50	48
Pretreatment	First Derivative	Second Derivative
Factor	7	8
RMSEP (%)	0.259	0.0134
R^2 (%)	99.94	99.29

Table 3. The t-test Results of Prediction

Component	Alcohol	Nonsugar Solids
Samples	10	10
True value (%)	20.2−3.1	0.33−0.014
$\mid$ Deviation $\mid$ (%)	0.544−0.025	0.027−0.002
t	0.8366	0.8818

Table 4. Repeatability of the Predicted Results

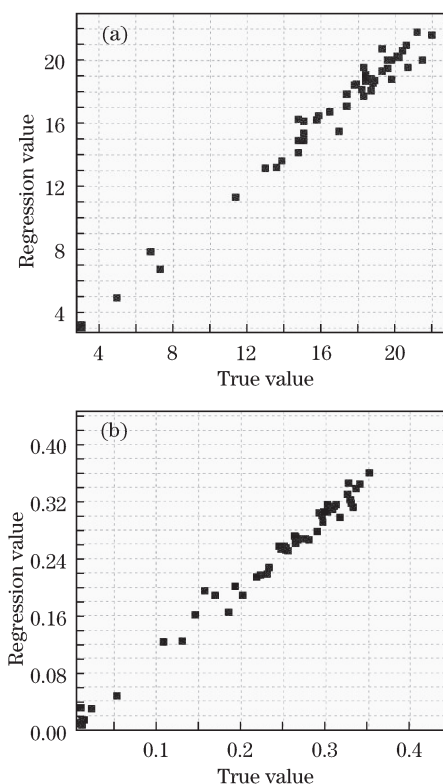| Component	Alcohol	Nonsugar Solids
True value (%)	17.4	0.218
Repetitions	10	10
Predicted Results (mg/mL)	16.566−17.322	0.218−0.238
RSD (%)	2.194	3.396

Fig. 2. Scattering plots of the calibration models. (a) Alcohol; (b) nonsugar solids.

"Components" for abbreviated name of the analyzed components and defining their units; "Load" for uploading the sample spectra and corresponding true values; "Pretreatment" for spectra pretreatment; and "Result" for showing the established models.

In pretreatment, there are four methods to choose from, including the first derivative, the second derivative, multiplicative scatter correction (MSC), and the standard normalization. After loading the spectra and reference data of the calibration samples and pretreating the spectra with pretreatment method, a model can be established. The correlation coefficient (R^2), the curve of root mean square error of cross-validation to factors, and the scattering plots of the true values and the prediction values are shown on this interface.

In the analysis interface, two analysis methods are provided, the real-time analysis and the spectral analysis. One or more models can be loaded by clicking the "Open Model" button. In real-time analysis, the prediction data are shown in the right table in real time with continuous spectra scanning. If the "Spectral Analysis" button is clicked, after loading a spectrum, then the prediction data are shown in the right table as well.

In all, 54 rice wine samples were used to establish models for conducting real-time analyses of the concentrations of alcohol and nonsugar solids in rice wine with this apparatus. The partial least square (PLS) algorithm was used to perform the calibration. The number of PLS factors for establishing the calibration model is chosen according to the root mean squared error of prediction (RMSEP) to the training set. Table 2 shows the value of factors, RMSEP and R^2 for the models of alcohol and nonsugar solids. Figure 2 shows the calibration results

included the grating, the entrance slit, and the fiber for dispersing the second diffraction. Table 1 shows the specifications of the spectrometer. The wavelength range of the spectrometer is between 1100 and 2150 nm with 1.5−12 nm full-width at half-maximum (FWHM) spectral resolution.

The controlling and data processing software was written in VC++ 6.0. It included three parts, i.e., spectra acquisition, model establishment, and analysis.

With the "scanning" procedure started, the spectra acquisition interface will appear. Prior to scanning the sample spectrum, the noise signal (the dark spectrum) and the reference spectrum must be scanned and saved firstly. The integration time can be adjusted from 1 to 20 (×50 ms) in this interface. The spectrum of the sample is shown on the interface after every scanning.

There are five buttons in the model establishment interface: "Method" for beginning a new method;

for alcohol and nonsugar solids.

The predicted and true values of 10 samples were examined using matching t test. The result showed that $t_{\text{alcohol}} = 0.8366$, and $t_{\text{nonsugar solids}} = 0.8818$; both were lower than $t_{0.05,9} = 2.262$ (Table 3). A sample was predicted repetitively for 10 times, after which the relative standard deviation (RSD) values for alcohol (2.194%) and nonsugar solids (3.396%) were calculated (Table 4).

In conclusion, a portable NIR spectroscopy analyzer is developed for rice wine analysis. The analyzer consists of a lamphouse, a temperature-controlled sample chamber, a mini-spectrometer, and a control unit. The mini-spectrometer is based on the crossed Czerny-Turner structure grating platform and a linear InGaAs array detector with spectrum range of 1100–2500 nm and spectral resolution of 1.5–12 nm FWHM. The control software written in VC++ consists of three parts: the spectra acquisition, the model establishment, and the sample analysis. The calibration models of determination of alcohol and nonsugar solids in rice wine are established with 54 samples. After calibration, the portable analyzer is used for rice wine sample real-time analysis. The test results show that the portable NIR rice wine analyzer is efficient, pollution-free, and user-friendly.

References

1. C. Chen, Z. Yin, and S. Xu, Liquor Making (in Chinese) **29,** 55 (2002).
2. Y. Yan, L. Zhao, D. Han, and S. Yang, (eds). *The Foundation and Application of Near-Infrared Spectroscopy Analysis* (in Chinese) (Chine Light Industry Press, Beijing, 2004).
3. Y. Chen, X. Zhou, W. Yu, P. Tang, and X. Zhou, Computers and Applied Chemistry (in Chinese) **25,** 361 (2008).
4. H. Zhu, D. Deng, and X. Zheng, Liquor Making Sci. Technol. (in Chinese) **9,** 113 (2008).
5. F. Liu, Y. He, and L. Wang, Acta Opt. Sin. (in Chinese) **27,** 2054 (2007).
6. T. Lin, H. Yu, H. Xu, and Y. Ying, Spectroscopy and Spectral Analysis (in Chinese) **29,** 950 (2009).