

# Discrimination between Chinese rice wines of different geographical origins by NIRS and AAS

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**Abstract** The feasibility of near infrared spectroscopy (NIRS) for discrimination between Chinese rice wine of different geographical origins (Shaoxing and Jiashan, China) is presented in this research. NIR spectra were collected in transmission mode in the wavelength range of 800–2500 nm. Qualitative analysis models were developed based on partial least squares regression (PLSR). The prediction performance of calibration models in different wavelength range was also investigated. The best models gave a 100% classification of wines of the two geographical origins in the range of 1300–1650 nm. The content of trace metals (potassium, magnesium, zinc, and iron) was also investigated to classify wines of the two categories by atomic absorption spectroscopy (AAS). The AAS results were in agreement with NIRS, with 100% classification for wines of the two categories. In addition, the correlation between NIRS and AAS was also investigated by PLSR. Potassium and magnesium were well predicted by quantitative models based on NIR spectra and AAS data. The correlation coefficient of calibration ( $R_{cal}$ ) for potassium and magnesium were 0.958 and 0.885, respectively, and the correlation coefficient of validation ( $R_{val}$ ) were 0.861 and 0.700, respectively. The results demonstrated that NIRS technique could be used as a rapid method for classification of geographical origin of Chinese rice wine, and AAS could be used as an alternative technique or to validate the discrimination results.

**Keywords** NIRS · AAS · PLSR · Chinese rice wine · Geographical origin · Discrimination

## Introduction

Determination of food authenticity is one of the most crucial issues in food control and safety. Wine identification or classification, mainly in terms of variety and geographical origin, has received increasing attention during the past 10 years. It has been attempted using several different compositional data including trace elements, aroma components, phenolic compounds, amino acids and volatile compounds, etc. [1–5]. Trace metals are considered as good indicators of wine geographical origin since they are not metabolized or modified during the fermentation process [2]. It was reported that sodium, potassium, calcium, magnesium, iron, and copper bore the denominations of origin of Lanzarote and La Palma islands (Canary Island, Spain) [6], and that linear discriminant analysis using rubidium, sodium, manganese and strontium gave 100% recognition ability and 95.6% prediction ability for identification of wine samples from El Hierro, Lanzarote and La Palma islands (Canary Island, Spain) [7]. Thiel et al. reported that provenance and authenticity of wines could be recognized on the basis of typical mineral and trace elements (aluminium, boron, barium, calcium, chromium, copper, iron, manganese, magnesium, phosphorus, plumbum, silicon, tin, vanadium, and zinc) [8]. According to Sperkova and Suchanek, the best results for identification of wine origin (Bohemian wine regions of Czech Republic) were achieved by the contents of aluminium, barium, calcium, cobalt, potassium, lithium, magnesium, manganese, molybdenum, rubidium, strontium, vanadium and elements ratio strontium/barium, stron-

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tium/calcium, strontium/magnesium [9]. All of the methods require complex and expensive analytical technique such as atomic absorption spectroscopy (AAS), inductively coupled plasma-mass spectrometry (ICP-MS), total-reflection X-ray fluorescence, and so on.

Near infrared spectroscopy (NIRS) technique has gained wide acceptance in the field of food processing and technology mainly due to its suitability for recording the spectra of solid and liquid samples at low cost, without any pretreatment and in a nondestructive way. The NIR region of the electromagnetic spectrum lies between the visible and middle infrared regions and spans the wavelength range between 780 and 2526 nm (ASTM). And it contains information concerning the relative proportions of C–H, N–H and O–H bonds which are the primary structural components of organic molecules [10]. NIRS technique has been used to classify finishing oils in acrylic fibres [11], to detect and quantify sunflower oil adulterants [12], to evaluate the feasibility of ginseng classification according to cultivation area (Korea and China) [13], to discriminate between samples of Australian commercial white wines of different varietal origins (Chardonnay and Riesling) [14], to detect and quantify the use of apple adulteration to extend or adulterate diluted and sulfited strawberry and raspberry purees [15], to classify wine vinegar and alcohol vinegar in north Spain [16] and to discriminate fishmeal batches made with different fish species [17].

And NIRS technique has also been used to determine inorganic components of seawater, such as chlorine, sodium, potassium, sulphur tetroxide, and calcium [18], trace metals (copper, iron, potassium, sodium, magnesium, and calcium) in white wines from Melbourne [19], and trace metal concentration (sodium, sulphur, copper, iron, manganese, zinc, and boron) in legumes [20].

Chinese rice wine is a sweet, golden wine made from glutinous rice and wheat with unique craft. The most well-known Chinese rice wine is Shaoxing rice wine, which is known by the generic name “Shaoxing”, which is comparable to the European declaration of Denomination of Origin. According to the National Standard of China GB 17946-2000 (Shaoxing rice wine), Shaoxing rice wine is made from high quality glutinous rice and wheat, as well as water from Mirror Lake in the region of Shaoxing, Zhejiang province. The common fraudulent practice in the commercialization of Chinese rice wine is to sell the wines from different geographical origins under the denomination of Shaoxing rice wine. The adulterated products constitute a fraud for consumers. Therefore, it is important to discriminate Shaoxing rice wine from wines from the other regions.

The aim of this work was to investigate the applicability of NIRS and AAS technique for discriminating between Chinese rice wine of different geographical origins (Shaoxing and Jiashan, Zhejiang province, China). NIRS technique

was developed as a rapid and low-cost tool, and AAS, as a commonly accepted chemical method, was used to validate the discrimination results.

## Materials and methods

### Samples

Thirty bottles of Chinese rice wine samples were sourced from “pagoda” brand Shaoxing rice wine brewery, and eight bottles were from “fen lake” brand Jiashan rice wine brewery. In all, 10 samples were of 1 year age, 14 were of 3 years age, and 14 were of 5 years age. All of the samples were semi-dry rice wine.

### Spectral measurements

Samples taken from freshly opened bottles of wine were scanned in transmission mode using a commercial spectrometer Nexus FT-NIR (Thermo Nicolet Corporation, Madison, WI, USA) which was equipped with an interferometer, an In-GaAs detector, and a broad band light source (Quartz Tungsten Halogen, 50 W). Samples were scanned in a 1 mm optical path-length rectangular quartz cuvette with air as reference at room temperature.

NIR spectra were collected using OMNIC software (Thermo Nicolet Corporation, Madison, WI, USA) and stored in absorbance format. The spectral range was from 800 to 2500 nm, the mirror velocity was  $0.9494 \text{ cm s}^{-1}$ , and the resolution was  $16 \text{ cm}^{-1}$  in this work. The spectrum of each sample was the average of 32 successive scans.

### AAS measurements

Potassium, magnesium, zinc, and iron were determined on a SHIMADZU AA6501 atomic absorption spectrometer (SHIMADZU Corp., Kyoto, Japan). The four trace metals were measured using potassium, magnesium, zinc, and iron hollow cathode lamp with an air–acetylene flame. To remove the matrix effect, the samples were treated with nitric acid in a high pressure vessel, prior to AAS analysis.

### Chemometrics and data analysis

#### *NIRS data analysis*

Chemometrics analysis was performed using the commercial software package, TQ Analyst v6.2.1 (Thermo Nicolet Corporation, Madison, WI, USA).

Principal component analysis (PCA) was performed before partial least squares regression (PLSR) models were developed. PCA is a method of data reduction that constructs

new uncorrelated variables, known as principal components (PCs) that are linear combination of the original ones. The PCs account for as much as possible of the variability as in the original data [14]. The spectral data was normalized so that the mean is 0 and the standard deviation (SD) is 1, and then PCA based on covariance was performed on the normalized spectral data.

#### Qualitative analysis

Twenty-nine samples were used for calibration set (23 bottles of Shaoxing rice wine and 6 bottles of Jiashan rice wine), and the remaining 9 for a validation set (7 bottles of Shaoxing rice wine and 2 bottles of Jiashan rice wine).

Qualitative models for geographical origins were developed using PLSR method together with NIR spectra. Each sample was assigned a dummy variable as a reference value. In this case, samples of Shaoxing rice wine were assigned a numeric value of 1, and Jiashan, 2. The PLSR model was then developed by the spectral data against the assigned reference value (dummy variable). A sample was classified as Shaoxing rice wine if the value was between 0.5 and 1.5, and it was Jiashan rice wine if the value was between 1.5 and 2.5. The criteria for the cutoff selected were similar to those reported by the others [14].

PLSR models were developed using seven spectral regions, full spectral range of 800–2500 nm, short- and long-wave spectral range of 800–1100 nm and 1100–2500 nm, and five discrete regions which were the main absorption bands of the spectra (1300–1650 nm, 1650–1850 nm, 1850–2200 nm, and 2200–2400 nm).

#### Quantitative analysis

The correlation between NIR spectra and AAS data for potassium, magnesium, zinc, and iron were established with the Shaoxing rice wine samples. The reason for only using Shaoxing rice wine samples was that the distribution of Shaoxing and Jiashan rice wine samples was not even. Thus, 23 samples were used for calibration analysis, and the remaining 7 were used for the validation.

Quantitative models for potassium, magnesium, zinc and iron were developed using PLSR and leave-one-out cross-validation. Cross-validation estimated the prediction error by splitting all samples into two groups. One group was reserved for validation (one in this case) and the others were used for calibration. The process was repeated until all the standards in the calibration set had been quantified as validation standards. Full spectral range was applied to the quantitative analysis.

The optimum number of factors used in PLSR was determined by the lowest value of predicted residual error sum of squares (PRESS). Calibration statistics included correlation

coefficient of calibration ( $R_{\text{cal}}$ ), and root mean square error of calibration (RMSEC).

In order to validate the equations, the models were applied to the spectra from the validation set, thus statistical parameters correlation coefficient of validation ( $R_{\text{val}}$ ) and root mean square error of validation (RMSEP) were obtained.

#### AAS data analysis

The multivariate analysis of AAS data was performed using the TQ Analyst v6.2.1 in the following steps:

1. Preliminary data analysis was performed by PCA on the whole data set to reduce the number of variables.
2. Calibration analysis was performed by PLSR method together with the variables selected from PCA results. Each sample was assigned a dummy variable, the same criterion with NIRS analysis.
3. Validation analysis was performed on the samples of the validation set by PLSR.

## Results and discussion

#### NIRS analysis results

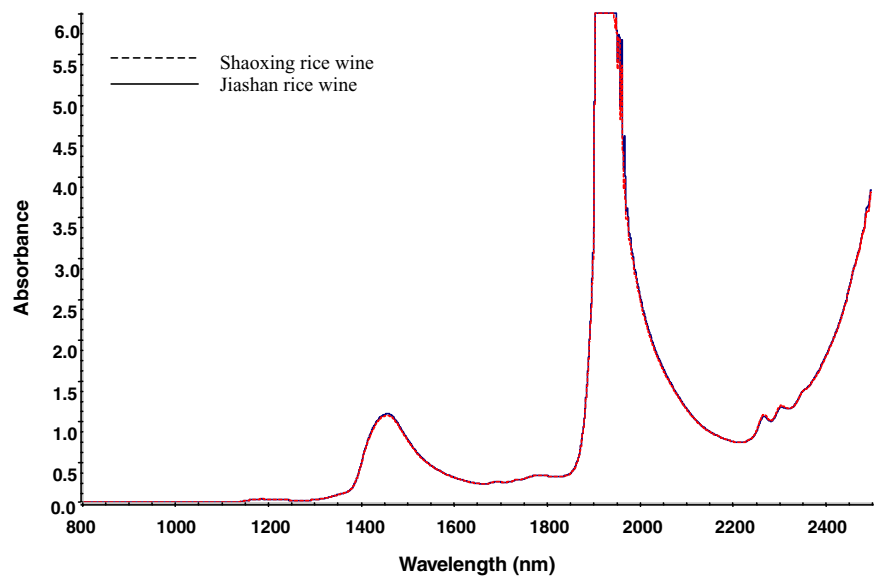
##### NIR spectral analysis

Figure 1 shows the average spectra of Shaoxing and Jiashan rice wine samples for the whole sample set. Both spectra have absorption bands at 1450 nm and 1900–1950 nm which are related to the first overtone of the O–H stretch of  $\text{H}_2\text{O}$  and a combination of stretch and deformation of the O–H group in  $\text{H}_2\text{O}$ , respectively. The absorption band at 2266 nm is likely related to C–H combinations and O–H stretch overtones, and that at 2305 nm is related to C–H overtones of ethanol [21]. The absorption band at 1790 nm is reported to be associated with sucrose, fructose, and glucose [22]. The bands of the two spectra are much overlapped, except that at 1450 nm the absorption intensity of Jiashan rice wine is a little higher than that of Shaoxing rice wine, and at 2266 and 2305 nm the absorption intensity of Jiashan rice is a little lower than that of Shaoxing rice wine.

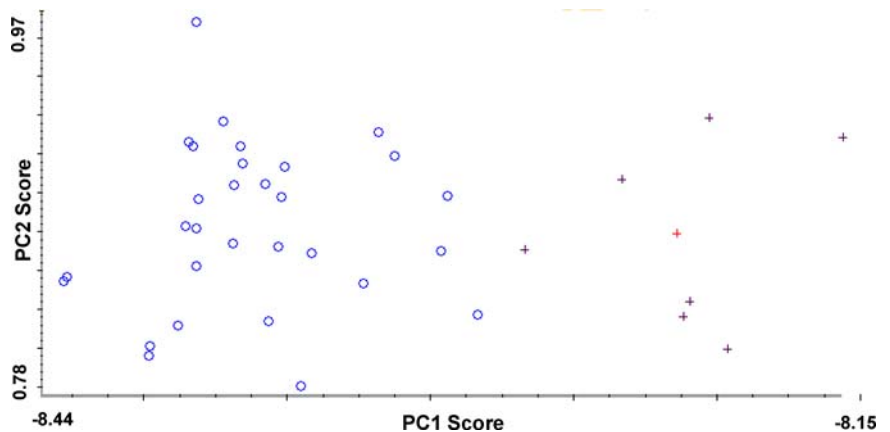
##### Principal component analysis for qualitative analysis

PCA was performed on the full spectral region, 800–2500 nm. All raw spectra of 30 Shaoxing samples and 8 Jiashan samples were used. It is obvious that the samples are most discriminated based on geographical origin when using two PCs (PC1 and PC2), as is shown in Fig. 2. This result indicates that the discrimination between wines of two geographical origins is viable.

**Fig. 1** Average of NIR spectra of 30 Shaoxing rice wine samples and 8 Jiashan samples



**Fig. 2** Sample score plot for PC1 and PC2 of NIR spectra (raw spectra, 400–2500 nm) for 30 Shaoxing rice wine samples and 8 Jiashan samples

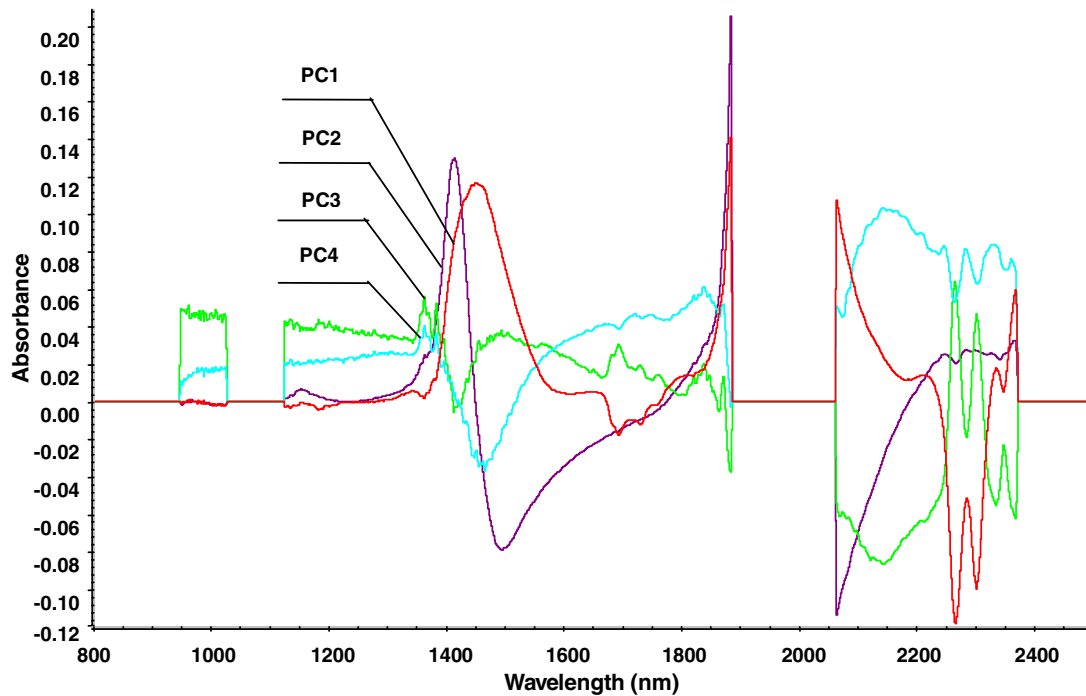


To investigate the basis for the spectral discrimination between the samples of the two geographical origins, the PCA eigenvectors were analyzed (Fig. 3). The first four PCs account for 98.86% of the variation in the spectra. PC1 explains 69.05% of the total variance, and the highest eigenvectors were found at 1450 nm associated with a combination of stretch and deformation of the O–H group in H<sub>2</sub>O, at 1884 nm related to O–H stretch and C=O second overtone combinations, at 2064 nm related to a combination of stretch and deformation of the O–H group, at 2336 nm related to a combination of stretch and deformation of the C–H group, and at 2370 nm related to C–H stretch. PC2 explains 24.22% of the variation, and the highest eigenvectors were found at 1410 and 1884 nm, both related to O–H overtones due to water. These spectral regions are characteristics of water, ethanol or sugar absorption in the wine.

#### Discriminant analysis

Table 1 shows calibration statistics of PLSR qualitative models developed on the NIR spectra using different wavelength regions. Wavelength range of 1300–1650 nm of the spectra gives the best calibration result, which demonstrates that the absorption band at 1450 nm, associated with a combination of stretch and deformation of the O–H group in H<sub>2</sub>O (Fig. 3), provides the necessary information for discrimination of the geographical origin of Chinese rice wine. The results for 800–2500 nm and 1100–2500 nm are a little worse than that for 1300–1650 nm. And the calibration result for 800–1100 nm is the worst of all the seven regions, which indicated that this spectral range is of little use in the geographical origin discriminant analysis.

The validation results using the best calibration models selected from Table 1 are summarized in Table 2. Wavelength



**Fig. 3** Eigenvectors for the PCA on the whole sample set

range of 1300–1650 nm gives the best validation results, with RMSEP = 0.239. Shaoxing and Jiashan rice wine are all classified correctly in 100%.

Figure 4 shows the NIR predictions of geographical origins of Chinese rice wine samples in the calibration and validation sets using best PLSR calibration model (1300–1650 nm). It indicates that NIRS together with PLSR method gives an excellent discrimination between samples of two geographical origins.

**Table 1** Calibration statistics of PLSR models developed on the NIR spectra using different wavelength regions

Wavelength range (nm)	$R_{cal}$	RMSEC	Percentage of samples correctly classified in calibration
800–2500	0.905	0.172	100
800–1100	0.301	0.386	86.2
1100–2500	0.905	0.172	100
1300–1650	0.909	0.169	100
1650–1850	0.681	0.297	96.6
1850–2200	0.849	0.214	96.6
2200–2400	0.895	0.181	100

**Table 2** Validation results for the PLSR models in the wavelength range of 800–2500 nm, 1100–2500 nm, and 1300–1650 nm

Wavelength range (nm)	RMSEP	Percentage of samples correctly classified (Shaoxing rice wine)	Percentage of samples correctly classified (Jiashan rice wine)
800–2500	0.259	100	100
1100–2500	0.259	100	100
1300–1650	0.239	100	100

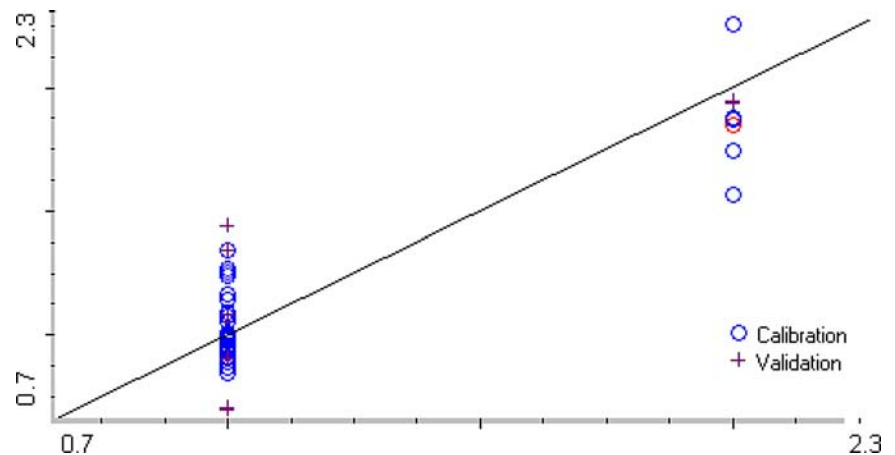
AAS analysis results

*AAS data for trace metals in the Chinese rice wine samples*

Table 3 shows the results for the range, the mean, and the SD values of potassium, magnesium, zincum, and iron in Shaoxing and Jiashan rice wine samples.

Mean metal concentrations of wines analyzed can be arranged in the sequence of potassium > magnesium > zincum > iron in the cases of the Shaoxing and Jiashan rice wine, which is in agreement with the literature [23]. From Table 3, it can also be seen that Shaoxing rice wines have higher mean values of potassium and magnesium than Jiashan wines, while Jiashan rice wines have a higher mean value of iron. To see if these differences between the two geographical origins were significant, one-way ANOVA (least-significant difference) was made for comparison of the mean values. Table 4 shows the values of  $F$  and  $p$  for the four trace metals, and as can be seen that the differences were significant with a probability higher than 95% for potas-

**Fig. 4** NIR predictions of geographical origins of Chinese rice wine samples in the calibration and validation sets using best PLSR calibration model (1300–1650 nm and raw spectra)



**Table 3** Results for the range, the mean, and the SD values of the metals potassium, magnesium, zincum, and iron in Shaoxing and Jiashan rice wine samples

Metal	Shaoxing rice wine			Jiashan rice wine		
	Range (mg l <sup>-1</sup> )	Mean (mg l <sup>-1</sup> )	SD (mg l <sup>-1</sup> )	Range (mg l <sup>-1</sup> )	Mean (mg l <sup>-1</sup> )	SD (mg l <sup>-1</sup> )
Potassium	416.67–593.13	503.63	50.71	231.89–308.28	258.96	22.32
Magnesium	165.58–197.39	175.02	7.77	81.03–91.66	85.53	3.56
Zincum	3.75–4.71	4.15	0.25	3.47–4.59	3.99	0.35
Iron	0.15–2.98	1.17	0.79	1.63–2.86	2.28	0.44

**Table 4** One-way ANOVA results for potassium, magnesium, zincum, and iron

Metal	<i>F</i>	<i>p</i>	Significance of difference
Potassium	297.4	0	Significant
Magnesium	990.8	0	Significant
Zincum	2.2	0.15	Not significant
Iron	14.4	5.79953E-4	Significant

sium, magnesium, and iron. This result indicates that these three metals may play an important role in the discriminant analysis of wines from the two geographical origins.

#### Discriminant analysis of AAS data

PCA was performed on the AAS data set of the four trace metals. PC scores plot of PC1 and PC2 is shown in Fig. 5. It is obvious that the samples are most discriminated based on geographical origin when using the two PCs. PCA results indicate that the first two PCs account for almost 100% of the variation, and PC1 explains 98.86% of the total variance in the samples.

PLSR models were developed using the combination of potassium and magnesium for discriminant analysis. Table 5 shows the calibration and validation statistics from the PLSR models. It is obvious that the combination of potassium and magnesium are sufficient to obtain a classification of 100% in each category.

Figure 6 shows the predictions of geographical origins of Chinese rice wine samples in the calibration and validation sets using potassium and magnesium. It indicates that trace metals together with PLSR method gives an excellent discrimination between samples of the two geographical origins. And the AAS results were in agreement with NIRS, which indicated that AAS could be used to validate the discrimination results.

The main crafts and storage methods for Shaoxing and Jiashan rice wine are similar. The crafts are to steep rice, steam rice, stir, ferment, squeeze, preserve in pot, store and bottle. And rice and wheat used in the brewing process are the ones circulated in the market, not from the local farmers. But on the whole, the proportion between rice and water for Shaoxing rice wine is higher than that for Jiashan rice wine, and water for Shaoxing rice wine is from Mirror Lake which is richer in trace elements. The NIRS analysis results may cover the differences in geographical origin and directions for producing wines. And the AAS results mainly covered the differences in geographical origin.

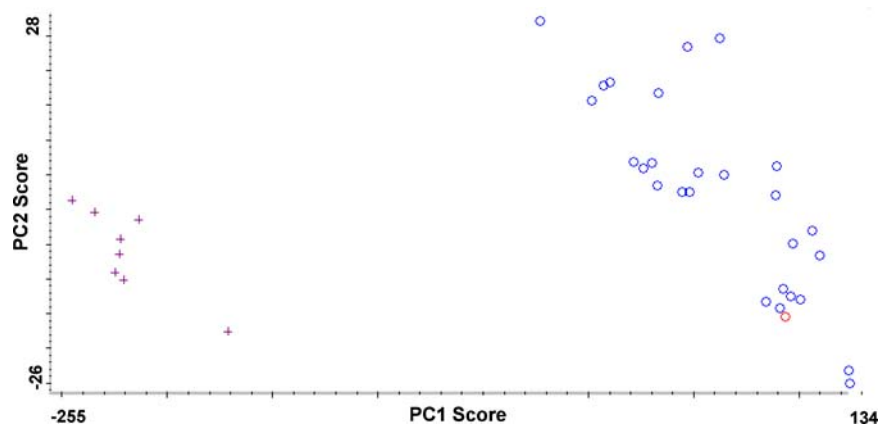
#### Correlation between NIR spectra and AAS data

The calibration and validation results for potassium, magnesium, zincum, and iron are listed in Table 6. The PLS models are good for potassium and magnesium, which shows that concentration of potassium and magnesium can be well predicted by NIRS.  $R_{cal}$  for potassium and magnesium are 0.958

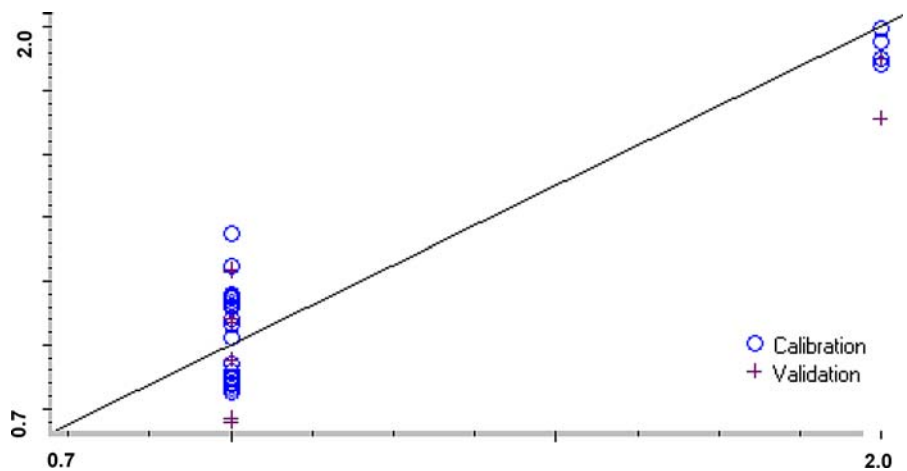
**Table 5** Calibration and validation statistics from the PLSR models using potassium and magnesium

Trace metal combination	$R_{cal}$	RMSEC	RMSEP	Percentage of samples correctly classified (calibration)		Percentage of samples correctly classified (validation)	
				Shaoxing	Jiashan	Shaoxing	Jiashan
Potassium and magnesium	0.914	0.169	0.246	100	100	100	100

**Fig. 5** Sample score plot for PC1 and PC2 of AAS data for 30 Shaoxing rice wine samples and 8 Jiashan samples



**Fig. 6** AAS predictions of geographical origins of Chinese rice wine samples in the calibration and validation sets using potassium and magnesium



and 0.885, respectively, and  $R_{val}$  are 0.861 and 0.700, respectively. However, the prediction performance of zincum and iron are worse than those of potassium and magnesium, which is most likely due to the narrow range and low value of the two elements in the Chinese rice wine samples (Table 3).

Although it was difficult to assign wavelengths to specific molecular absorptions in the NIR region, especially in the

case of dissolved metal ions in solution, the NIR absorption of metal ions was most likely associated with changes in localized hydrogen bonding around the metal cations in solution [19]. Moreover, the absorption peak of water could be changed by ion–dipole interaction between water molecules and inorganic ions [24], which is the possible reason for geographical discrimination by NIRS and AAS.

**Table 6** Calibration and validation results for potassium, magnesium, zincum, and iron in the Shaoxing rice wine samples

Metal	Calibration		Validation	
	$R_{cal}$	RMSEC (mg l <sup>-1</sup> )	$R_{val}$	RMSEP (mg l <sup>-1</sup> )
Potassium	0.958	12.10	0.861	16.90
Magnesium	0.885	3.78	0.700	4.17
Zincum	0.351	0.25	0.621	0.13
Iron	0.403	0.73	0.584	0.58

**Conclusions**

Discriminant models based on NIR transmission spectra with PLSR method appeared to be good for classification of Chinese rice wines of different geographical origins (Shaoxing and Jiashan). The NIR spectra in the range of 1300–1650 nm was found to provide the best results, with 100% classification of wines of the two geographical origins. AAS with

PLSR method showed that two variables, potassium and magnesium were sufficient to discriminate between Shaoxing and Jiashan rice wine, with 100% classification for wines of the two categories, respectively. The AAS results were in consistent with NIRS, which indicated that AAS could be used to validate the discrimination results. Meanwhile, potassium and magnesium were well predicted by quantitative models based on NIR spectra and AAS data. Thus, it can be concluded that NIRS together with AAS technique offers a robust method for discrimination between Chinese rice wines of 'Shaoxing' geographical origins considered here.

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