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Monitoring of alcohol strength and titratable acidity of apple wine during fermentation using near-infrared spectroscopy

Bangzhu Peng [a,*], Na Ge [a], Lu Cui [b], Hui Zhao [b]

[a] Key Laboratory of Environment Correlative Dietology, Ministry of Education, College of Food Science & Technology, Huazhong Agricultural University, 430070, Wuhan, Hubei, China

[b] College of Food Science & Engineering, Northwest A & F University, 712100, Yangling, Shaanxi, China

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ABSTRACT

Rapid development in apple wine industry has raised a growing need for effective fermentation process monitoring. The alcohol strength and titratable acidity have been used as indicators for process control and optimization in apple wine making. The objective of this study was to investigate the efficacy of using near-infrared (NIR) spectroscopy to monitor and assess the process parameters during apple wine fermentation. A simple and rapid NIR spectroscopy method was developed for simultaneous determination of alcohol strength and titratable acidity in apple wine. The selected spectral regions of 6101.9–5446.2 cm^{-1}, and 11,995.4–7498.1 cm^{-1} were pretreated by second derivative (SD) and straight line subtraction (SLS) for alcohol strength and titratable acidity, respectively, prior to developing calibration models using partial least squares (PLS) regression with cross-validation. The highest R_c^2 and the lowest *RMSECV* in the calibration set were obtained for the alcohol strength (0.923 and 4.63 mL/L) and for the titratable acidity (0.930 and 0.264 g/L). The NIR calibration models showed good correlation of determination and low predictive errors. Application of the NIR calibration models demonstrated the feasibility of NIR spectroscopy to be used as a quality control tool for monitoring the apple wine fermentation process.

1. Introduction

Apple wine is widely used as a fermented alcoholic beverage in most countries. The steadily-growing diversity of commercial apple wine, coupled with increasing consumer demand, has prompted a need to monitor their major chemical composition changes for process control and quality assessment in order to better manage fermentation process. Alcohol strength and titratable acidity have been used as indicators during apple wine fermentation process for quality control and optimization in apple wine industry; hence, it is of great importance to monitor the changes of alcohol strength and titratable acidity in fermentation process for improving apple wine quality. A variety of methods have been developed for monitoring the parameters of alcohol strength and titratable acidity in previous research, including chemical assays, pH meters, and high pressure liquid chromatography, etc. (S.A.C., 2008; Satora, Tarko, Sroka, & Blaszczyk, 2014; Wilson, Maguer, Duitschaever, Buteau, & Allen, 2003; Ye, Yue, Yuan, & Li, 2014; Zhang, Fang, & Li, 2011), but these methods generally had the disadvantages of sample preparation, reagent consumption and complicated operation with time-consuming that might hamper the quality of final apple wine. With the development of brewing technology and the demand of quality control, it is important to perform fast and accurate determination of target composition for cost saving and online monitoring product quality (Blanco, Peinado, & Mas, 2004).

Nowadays, apple wine brewing industry needs to introduce and develop more advanced analysis method online in order to get reliable feedback data in time, which applied to guide the fermentation process. The monitoring of a fermentation process often requires fast and frequent measurements, which makes Near-infrared (NIR) spectroscopy an excellent candidate for online analysis to provide rapid determination and straightforward monitoring. Bioprocess monitoring is becoming one of the most challenging applications of NIR spectroscopy (Lachenmeier, 2007). With the application of chemometric techniques, a single spectrum can be subjected to many different calibration models, to measure many compositions in various types of food (Shen et al., 2010). NIR spectroscopy is a widely used rapid and non-destructive technique

* Corresponding author.
E-mail address: bzpeng@mail.hzau.edu.cn (B. Peng).

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for quality measurements and control in food and agricultural product analyses (Antonucci et al., 2011; Chen, Ding, Cai, Sun, & Zhao, 2012; Chen, Zhao, Fang, & Wang, 2007; Grassi, Amigo, Lyndgaard, Foschino, & Casiraghi, 2014; Pouliot, Paquin, Martel, Gauthier, & Pouliot, 1997; Xie, Ye, Liu, & Ying, 2011), and is currently an ideal alternative to traditional analytical techniques for monitoring bioprocess (McLeod et al., 2009). With the application of chemometric techniques, a single spectrum can be subjected to many different calibration models, to measure many compositions (McLeod et al., 2009), and this modern analytical technology has been used as a well-suited method for general process real-time monitoring, which is of great interest for many wine makers to conduct process control. Di Egidio, Sinelli, Giovanelli, Moles, and Casiraghi (2010) obtained good calibration models for the prediction of the main compositional changes with FT-NIR to evaluate alcoholic fermentation online in red wine. Buratti et al. (2011) demonstrated that the non-destructive method of NIR are suitable for the monitoring of must-wine fermentation giving crucial information about the quality of the final product in agreement with chemical parameters. Giovenzana, Beghi, and Guidetti (2014) carried out a preliminary study to verify the possibility of employing a device based on NIR spectroscopy, directly on the production line of craft beer. Grassi et al. (2014) investigated the capability of FT-NIR spectroscopy to monitor and assess process parameters in beer fermentation with two different yeast strains at different temperatures, and the results showed that FT-NIR spectroscopy demonstrated to be a perfectly suitable quantitative method to be implemented in the production of beer. NIR spectroscopy has already shown promise as a rapid and non-destructive method for determining various compounds simultaneously in the fermentation industry (Liang et al., 2013). Nevertheless, less research exists on the application of NIR spectroscopy for monitoring and assessment of changes in relevant physico-chemical parameters in the apple wine fermentation process. Therefore, much more intensive work is needed to really evaluate the feasibility of NIR spectroscopy in monitoring apple wine fermentation process. The objectives of this study were to develop fourier transform near infrared (FT-NIR) spectroscopy calibration models for predicting alcohol strength and titratable acidity of apple wine in order to monitor fermentation process. In addition, the feasibility of using FT-NIR spectroscopy to monitor these two indicators simultaneously during the fermentation of apple wine was explored.

2. Materials and methods

2.1. Apple wine fermentation and sampling

Ripe *Fuji* apples used in this study were harvested from different commercial orchards located at Luochuan county in northwest China, and stored in a cold room at 4 °C until use. Apple juice was obtained by squeezing the mixed apples from different orchards from *LZ* juice extractor with a vacuum pressing and filtration system in room temperature (Gongda company, Wenzhou, China), during which the sodium bisulfite of 100 mg/L was added to apple juice in order to inhibit the bacteria growth immediately (Peng, Yue, & Yuan, 2008). The total sugar and pH of all juice samples for fermentation were then adjusted to 200 g/L with saccharose and 3.4 with 0.5 M hydrochloric acid, respectively. A total of nine apple wine fermentation trials were conducted using three different yeasts (*Saccharomyces cerevisiae* 1750, PF12 and WP45) at three temperatures of 17, 20 and 23 °C according to dry type brewing technology in laboratory. The apple wine samples were collected right after yeast inoculation (0 h, starting time), and then every 12 h until the 10th day of fermentation in each trial. When sampling, about 30 mL of samples were discharged out directly from fermented supernatant. One hundred and sixty samples were collected from eight trials for developing the models of quantifying the alcoholic strength and titratable acidity, while another trial with twenty collected samples was used to evaluate the feasibility of FT-NIR spectroscopy in monitoring apple wine fermentation process.

2.2. Chemical analysis

The performance of a quantitative NIR model is mainly dependent on the performance of reference method (Bock & Connelly, 2008). Poor precision and accuracy of the reference method will limit the performance of the NIR model. Therefore, it is important to ensure the targeted parameter as close to the true value as possible. In this study, samples were centrifuged at 5000 rpm for 5 min and supernatants were collected for chemical analysis. Alcoholic strength was measured by using an PT-1 alcoholmeter (Pute company, Beijing, China), immersed in a distillate at a temperature of 20 °C; titratable acidity was determined by acid–base titration method according to SAC standard GB/T 15038 (S.A.C., 2008) and the results were expressed as malic acid. All reagents used were of analytical grade, and related solutions were prepared by ultrapure water produced with a millipore purification system (Millipore, Mississauga, Canada) in the experiments. All analyses were done in triplicate.

2.3. Spectrum collection

FT-NIR spectra collected directly from the supernatant of apple wine samples using a MPA FT-NIR spectrometer (Bruker Optics, Germany) in the range of 12,000 to 4000 cm^{-1}, which equipped with an InGaAs detector and a liquid fiber optic probe with a 2 mm path length. An air background was employed as a probe reference, and the liquid probe directly immersed into samples for collecting the spectra at 25 °C. The spectral resolution was set at 8 cm^{-1} with an interval of 2 cm^{-1} and the spectra were recorded performing 20 scans for both the reference and tested samples. The spectra were recorded in triplicate and an average spectrum was collected to ensure an adequate signal-to-noise ratio for the subsequent analysis. The NIR instrument control, spectra scanning, and initial data processing were performed by using OPUS software version 5.5 (Bruker Optics, Germany).

2.4. Preprocessing of spectra and chemometric analysis

NIR spectra preprocessing in multivariate calibration is still trial and error due to the lack of sufficient prior information and an incomplete knowledge of the raw data (Xu et al., 2008). How to select a proper preprocessing method depends largely on the expertise and experience of practitioners. To establish best calibration model, spectra data from samples need to conduct different treatments for reducing noise interference and increasing the resolution of small absorbance bands (Huang, Yu, Xu, & Ying, 2008). Chemometric analysis is usually used as a tool to correlate the effective information in the NIR spectra signal to a number of latent variables or factors, constrained by a set of calibration reference data (Massart et al., 1997). Partial least squares (PLS) regression is a well-established chemometric technique whereby factors are derived by taking into account the variation in the spectral data that is relevant for explaining variation in the characteristics of interest in the original samples (Williams, 2001).

In this study, spectra preprocessing was made by straight line subtraction (SLS), first derivative (FD), second derivative (SD), vector normalization (VN), min–max normalization (MMN),