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Fast compositional analysis of ramie using near-infrared spectroscopy

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ABSTRACT

Rapid and accurate determination of chemical compositions of ramie is crucial to its application. In this paper, calibration models were established using near-infrared (NIR) spectroscopy to predict the main chemical compositions of ramie. A wet chemical analysis method which was improved on the basis of Chinese national standard for getting calibration data was used in this paper. NIR data of 59 ramie samples were collected using Fourier transform near-infrared spectrometer. The calibration models of chemical compositions of ramie were derived by partial least square (PLS) regression. Prediction of chemical composition of independent ramie samples showed that these models were rapid and accurate in the chemical composition analysis, giving residual predictive deviation (RPD) value higher than 2.5. Such NIR calibration models can be utilized by ramie fiber manufacturers and breeding workers, in order to better manage the degumming process and evaluate the quality of ramie varieties.

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1. Introduction

Ramie is widely used in textile industry due to its good properties (Zheng, Du, & Zhang, 2001); it is also a good raw material for reinforced composites (Angelini, Lazzeri, Levita, Fontanelli, & Bozzi, 2000; Kishi & Fujita, 2008). As a kind of bast-fibre material, ramie needs degumming process before it can be used for textile industry. Many degumming methods were investigated for the application of ramie (Brühlmann, Leupin, Erismann, & Fiechter, 2000; Zheng et al., 2001; Basu & Saha, 2009; Saikia, Boruah, & Samanta, 2009). It is noteworthy to point out that the contents of hemicellulose, cellulose and gum in ramie play an important role in selecting the technological parameters for the degumming process in industry and laboratory. And they can provide important information for breeding work. Moreover, hemicellulose has a great value for papermaking (Bhaduri, Ghosh, & Deb Sarkar, 1995). Therefore, it is important to analyze hemicellulose, cellulose and gum in ramie quantitatively before it can be used in industry and other areas.

China is the provenance of ramie, and has the most varieties and productions of ramie in the world. A national standard which is named "method of quantitative analysis of ramie chemical components" (ramie criterion in short) (Jiang & Shao, 1986) has been promulgated as wet chemical analysis method for the determi-

nation of ramie chemical compositions. In most cases, the ramie criterion is the only method to quantitatively analyze the chemical compositions in ramie and other plant fibrous materials in textile industry in China. However, to the best of our knowledge, the wet chemical analysis has its drawbacks such as time consuming, complicated steps, potential dangers to tester and pollution to environment (Kelley, Rowell, Davis, Jurich, & Ibach, 2004; Zheng et al., 2001). Therefore, simple and fast analysis of the chemical compositions of ramie is essential in textile industry and other fields.

As a spectral analysis technique, NIR technique can achieve the requirement of the fast and multiple composition analysis (Cornish, Myers, & Kelley, 2004; Jin & Chen, 2007). What is more, the sample does not need pretreatment (Jin & Chen, 2007), so the test can be free of chemical substances. In recent years, a lot of research works have shown the value of fast analyzing chemical composition of plant materials using NIR technique. Ye et al. (2008) demonstrated the potential of Fourier transform near-infrared (FT-NIR) techniques in quantitatively analyzing chemical composition of cornstover. NIR models were also found to be suitable for fast and accurate analysis of the chemical composition in maize silage (Cozzolino, Fassio, Fernández, Restaino, & Manna, 2006), rice straw (Jin & Chen, 2007), and tobacco (Zhang, Cong, Xie, Yang, & Zhao, 2008). Kelley et al. (2004) got the high-quality calibration model for most of biomass components in various agricultural samples. However, very limited research work that applied NIR spectroscopy to analyze chemical compositions of ramie was found.

In this study, Fourier transform near-infrared spectroscopy has been used to establish the calibration models of the contents of hemicellulose, cellulose and gum in ramie. The models derived

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from this work can be used to fast analyze the chemical compositions of ramie.

2. Methods

2.1. Sample preparation

A total of 59 samples were collected from the research center of bast-fibre plant in Hunan Province, People's Republic of China. The weight of each sample was more than 200 g. After the storage of all the samples (all the samples were tiled <1cm thick) under the ambient condition $(20-30\,^{\circ}\text{C}$ and <50% humidity) for more than one week, around 100 g of each sample was cut into pieces of $3\,\text{cm} \times 0.5\,\text{cm} \times 0.5\,\text{cm}$, well mixed, then milled to powder which was allowed to pass #60 mesh screen (\sim 250 μ m), and oven-dried at $105\,^{\circ}\text{C}$ for at least 6 h until a constant weight is obtained, finally cooled to $20\,^{\circ}\text{C}$ in desiccator. $20\,\text{g}$ of the powder was placed into plastic automatic sealing bag for NIR data acquisition, and wet chemical analysis was carried out using another $20\,\text{g}$ of powder.

2.2. FT-NIR data acquisition

About 20 g of each sample was put in a non-NIR absorbing specimen cup, the sample spectra were collected in diffuse reflectance mode. MPA Fourier transform near-infrared spectrometer (BRUKER OPTICS, Germany) equipped with a 10 cm integrating sphere was used in this experiment. The spectrum covers a range of 12,000–4000 cm⁻¹ with a spectral resolution

of 8 cm⁻¹. Each spectrum is the average of 64 co-additions of scans

2.3. Wet chemical analysis

Reliable calibration data is one of the key factors to determine a successful application of NIR techniques for the fast chemical characterization (Ye et al., 2008). As indicated in Section 1, ramie criterion is the only wet chemical analysis method, which is shown in Fig. 1. Firstly, the samples are dried at 105 °C for at least 6 h, then 3 dried samples are prepared separately, each sample is about 5 g. The procedure of sample 1: Benzene/Ethanol for extraction of wax →water boil for extraction of water soluble matter → dilute Ammonium oxalate for extraction of pectin \rightarrow Soda boil for extraction hemicellulose. Sample 2 is treated using klason method to analyze lignin, and sample 3 is treated using soda boil to analyze gum. But we found that some flaws make the ramie criterion inaccurate for some chemical composition analysis. The content of hemicellulose was considered to be the weight loss after the soda boil using ramie criterion. However, the lignin can be partially dissolved during soda boil because of the nucleophilic reaction (Tan, 2002; Yang, 2001), and then the content of hemicellulose was a little higher than the true value because some of lignin (lignin A) was mistaken for hemicellulose. The content of cellulose was calculated by 100% deducting other chemical composition contents using ramie criterion, therefore, it was inaccurate, either.

So we carried out a thorough investigation of the principle for the ramie criterion, and optimized the traditional criterion. A new

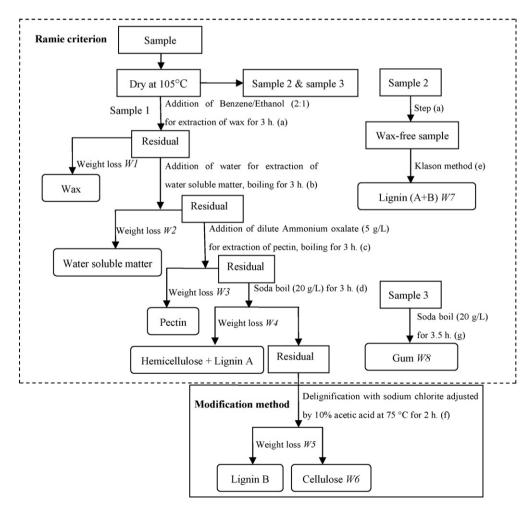


Fig. 1. Schematic diagram for the wet chemical analysis process.

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