Rapid Determination of Urea Formaldehyde Resin Content in Wood Fiber Mat Using Near-infrared Spectroscopy

Qinglin Yu,[a] Hanwen Zhu,[a] Guanben Du,[b] Zhangjing Chen,[c] and Zhong Yang [a,*]

Monitoring the process is crucial for ensuring high quality in wood-based panel production. Interest in the distribution of resin in fibers and particles has increased during the last couple of decades. This study considered the potential to determine urea formaldehyde (UF) resin content in fiber mat using near-infrared (NIR) spectroscopy. Fiber mats with various resin contents were investigated with NIR combined with the partial least squares (PLS) regression and root mean square error of calibration and validation (RMSECV). The external factors, such as the distance between the fiber optic probe and the sample surface and light sources, were also evaluated. The results showed that this technique can sufficiently determine the resin content in the resinous fiber mat with accuracy of up to 95%. The light sources and the distances from the probe to the surface did not significantly influence the discrimination and prediction of resin content.

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*Contact information: a: Research Institute of Wood Industry, Chinese Academy of Forestry, Beijing 100091, China; b: International Joint Research for Biomass Materials, Southwest Forestry University, Kunming 650224, China; c: Department of Sustainable Biomaterials, Virginia Tech University, Blacksburg, VA 24060, USA; *Corresponding author: zyang@caf.ac.cn*

INTRODUCTION

A major aspect of the industrial production of wood-based panels is quality control. In wood-based panels, an adhesive plays an important role in holding wood particles or fibers together. The most common adhesives used to produce fiberboard are urea-formaldehyde (UF) resins (Gavrilović-Grmuša *et al.* 2008). Urea formaldehyde is indispensable for the process of manufacturing wood products in the wood industry, especially in the production of wood-based panels and the gluing of furniture elements. Nevertheless, not only the type of resin, but also the distribution of the fibers is equally important for quality of final products. The resin distribution and resin content affect the mechanical properties of the wood-based panels (Andre *et al.* 2010; Irle *et al.* 2010; Taheri *et al.* 2016).

It is useful to have tools that can determine resin content and can be applied for online process determination. Many research studies have monitored the UF distribution in wood-based panels using various technologies, such as confocal laser scanning microscopy (CLSM) (Grigsby *et al.* 2005; Cyr *et al.* 2006), fluorescent UV spectroscopy (Radotić *et al.* 2006), and X-ray photoelectron spectroscopy (XPS) (Grigsby and Thumm

2004; Xing *et al.* 2005; Pakdel *et al.* 2008). However, these methods were unable to provide the rapid online determination of the resin content for better quality control.

Near-infrared spectroscopy (NIR) technique has gained widespread acceptance as a powerful tool with the potential to rapidly predict chemical and physical properties on the production line or with the use of handheld instruments (Cooper *et al.* 2011). In NIR spectroscopy, the wavelength between 800 to 2500 nm is used for the non-destructive measurement of organic materials such as agricultural products or foods (Tsuchikawa 2007). The NIR wavelength region has two major advantages: the high velocity and low cost of spectral acquisition that facilitates the collection of the data, as it requires little or even no preparation of the material (Schwanninger *et al.* 2011).

Recently, the NIR technique has gained more attention. It has been used as an on-line or on-site measurement of wood chemical composition (Alves *et al.* 2011; Lepoittevin *et al.* 2011), wood mechanical and physical properties (Schimleck *et al.* 2011; Alves *et al.* 2012), and even on wood species identification (Lazarescu *et al.* 2017; Ramalho *et al.* 2018). In the case of wood and biomass material, NIR spectra are collected in transmission or diffuse reflection mode. However, due to the weak penetration ability of NIR spectra into wood, the NIR diffuse reflective mode is widely used (Raymond *et al*. 2001; Tsuchikawa *et al.* 2001).

The NIR spectroscopy together with chemometric tools and multivariate techniques, such as partial least squares (PLS), has increased its applications in diverse academic and industrial fields (Wold and Sjöström 1998; Hulland 1999). An advantage of PLS compared to other chemometric methods is that strong collinearities are tolerated in X and the moderate amounts of missing data. Additionally, it can handle many Ys at the same time as well (Wold *et al.* 2002). Thus, it has come into wide use in the multivariate calibration of the NIR spectroscopy during the last couple of years (Jiang *et al.* 2007; Snel *et al.* 2018). Partial least squares was originally developed as a multivariate calibration method and has been widely used in discrimination analysis (Rambla *et al.* 1997). Because the partial least squares discriminant analysis (PLS-DA) was based on the regression model between the category variables of the sample and the NIR spectral characteristics, the category variables of the training set sample were assigned according to the amount of resin content. Savitzky-Golay smoothing was used as a preprocessing method to reduce the high frequency signal noise due to hardware set-up imperfections. Then, the first derivatives were used according to the Savitzky-Golay algorithm.

The objective of this research was to investigate the feasibility of the rapid determination of UF resin content in resinous fiber mat using the NIR spectroscopy technique combined with PLS analysis. The major external factors that could affect the results, such as the distance from the fiber optic probe to the sample surface and external light sources, were evaluated.

EXPERIMENTAL

Resinous Fiber Mat Preparation

The fresh eucalyptus (*Eucalyptus robusta*) fiber of 5% moisture content (MC) was obtained from the production lines at a wood-based panel plant (Fenglin Group, Nanning, Guangxi, China). When the length of the fiber was in the range 0 to 0.6mm, the weight percentage was 9.2%; when the fiber length was 0.6 to 2.5mm, the weight percentage was 78.8%; when the length of the fiber was 2.5 to 30mm, the weight percentage was 12.0%.

Urea formaldehyde was prepared at Shenglin Timber Slab Co., Ltd. (Yi County, Hebei, China) with a solids content of 50%. A variable amount of resin was mixed to 2000 g dry fiber using a glue spreader (XWT5, Changzhou Weierqiang Transmission Machinery Co., Ltd., Jiangsu, China). The blending time depended on the amount of UF added. The temperature during blending was controlled at 7 °C. Twelve resin contents, which are ratios of solid resin to oven-dry fiber were 2%, 4%, 6%, 8%, 9%, 10%, 11%, 12%, 14%, 16%, 18%, and 20%. Urea formaldehyde was blended and evenly distributed in wood fibers to form the resinous fiber mat. The mats were then placed on a petri dish with a diameter of 32 cm and a depth of 5 cm and evenly were pressed by hand in 10 min. After spreading the resinous fibers, the surface was compacted with a flat plate to make it even.

NIR Spectroscopic Method and Multivariate Techniques

NIR spectroscopic method

The NIR spectral information was captured with an ASD Lab Spec Pro spectrometer (Analytical Spectral Devices, Boulder, CO, USA) at a wavelength ranging from 780 to 2500 nm at a 1 nm interval. The diameter and thickness of the sample were 32 cm and 5 cm, respectively. The light-emitting diodes (TG905) were used as the illuminator. The fiber optic probe had a spot diameter of 50 mm. The hand-held fiber optic probe was set perpendicularly to the sample surface. The distance between the fiber optic probe and the sample surface was 55 mm. After the fiber with different resin contents were evenly mixed, 30 resinous samples were randomly selected to gather near-infrared spectra and then 30 spectra were averaged. The white reference spectra were obtained with the commercial microporous Teflon (JY-WS1) to assure 100% light reflection in the NIR range.

Partial least squares discriminant analysis

The software Unscrambler X 10.4 (CAMO Software AS, Oslo, Norway) was used for multivariate analysis. In the model, two-thirds of the samples were randomly chosen to be a part of the calibration group. The remaining samples were used as the validation group.

Reportable statistical parameters to describe the resulting calibration were the respective determination coefficient R_{cal}^2 and R_{cv}^2 and the root mean square errors and for calibration (RMSEC) and cross-validation (RMSECV), respectively.

To consider the uneven gluing on the production line, the different resin contents were divided into three categories: low "L", medium "M", and high "H". The L was defined at the range of 2 to 6% resin content; the M, at 8 to 12%; and the H, at 14 to 20%.

The statistical parameters, such as the category variable values (Y_p), the target values (Y_t), and deviation (D) of the prediction sub-set were calculated. If the Y_p was greater than 0.5 and D was less than or equal to 0.5, then the result indicated that the sample belonged to the corresponding category. If the Y_p was less than 0.5 and D was less than or equal to 0.5, then the result predicted the sample did not belong to the category. If the D was greater than 0.5, that meant that the discriminant was unstable.

The formula for Y_p was as follows,

$$Y_p = Y_t \pm D \quad (1)$$

where Y_t is the predicted target value of 1. The deviation indicated that the sample used for prediction was similar to the samples used to make the calibration model.

Distance from the fiber optic probe to the sample surface

To explore the effect of the distance from the fiber optic probe to the sample surface on the accuracy of the model, three distances of 25, 55, and 85 mm with the corresponding spot diameters of 30, 50, and 70 mm, respectively, were used. The NIR spectral and the white reference with 100% reflectance were acquired on the corresponding samples simultaneously. The various resin contents consisted of 4%, 8%, 12%, and 20%. The spectrum was collected per second on the sample surface, and 30 spectrums were collected for each resin content.

External light sources

To simulate the conditions with the different external light sources in the production line, four light sources of the indoor fluorescent light, dark, luminescence, and the warm light, were selected in this experiment. The distance of the fiber optic probe to the sample surface was 55 mm with the spot diameter of 50 mm. The colour temperature of luminescence was 6000 K, and color temperature of warm light was 3000 K. The external light sources were 30 cm away from the sample, and the angle between the irradiation direction and the sample surface was 45°. Three resin contents used were 4%, 12%, and 20%. Thirty spectra were collected at each adhesive content.

RESULTS AND DISCUSSION

NIR Spectra

The NIR spectra with the absorption bands in the range of 500 to 2500 nm collected from the different resin contents are shown in Fig. 1.

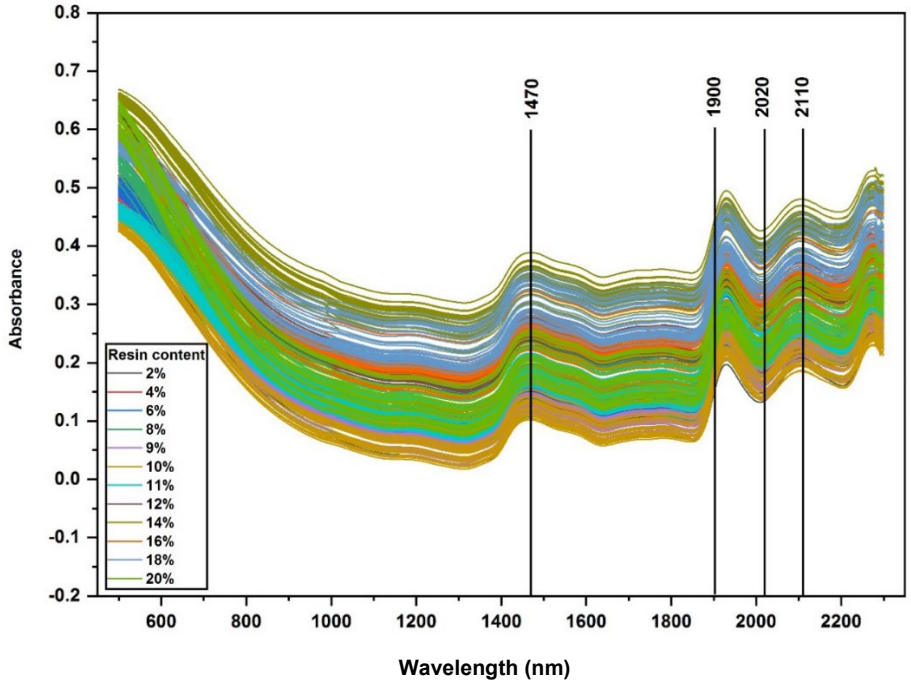

Fig. 1. NIR spectra for various resin content in resinous fiber

The curves are similar for the different absorption intensities (spectrum peaks). There are some overlaps in the spectra in Fig. 1 due to the non-uniformity of the material. Figure 3 shows three spectra of resin content for the ranges of L, M, and H. The fiber mats with a resin content of 18% (high, H) had a higher absorbance; the fiber mats with a resin content of 12% (medium, M) had medium absorbance; and the fiber mats with a resin content of 6% (low, L) had lower absorbance. Within a certain range, higher resin contents resulted in higher absorbance of the resinous fiber mats.

Although the NIR spectral region provides much information, the spectra-structure correlations were still more difficult to establish in the NIR region than in the mid-IR (Schwanninger *et al.* 2011). Figure 2 shows the first derivatives of the spectra of the samples with the resin contents.

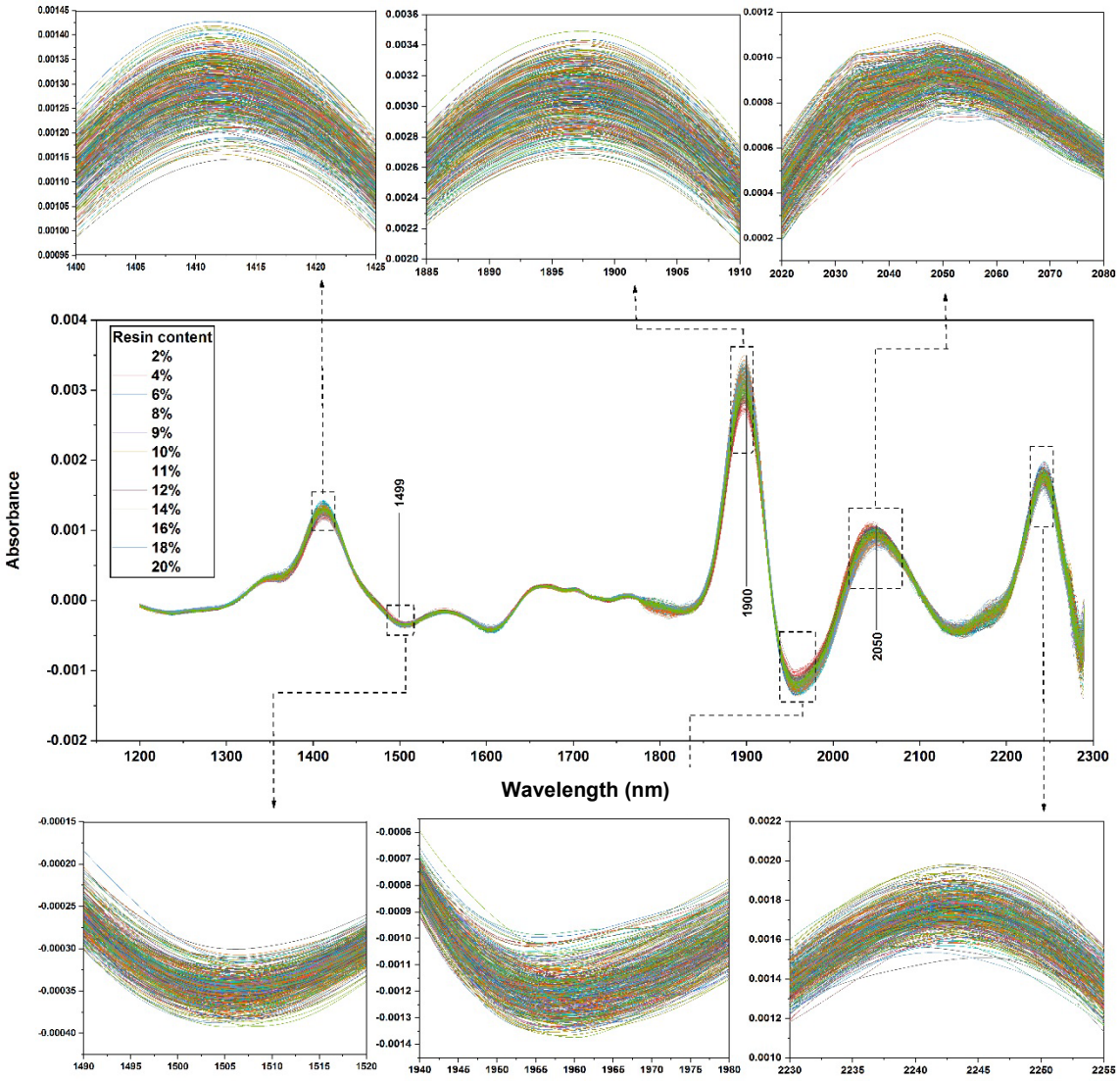

Fig. 2. First-derivative of NIR spectra for resin content in resinous fiber; wavelength ranging from 1200 to 2300 nm

Characteristic wavelengths representing resin can generally be found at 1470 nm and 1499 nm (1st overtone NH stretching) (Workman and Weyer 2008). Niemz *et al.* (1992) used the wavenumbers 1900, 2020, and 2110 nm for analyzing wood chips with UF resin. Rials *et al.* (2002) measured the physical properties of medium-density fiberboards and infrared wavelengths of 2050 and 2250 nm characteristic for the N-H overtones of the UF adhesive. For the secondary compounds, the determination was limited to a mass fraction greater than approximately 0.1 to 0.5%, although it also was related to the functional groups presented in these compounds (Schwanninger *et al.* 2011). Homogeneous materials were the reason for spectral overlap. At the same time, for the near-infrared spectroscopy, the multivariate data analysis was necessary because a nonlinear relationship existed. The PLS-DA model can be useful to analyse the chemical composition in a NIR spectrum.

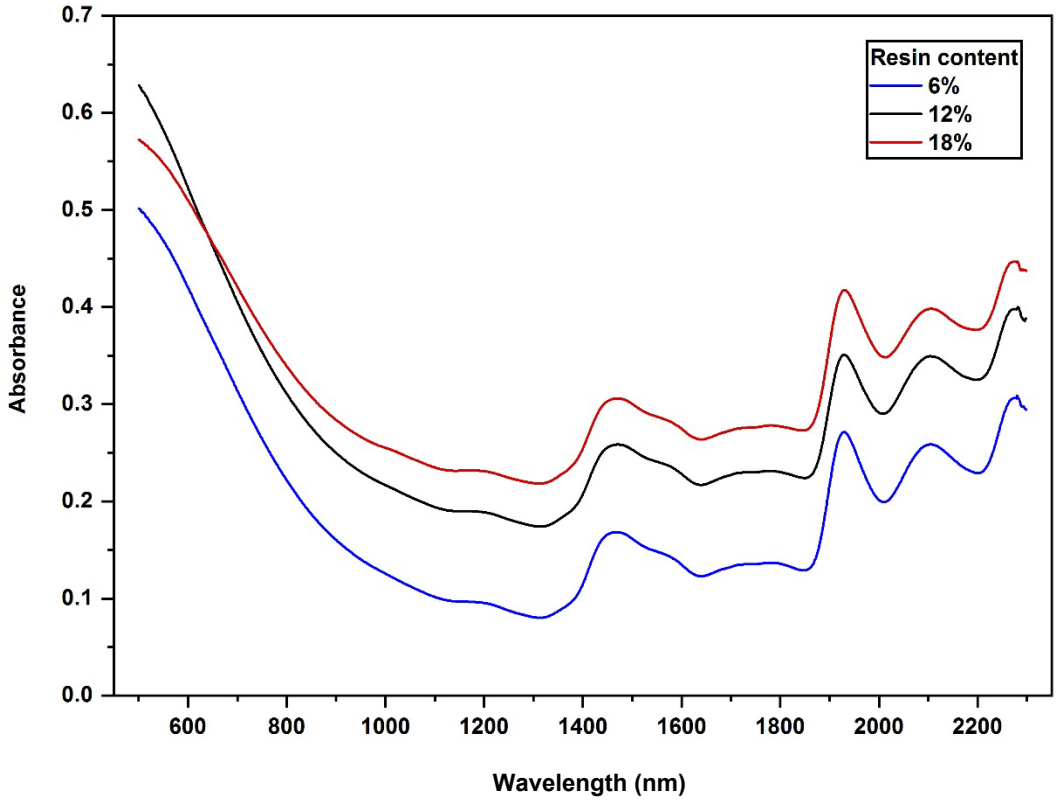

Fig. 3. NIR spectra for three resin contents (6%, 12%, and 18%) in resinous fiber

PLS-DA Analysis

The classification models were developed based on the raw spectral (500 to 2300 nm) using the PLS-DA algorithm according to a discriminant approach. Table 1 shows the statistics of calibration. The R_{cal}^2 values were 0.95, 0.95, and 0.97 with low RMSEC values of 0.10, 0.11, and 0.08, respectively. For the validation, the R_{cv}^2 values were 0.87, 0.86, and 0.92 corresponding to the RMSECV values of 0.16, 0.18, and 0.13, respectively. The higher correlations were found for the large ratios of adhesive to fiber. The low coefficient of discrimination was found in the heterogeneous resinous fibers and the rough surfaces (Campos Barbosa *et al.* 2009).

Due to the fact that raw materials in fiberboards industry are always changing, controlling the quality of products is required to maintain the same quality standard. To minimize these disturbances, more measurements could improve the situation.

Table 1. Reportable Statistics of Calibration of Gluing Quantity Models

Gluing Quantity	Range of Resin Content	Calibration		Validation	
		R_{cal}^2	RMSEC	R_{cv}^2	RMSECV
L (n = 90)	2 to 6%	0.95	0.10	0.87	0.16
M (n = 150)	8 to 12%	0.95	0.11	0.86	0.18
H (n = 120)	14 to 20%	0.97	0.08	0.92	0.13

Tables 2 and 3 show the statistical results of the accuracy values obtained for the determination of gluing samples using the PLS-DA model. The Y_p values of the sample were greater than 0.5 and the deviation values were less than 0.5. Using the discriminant analysis of PLS-DA, the accuracy values of the three gluing quantities were 100%. It was possible to figure out the resin content in the wood fiber mat using near-infrared spectroscopy combined with the PLS-DA model. The NIR can be an useful tool for online process control (Sjöblom *et al.* 2004; Gosselin *et al.* 2011; Tsuchikawa and Kobori 2015; Schimleck *et al.* 2020). Spectral preprocessing techniques were also employed with the Savitzky-Golay smoothing and with the first derivative spectra providing slightly improved results. Therefore, it was feasible to rapidly detect the UF resin content in resinous fiber mats using NIR spectroscopy combined with PLS analysis during the gluing process.

Table 2. Discrimination Analysis for the Resinous Fiber Mats Using PLS-DA Model with $Y_t = 1$

Category	Resin Content	Y_p			Deviation		
		Avg.	Max.	Min.	Avg.	Max.	Min.
L (n = 30)	2%	0.964	1.25	0.69	0.138	0.16	0.12
	4%	0.884	1.09	0.74	0.147	0.19	0.13
	6%	0.997	1.20	0.87	0.138	0.15	0.12
M (n = 50)	8%	0.947	1.16	0.57	0.172	0.20	0.14
	9%	0.944	1.19	0.55	0.191	0.24	0.16
	10%	0.902	1.08	0.61	0.214	0.23	0.2
	11%	0.947	1.14	0.78	0.199	0.23	0.17
	12%	0.806	1.30	0.55	0.207	0.24	0.18
H (n = 40)	14%	1.004	1.08	0.92	0.13	0.15	0.12
	16%	0.925	1.14	0.70	0.113	0.14	0.10
	18%	0.923	1.31	0.73	0.139	0.16	0.12
	20%	0.843	1.11	0.51	0.127	0.14	0.12

Table 3. Discrimination Accuracy Rates for the Resinous Fiber Mat with PLS-DA Models

Gluing Quantity	Range of Resin Content	Correctness	Errors	Discrimination Accuracy Rate
L (n = 30)	2 to 6%	30	0	100%
M (n = 50)	8 to 12%	50	0	100%
H (n = 40)	14 to 20%	40	0	100%

External Factors That Could Affect the Resinous Determination

Distance from fiber optic probe to the measured surface

The accuracy values affected by the distances from the fiber-optic probe to the sample surface on samples of gluing fibers are shown in Table 4. The accuracy values for high resin contents were 100% when the probe was 25 mm and 85 mm apart from the test surfaces. However, when the resin content was in the medium range (8 to 12%), the accuracy value decreased to 95%. The results indicated that the various resin contents were discriminated using the PLS-DA model combined with NIR spectroscopy technology. The high accuracy values show that the distance between the probe and the test surface may not significantly influence the results of discrimination. Theoretically, under certain conditions, a farther distance and a larger spot diameter results in a larger spectrum collection range. However, the absorbance of the near-infrared spectrum was also weakened. The spot diameter was proportional to the distance in a certain range. However, as the distance increased, the effective illumination boundary of the light source was not easy to determine. There was no need to strictly limit the distance from the fiber optic probe to the surface of material conditions of the production line.

Table 4. Discriminated Accuracy Rate for the Different Distance between the Fiber Optic Probe and the Measured Surface

Gluing Quantity	Range of Resin Content		Distance		
			25 mm[a]	55 mm[b]	85 mm[c]
L (n = 10)	2 to 6%	**Correctness**	10	10	10
		Errors	0	0	0
		Accuracy Rate	100%	100%	100%
M (n = 20)	8 to 12%	**Correctness**	20	19	20
		Errors	0	1	0
		Accuracy Rate	100%	95%	100%
H (n = 10)	14 to 20%	**Correctness**	10	10	10
		Errors	0	0	0
		Accuracy Rate	100%	100%	100%

[a] The corresponding spot diameter was 30 mm.
[b] The corresponding spot diameter was 50 mm.
[c] The corresponding spot diameter was 70 mm.

Light sources

Table 5 also shows the discriminated results of the different light sources using PLS-DA modelling and predicting. The discrimination accuracy rates for four types of light sources were nearly the same, about 100%. It seems that light sources did not affect the prediction results. There have been a few studies on the effects of external light sources using NIR. There was no need to strictly designate the light sources conditions of the production line.

Table 5. Discriminate Accuracy Rates for Various Light Sources

Gluing Quantity	Range of Resin Content		Light Sources			
			Dark	Fluorescent Light	Luminescence	Warm Light
L (n = 10)	2 to 6%	Correctness	10	10	10	10
		Errors	0	0	0	0
		Accuracy rate	100 %	100%	100%	100%
M (n = 20)	8 to 12%	Correctness	20	10	20	20
		Errors	0	0	0	0
		Accuracy rate	100 %	100%	100%	100%
H (n = 10)	14 to 20%	Correctness	10	10	10	10
		Errors	0	0	0	0
		Accuracy Rate	100 %	100%	100%	100%

CONCLUSIONS

1. The near infrared (NIR) spectroscopy technology combined with partial least squares discriminant analysis (PLS-DA) ranging from 500 to 2500 nm can be useful to instantaneously determine UF resin presence in resinous fiber mats with accuracy of up to 95%.
2. The model correlation using raw NIR spectra were strong in both calibration and validation. Strong correlations were obtained in calibration and validation yielding high R^2_{cal} and R^2_{val} values of 0.95 to 0.97 and 0.86 to 0.92 with low root mean square error of calibration (RMSEC) and root mean square error of calibration and validation (RMSECV) of values of 0.08 to 0.11 and 0.13 to 0.16, respectively.
3. The effects of external factors, such as light sources and distances from the fiber optic probe to the measured surface, were not significant in the rapid determination of the resin contents.

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