Application of near infrared spectroscopy for quality evaluation in Para rubber industry

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Abstract. Quality of products manufactured from Para rubber was an important aspect for merchandizing. Near infrared spectroscopy in a wavelength range of 900 to 1700 nm, which was a rapid and non-destructive technique, was applied to create quality predicting models for various Para rubber products. Near infrared hyperspectral imaging (NIR-HIS) technique was implemented to predict dry rubber content (DRC) of cup lump. Classification of adulterated Para rubber sheet was investigated using NIR-HSI. And NIR portable system was also developed for moisture content determination of Para rubber timber. The results showed that the DRC of the cup lump could be predicted with high accuracy with the coefficient of determination (R²) of 0.99 and root mean square of error of prediction (RMSEP) of 0.64%. Regarding the Para rubber sheet, the sulphuric acid coagulated sheets was classified from the formic acid coagulated sheet very good accuracy of 98.3%. Finally, The NIR portable system was developed and could be used to determine the moisture content of the timber with R² and RMSEP of 0.96 and 5.37%, respectively. The application of NIRS was successful and provided feasibility for development into a future commercial use.

1. Introduction
Para rubber is one of the strategic crops in Thailand as it generates very high income and has promoted Thailand to one of the biggest exporters of Para rubber. At present, farmers commonly make rubber cup lumps from latex to add value to the latex. The cup lump can be further used to produce rubber blocks. Merchandizing the Para cup lump is based on the dry rubber content (DRC) in the cup lump. However, the buyer evaluates the DRC by visual inspection which is a subjective method and depends on the experiences of the buyer. Rubber sheet is one form of early processing from the latex. The popularity of this form is mainly attributable to the low cost and simplicity of the processing machinery. One common problem in the rubber sheet processing is the rubber sheet adulteration, which occurs during the coagulation process by the addition of acids other than formic acid. The common methods for determining the type of acids used in the Para rubber sheet are based on parameters such as Mooney viscosity or the plasticity retention index. However, these common methods are time-consuming and destructive. Para rubber wood is product from Hevea trees that give low content of latex. The low latex producing trees are normally fell and transformed to sawn timber.
for exporting. The sawn timber from these trees have made a lot of profit to Thailand. Before exporting, sawn timber Hevea wood must be checked to ensure that the moisture content is lower than 12% dry basis following Thai Industrial Standards Institute. The sawn timber companies generally use commercially available pin moisture meter and digital moisture meter to determine the moisture content. However, the pin meter can only be used to measure the moisture content at a specific point and makes hole damage in the wood. Regarding the digital moisture meter, specific gravity of wood must be known to program in meter. In order to maintain the quality of the products from the Para rubber, a rapid and nondestructive testing technique is needed to retain standards.

NIR spectroscopy is a type of high-energy vibrational spectroscopy performed in the wavelength range 750–2500 nm. NIRS is a fast and nondestructive analytical method. The NIR light interacts with the molecules of C-H, O-H and N-H causing the bond to vibrate and absorbance occurrence. Therefore, the chemical content in the organic materials is sensitive to the NIR light. The absorbance of the NIR light is related to the quantity of the chemical content according to Beer-Lambert law. It has proven its effectiveness for both qualitative and quantitative analyses in several fields. Detection limits are typically 0.1%. Hyperspectral imaging has been integrated with NIRS which provides spatial information other than spectral information of the sample. In addition, more spectral information called hypercube is acquired by NIR-HSI. One important benefit of the NIR-HSI is that the color-mapped image of the chemical can be produced from the NIR-HSI image displaying the distribution of the chemical content across the sample surface.

Our research group has investigated the application of NIRS to evaluate the dry matter content (DRC) in the cup lump. The classification of adulterated rubber sheet was also achieved by the use of NIR-HSI. Finally, the moisture content determining system based on a portable spectrometer was developed in order to replace currently used devices which were either destructive or could be used only for a limited range of moisture content.

2. Experimental

2.1 Evaluation of dry rubber content of cup lump

This research developed a predictive model for the DRC of cup lump using NIR-HSI system. The result led to a development of a measuring system which could accurately evaluate the DRC in the cup lump which would help improve the quality control in the cup lump merchandizing.

The cup lump rubber was prepared from fresh latex collected in eastern Thailand in October 2017. The cup lump rubber was subjected to NIR-HSI scanning (Figure 1) Specim, Spectral Imaging Ltd., Finland: Three 20 W light sources shone the light on to the surface of the cup lump rubber placed on a moving translation stage at a speed of 10 mm/s. The calibration model for the DRC prediction was built by the least square support vector machine (LS-SVM).

Figure 1. (a) A cup lump rubber in scanned position by hyperspectral imaging system and (b) acquisition of the Para rubber sheet using the NIR-HSI system.
2.2 Classification of adulterated Para rubber sheet
The classification model was created using NIR-HSI system to separate adulterated Para rubber sheet. The result provided a means for quality control as the adulterated Para rubber sheet could not be visually differentiated from the normal sheet. The transflectance mode was achieved by placing the sheet on the translation stage and a spectralon with high reflective characteristic was placed underneath the sheet during the scanning to enable double light path through the sheet providing higher reflection back to the spectrograph. Partial least squares-discriminant analysis (PLS-DA) was performed to develop a classifying model with a class variable consisting of two levels as a response.

2.3 Development of NIR portable system for quality evaluation of Para rubber timber
This research developed a moisture content determining system for sawn timber, which comprised a portable spectrometer and a controlling software. The calibration model was first created and validated and then used as part of codes in controlling Android application. The housing of the spectrometer was designed and made to provide a hand-held system for measuring the moisture content in the timber which was used with a smartphone. The outcome of this research provided a system which could non-destructively measure the moisture content in timber. The developed system could replace a currently used device which had some limitation in the application.

Six hundred Para rubber sawn timber samples consisting of 5 different grades and three groups of moisture content were collected from sawmill companies in Southern, Eastern and Northeast regions of Thailand in 2019. Each sample was scanned twice at four positions by a portable spectrometer (DLP NIRscan Nano, Texas Instruments, Dallas, Texas, USA) in a range 900 nm to 1700 nm at an interval of 3.51 nm. The predictive model was developed using Unscrambler V.9.8 (Camo, Oslo, Norway) assigning the moisture content as the dependent variable and the NIR absorbance values for the whole range of wavelength as an independent variables.

3. Results and discussion
3.1 Evaluation of dry rubber content of cup lump
The reflectance spectra obtained from the NIR-HSI system showed prominent peaks (Figure 2a). At around 1200 nm, the second overtone vibration partially corresponded with C-H bonding of polymer \(^1\). The developed models were used to predict the DRC of all samples in the prediction set and the results are shown in Table 1. The model developed from spectral data of NIR-HSI system yielded very good performance providing the residual predictive deviation of calibration (RPD) greater than 5.0, which was considered good for application of quality control \(^2\).

![Figure 2](image)

**Figure 2.** (a) Average reflectance spectra of the cup lump rubber for different levels of DRC and (b) the spectral data preprocessed with the second derivative of Para rubber sheet for transflectance mode.

3.2 Classification of adulterated Para rubber sheet
The second derivatives of the spectral data, which resolved peaks in the spectra, are displayed in Figure 2b for transflectance mode. Strong peak at 1200 nm was observed, which was assigned to C-H stretching
second overtones or part of isoprene \(3\). The model based on the transreflectance mode yielded good classification performance in terms of \(R^2\) of 0.88, standard error of prediction (0.88) and accuracy of 98.33\%. For the purpose of visualized classification, the HSI image was mapped into color with the class value on each pixel predicted by the best model and showed in Figure 3.

3.3 Development of NIR portable system for quality evaluation of Para rubber timber

The timber samples contained the moisture content in a range 2.59\% to 88.59\%. The PLSR model gave good performance in cross validation providing \(R_{cv}\) of 0.98 and root mean square error of cross validation of 5.48\%. The portable NIR spectrometer (Figure 4) was developed based on the predictive model and could be used to non-destructively and rapidly measure the moisture content in Para rubber timber with acceptable accuracy.

**Table 1. Predictive performance on DRC based on different preprocessing.**

| Preprocessing method          | \(R^2\) | RMSEP | RPD    |
|-------------------------------|---------|-------|--------|
| SNV                           | 0.99    | 0.90  | 12.10  |
| SNV+first derivative          | 0.99    | 0.74  | 14.69  |
| SNV+second derivative         | 0.99    | 0.64  | 16.83  |

![Figure 3](image)

**Figure 3.** Color-mapped images for (a) formic acid, and (b) sulfuric acid.

![Figure 4](image)

**Figure 4.** (a) Developed hand-held system for measuring the moisture content in timber and (b) android application.

4. Conclusions

The NIR has benefit of being fast and non-destructive technique. NIR limitation is light penetration depth into the material, which allows detection depth of about 5-10 mm from the sample surface. Our research group has applied NIR successfully in determination of DRC in rubber cup lump, classification of adulterated rubber sheet and development of a portable system for measuring moisture content in rubber timber.
However, the results could be implemented in development a commercial instrument for quality control in near future.

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