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Rapid non-destructive evaluation of moisture content and higher heating value of *Leucaena leucocephala* pellets using near infrared spectroscopy

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ABSTRACT

The MC (moisture content) and HHV (higher heating value) of *Leucaena leucocephala* pellets using NIR (near infrared) spectroscopy was investigated in this study. The MC of the pellets was adjusted by subjecting the samples to different relative humidity environments. The samples were scanned in diffuse reflection mode at wavenumbers of 12,500–4000 cm^{-1}. Partial least squares regression models correlating the MC and HHV with the NIR spectra were developed and validated by full cross validation. The model for MC and HHV provided coefficients of determination (R^2) of 0.995 and 0.964, a root mean square error of cross validation (RMSECV) of 0.187%wb and 79.2 J g^{-1}, bias of −0.0008%wb and 1.29 J g^{-1} and a RPD (ratio of prediction to deviation) of 13.9 and 5.30, respectively. The models had excellent accuracy. This rapid quality evaluation method may be used for trading of biomass pellets. An equation related MC and HHV was also developed.

1. Introduction

Recently, biomass fuels have garnered much interest because of the increase in global energy demand. Biomass energy is a renewable energy resource that can be obtained from agricultural waste or fast growing plants. *Leucaena leucocephala* is one type of fast growing tree that can be planted in all parts of Thailand and is an alternative to fossil fuels. The large area of growing *Leucaena leucocephala* in Thailand is in Lopburi Province in the central area of the country and the Nakhonrachasima Province in northeastern area. Although not large, the product of *Leucaena leucocephala* has been used as wood chips and pellets as biomass fuel to supply boilers in factories. Mainoo and Ulzen-Apiah [1] studied on energy characteristics of *Leucaena leucocephala*, *Gliricidia sepium* and *Senna siamea* at four years of age. The results indicated that *Leucaena leucocephala* had the highest heating value, and it was a better choice for their high burning quality. Sripongpakapun et al. [2] studied the wood productivity of 5 varieties/lines of *Leucaena leucocephala* (Cunningham, Tarramba, Peru, 5/7 and 4/14) three years after planting. The Tarramba variety showed the greatest height and stem diameter (1043 cm and 4.90 cm, respectively). Furthermore, they also reported that there were no significant differences in wood density, ash or calorific value of all 5 varieties/lines.

Generally, the biomass forms are intact raw material and pellets, and the form of pellets is interesting. Adapa et al. [3] noted that the biomass pellets were a main type of bioenergy, and densification into pellets could reduce the material waste and improve the ease of transportation and storage. Kaliyan and Morey [4] reported that the low density of biomass involved a large amount of handling, transportation and storage costs; it should be kept in baled, briquettes or pellet forms. In addition, Rhén et al. [5] reported that pellet density increased the char combustion time, and the type of woody raw material had a major influence on char combustion time.

In trading, moisture content and heating value are important parameters in the specification of biomass pellets to set a price. The higher heating value is an important parameter for planning and control of power plants using biomass fuel [6–8]. Moisture content of biomass has a positive effect on higher heating value and a negative effect on the combustion process. Water in biomass must to evaporate before the biomass will burn. This evaporation reduces the net energy released as useful heat. Drying can increase the heating value of biomass. In 2010, Mahapatra et al. [9] reported that

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Notation

C	carbon
f	final
FT	Fourier transform
H	hydrogen
i	initial
LV	PLS latent variable
m	mass, g
Mc	moisture content, % wet basis
R^2_{Max}	maximum coefficient of determination
N	nitrogen
NIR	near infrared
O	oxygen
PLS	partial least squares
R^2	coefficient of determination
RMSECV	root mean square error of cross validation
RPD	ratio of prediction to deviation
SD	standard deviation of the measured values
RMSEE	root mean square error of estimation
RH	relative humidity
wb	wet basis
X_i	measured value of sample *i*
$\overline{X}$	average of the measured values of all samples
Bias	average error of prediction
HHV	higher heating value

the higher heating value of *Sericea lespedeza* pellets increased with decreasing moisture content in a range of 18.0 MJ kg^{-1} at 7.26% m.c. to 16.5 MJ kg^{-1} at 15.6% m.c. Komilis et al. [10] investigated the effect of moisture content on the heating value of solid waste; they also reported that heating value increased with decreasing of moisture content.

Normally, the higher heating value of a biomass fuel can be determined by a bomb calorimeter [7]; however, the complicated process requires chemicals and a long period of time. The moisture content of biomass measurements by a traditional hot air oven method, though, is accurate but takes a longer amount of time by a more complicated method. Thus, it is necessary to develop a new technique that is rapid, accurate, chemical-free and simple. NIR (Near infrared) spectroscopy is a rapid method for evaluation the chemical, physical and thermal properties of agricultural products. It uses 2–3 min per sample and is environmentally friendly because no chemicals are used. NIR spectroscopy is widely used in the food and agricultural industries; however, researchers have attempted to use NIR spectroscopy for evaluating the characteristics of biofuels and biomass. Gillon et al. [11] studied the relationships between the initial moisture content and the spectral properties of foliage samples from eight species. The foliage species including *Quercus ilex* L. (QI, Fagaceae), *Q. coccifera* L. (QC), *Q. pubescens* Willd. (QP), *Cistus albidus* L. (CA, Cistaceae), *Juniperus oxycedrus* L. (JO, Cupressaceae), *Spartium junceum* L. (SJ, Leguminosae), *Arbutus unedo* L. (AU, Ericaceae), and *Erica arborea* L. (EA) were studied. The calibration results on the foliage moisture content in % dry-weight in all species combined, for each year or for both years, were also predictive. The coefficient of determination (R^2) was 0.920–0.950 and the SECV (standard error of cross validation) was 7.00%. In addition, NIR spectroscopy can be used to evaluate the thermal properties of biomass. Posom and Sirisomboon [12] developed the NIR spectroscopy models for the evaluation of moisture content of *Jatropha curcas* L. kernels and the higher heating value of its residue after oil extraction. The R^2 values were 0.969 and 0.860, the RMSEP (root mean square errors of prediction) were 4.00%wb and 360 J g^{-1}, the biases were −0.700%wb and −17.0 J g^{-1} and the RPD (ratios of prediction to deviation) were 5.70 and 2.60, respectively. Lestander et al. [13] utilized on-line NIR techniques for the moisture content prediction of sawdust in pelletization processing. The results were excellent, and the NIR calibration model provided an R^2 of 0.842, a bias of −0.484% and a RMSEP of 0.636. Fagan et al. [14] predicted the moisture, heating value, ash and carbon content of biomass (*Miscanthus x giganteus*) using NIR spectroscopy; the results showed that a RMSECV (root mean square error of cross validation) were 0.900% ($R^2 = 0.990$), 130 J g^{-1} ($R^2 = 0.990$), 0.420% ($R^2 = 0.580$), and 0.570% ($R^2 = 0.880$), respectively. Everard et al. [15] predicted the biomass heating values from dedicated Irish bioenergy crops, i.e., *Miscanthus* and two varieties of SRCW (Short Rotational Coppice Willow), using visible and NIR remote spectroscopy. The RMSECV and R^2 for *Miscanthus* and SRCW were 300 J g^{-1} and 280 J g^{-1} and 0.970 and 0.960, respectively. Gillespie et al. [16] predicted the moisture, carbon and ash contents and heating value of a diverse range of biomass including wood, *Miscanthus* and *herbaceous* energy grasses. The result showed the RMSECV and R^2 were 0.730% and 0.850, 2.74% and 0.780, 0.620% and 0.820, 240 J g^{-1} and 0.940, respectively. However, no researcher has used NIR spectroscopy to evaluate the moisture content and higher heating value of *Leucaena leucocephala* pellets, which is a promising biomass energy source. There is also no literature studying the application of NIR spectroscopy to the higher heating values of biomass pellets with varying moisture content. Thus, the aim of this work was to investigate the effect of moisture content on the higher heating value of *Leucaena leucocephala* pellets and evaluate the properties using near infrared spectroscopy calibration models developed by PLS (partial least squares) regression.

2. Materials and methods

2.1. Sample

Leucaena leucocephala variety "Tarramba" samples were collected from Nakhonrachasima Province, Thailand. It was consecutively chopped, dried, and ground. The ground wood was formed to 8-mm diameter pellets by a pelletization process. The pellet samples were subjected to a 7 RH (relative humidity) environment including 22.6, 32.7, 43.8, 57.5, 63.5, 75.3 and 84.3% RH by putting the samples into 7 plastic boxes that each contained a saturated aqueous solution of CH_3COOH, $MgCl_2$, K_2CO_3, NaBr, $NaNO_2$, NaCl and KCl, respectively, for 2 months. Furthermore, 6 samples (1 L per sample) were placed in one box.

2.2. *Near infrared scanning of the* Leucaena leucocephala *pellets*

A FT (Fourier transform)-NIR NIR) spectrometer (Bruker Ltd., Germany) was used for scanning. Each sample at the same volume of 200 ml in a quartz-sampling cup (87.0-mm diameter and 87.5-mm height) was scanned through the quartz window in a rotary diffuse reflectance mode at a wavenumber of 12,500–4000 cm^{-1} (800–2500 nm) with a resolution of 16 cm^{-1}. The scanning was completed 64 times per one average spectrum. Before each sample