Fuel 217 (2018) 650–655

Contents lists available at ScienceDirect

Fuel

journal homepage: www.elsevier.com/locate/fuel

Full Length Article

Near infrared spectroscopy for the discrimination between different residues of the wood processing industry in the pellet sector

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ARTICLE INFO

Keywords:
Traceability
Wood composite
Quality control
Variables selection
PLS-DA

ABSTRACT

The increasing concern regarding energy supply and the consequent rapid growth of the pellet market lead to the need to classify the product quality. To this aim, chemical-physical parameters and qualitative attributes are defined by the technical standards EN ISO 17,225 to classify the quality of biofuels, but, while the former can be determined by traditional chemical analysis, no methodologies have been set for the latter one. Hence, near-infrared spectroscopy was tested to obtain information about the origin and the source of the pellet, at the moment only declared by the producers and difficult to be achieved by conventional analysis. In fact, the great strength of the technique is based on the fact that biomass features could be read simultaneously with a rapid and cheap NIR measurement. Checking the presence of treated wood (e.g. residues from wood processing industry) especially in densified products, such as pellets and briquettes, is particular important since in several European countries, e.g. Italy, these materials are considered as waste. In this study more than a hundred samples of virgin and treated wood (residues from wood processing industries) were analysed by means of FT-NIR. Partial Least Square regression – Discriminant Analysis was used in order to classify samples between the two classes and different variables selection methods were tested in order to improve the classification performance of the models. The results obtained demonstrated that near infrared analysis coupled with multivariate analysis can be used in screening applications to classify virgin wood from glue-laminated wood and treated wood. In particular, the model for the discrimination of treated wood (except glue-laminated samples) from virgin wood performs 100% correct classification and the model for the discrimination between virgin wood and glue-laminated wood only has a 3.6% misclassification rate. The methodology can be considered as the first one able to provide information about the origin of the biomass in a rapid and cheap way.

1. Introduction

The use of wood-based pellets will open up new opportunities for the reduction of fossil fuels consumption and for facilitating the achievement of the climate and renewable energy goals set by the European Union. The main one is to increase the use of green-energy considering the ever-growing energy demand [1].

Among the different types of biomass, pellets are one of the most competitive because they represent a higher energy density, a reduced transportation and storage cost and they are durable [2,3]. Different materials can participate to their blends making it adaptable to different location and feedstock [2–4]. The pelletization process creates a product with regular cylindrical shape and low moisture content, making possible its use not only in heat and power plants, but also for automatic feeding in small-scale applications [3]. Moreover, pellets ensure a clean burning and a reduction in ashes and emission produced [2].

For all these reasons, pellets are becoming increasingly used in different countries, especially in Europe where the market is increasing rapidly. Italy is the third largest European producer (0.77 million tonnes), but the domestic supply side doesn't cover the demand: its consumption in 2009 was 1.1 million tonnes [5].

In the past, pellets were mainly made up by pure wood (sawdust, chips or shavings), but nowadays – especially in some countries – the supply of pure wood is no longer enough and other raw materials are used [6].

The variability of the material characteristics used for pellet production leads to the need to validate its quality in order to ensure the good quality of the product and make it competitive with the other fuels. For this purpose, CEN/TC 335 has developed EN ISO 17225-2 [7] technical standard that classifies pellets into three quality classes – A1, A2 and B – in accordance to specific limits of chemical and physical

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https://doi.org/10.1016/j.fuel.2018.01.008
Received 22 November 2017; Received in revised form 22 December 2017; Accepted 3 January 2018
Available online 12 January 2018
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parameters and the origin and source of the material.

In accordance to EN ISO 17225-2, pellet B could be made up by by-products and residues from wood processing industry, but the Italian national legislation considers these residues as waste. Currently, laboratory analysis cannot provide information about qualitative attributes, at least not within a short time and at a low cost. In fact, they are expensive and time consuming, complex and require skilled operators, not matching the real necessities of the market that needs the results as fast as possible in order to make its decision and ensure the biomass quality. Moreover, considering the economic returns of the sector, regular laboratory analyses are too costly and a new and efficient method of biomass control is necessary.

Fourier Transform Near Infrared (FT-NIR) Spectroscopy is a valid alternative returning results in short time and allowing a total control of the biomass if applied in-line. It is already widely used in different sectors (food, pharmaceutical and agricultural industries) to predict qualitative and quantitative attributes [8]. In recent year, near infrared (NIR) spectroscopy coupled with chemometric techniques has been applied also to the wood sector to investigate the identity of the biomass and predict its parameters [9–15].

Several works have used NIR analysis to predict wood parameters, but to our knowledge no studies have been carried out for the discrimination between by-products and residues from wood processing industry and virgin wood in the pellet sector.

This study aims at developing a non-destructive, cheap and fast analytical methodology based on FT-NIR spectroscopy for discriminating virgin wood from treated wood, in particular glue-laminated wood, plywood, OSB and chipboard. For this purpose, more than a hundred samples have been collected and analysed by means of NIR spectroscopy. Principal Component Analysis (PCA) was used as an unsupervised technique to explore the spectral information about biomass origin and source. Partial least squares regression-Discriminant Analysis (PLS-DA) was applied in order to search for separation between groups of observations and classify them in accordance to their spectral differences. To evaluate the classification performance, the models were validated by independent test samples. Different variable selection methods were used in order to improve the classification performance of the models and as a tool for detecting the spectral variables of the NIR region more important for discrimination purposes.

2. Materials and methods

2.1. Material selection

Sets of wood samples collected in different sawmills and considered representative of the national scene was used to carry out this study. A total of 106 samples of virgin wood and residues from wood processing industry (hereafter referred as treated wood) were collected, the summary of wood characteristics is reported in Table 1.

Regarding virgin wood samples, whole pieces of wood such as tree log disks, wood slices, beams or boards of the three most common European species for the energy pellet sector, i.e. fir, pine and beech have been collected. Treated wood, i.e. glue-laminated wood, plywood, oriented strand board (OSB) and chipboard, were collected like beams or boards.

Table 1
Dataset of treated and virgin samples.

	Number
Virgin fir	13
Virgin pine	10
Virgin beech	16
Glue laminated hardwood (ash, sessile oak)	10
Glue laminated softwood (pine)	16
Plywood	14
Oriented Strand Board (OSB)	16
Chipboard	11
Total	106

First of all, the wood slices were dried at 45 °C for 24 h in order to limit the moisture content variability among samples. Subsequently, they were reduced to smaller pieces and ground to a particle size of below < 1 mm using a cutting mill (mod. SM 2000, RETSCH). Ground samples were stored in plastic containers at room temperature until measurement by the near infrared instrument.

2.2. Near-infrared spectroscopy

The set of wood samples were analysed by means of a FT-NIR spectrophotometer (mod. Nicolet iS 10, THERMO) equipped with a diffuse reflectance accessory (mod. Smart NIR UpDRIFT, THERMO). The analyses were performed in the spectral range from 10,000 to 4000 cm^{-1}; the spectral resolution was 4 cm^{-1} and each spectrum was an average of 64 successive scans.

A blank spectrum was acquired every hour in order to minimize the signals associated to the instrument or environment and not to the sample. The ground wood of each sample was recorded in triplicate and at room temperature (18–20 °C).

2.3. Data processing and multivariate data analysis

The PCA was used as an exploratory method in order to detect for outliers and to observe clustering or separation trends in the samples on the basis of chemical differences. The PCA loading plots were investigated to identify the compounds responsible for the discrimination between treated and virgin wood. Before multivariate data-analysis, the spectral database was subjected to different pre-treatments, i.e. SNV, first and second derivative (Savitzky-Golay [16] with 13 or 21 smoothing points and 2nd order polynomial).

Successively, PLS-DA [17,18] was used. This well-known supervised statistical technique can be applied when some a priori information about the sample is available and it assesses the relationship between a *y*-value – dependent variable – and the spectral data matrix, maximizing the covariation between them. The dataset was divided into calibration set and test set using the Kennard-Stone algorithm [19]. The PLS-DA models were cross-validated using Venetian blind-cross validation (5 segments) and an external test set was used to validate the classification models.

As the *y*-vector is a dummy vector indicating if the sample belongs to a class or not (i.e. 1 for treated wood, and 0 for virgin wood), the *y*-predicted will also have values in the range from 0 to 1, so a classification rule must be applied in order to include a sample in a classification class rather than in another one. The Receiver operating characteristic (ROC) curve is a widely used statistical tool that asses the ability of a classification model, defying a threshold limit on the *y* variable when a binary classification system is used to drive the decision.

The accuracy of the classification model could be determined with sensitivity and specificity values. Sensitivity or True-Positive Rate (TPR) is a statistical measure of how well the classification model is able to recognize a sample belonging to a given class. Specificity or True-Negative Rate (TNR) measures the ability of the model to reject all samples not belonging to that given class. The misclassification rate was also taken into account for evaluating the model performance since it is the ratio of false positives and false negatives to the total number of samples.

As for regression models, variable selection may also be applied to classification techniques in order to enhance the classification performance of the model and/or to simplify it by reducing the number of variables. Different methods for variables selection were applied, in particular regression vector (B), Variables Important in Projections (VIP) [17,20], Selectivity Ratio (SR) [21], interval Partial Least-Squares

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