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Soft Independent Modelling of Class Analogy applied to infrared spectroscopy for rapid discrimination between hardwood and softwood

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ABSTRACT

European policy promotes renewable energies and sets specific targets. Solid biofuels can play a significant role and the quality is an important aspect to be checked. Quality parameters such as origin and source are also required by specific biofuel standards. Therefore it could be useful to develop a rapid and cheap tool to distinguish between hardwoods and softwoods especially in unstructured, milled or densified wood in order to check the compliance of producer's declarations. Compared to other analytical methods, infrared spectroscopy is fast, non-destructive and low cost. In this study Fourier transform infrared (FTIR) spectroscopy coupled with Soft Independent Modelling of Class Analogy (SIMCA) has been evaluated as a method for discrimination purpose. A large dataset of 110 wood samples belonging to 12 species were analysed. In addition 4 blends were also analysed to test the discrimination performance of the tool. FTIR-SIMCA has correctly classified 93% of hardwood samples and 100% of softwood samples at high significance level. Furthermore, 100% of tested blends were associated with no class, showing a discrimination ability of the technique in recognizing blends from pure material. This method could be useful to verify the compliance of producer declarations about wood origin and source.
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1. Introduction

Currently one of the main priorities of European policy is the promotion of renewable energies implemented by different European countries with specific sustainability targets in particular for greenhouse gases reduction. In order to fulfill these goals solid biomass can play a significant role, especially densified biofuels such as wood pellet [1]. The market of this product is growing especially in Europe with an increase of over 40% from 2009 to 2013. In this period the consumption was more than 13 million tons, expecting for 2020 in EU-27 a forecast of 20–50 million tons and an import volume in the range of 15–20 million tons [2]. The high energy density and the dimensions of pellet make more easy and cheaper the logistics in comparison to other solid biomass, promoting its international trade. Despite the growth of pellet market there was not an equivalent development of quality control techniques. This control is very important because influencing combustion behaviour and resulting emissions [3]. The quality is defined by technical standards indicating parameters and specific limits. These parameters and their correlations have been studied by the authors [4,5]. In addition to chemical-physical parameters the standards set also quality attributes such as origin and source (EN 14961-2 [6], recently superseded by EN ISO 17225-2 [7] and ISO 17225-1 [8]), difficult to check by conventional chemistry or microscopy techniques because time- and money-consuming.

In more detail, according to Clause 6 of ISO 17225-1 and related Table 1 several information about the materials employed for biofuel production should be stated. This information mainly regards biomass origin (woody, herbaceous, fruit, aquatic, blends and mixtures) and source (virgin biomass, byproduct, industry residues). As consequence, hardwood and softwood material employed for biofuel production should be stated and, if appropriate, also information regarding species.

Therefore it could be useful to develop a rapid and cheap tool to distinguish hardwoods and softwoods especially in unstructured, milled or densified wood employed for pellet production, in order to check the compliance of producer's declarations. To this aim infrared spectroscopy could be employed.

Infrared spectroscopy is a fast, non-destructive and low cost

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Table 1
Dataset divided into training set and test set.

Hardwood	Training set (n = 60)	Test set (n = 15)	Softwood	Training set (n = 22)	Test set (n = 13)
Sessile oak (*Quercus petraea*)	12	3	Larch (*Larix decidua*)	2	1
Beech (*Fagus sylvatica*)	12	2	Pine (*Pinus* spp.)	9	7
Ash (*Fraxinus excelsior*)	8	1	Fir (*Abies* spp.)	11	5
Sycamore maple (*Acer pseudoplatanus*)	5	2			
Sugar maple (*Acer saccharum*)	5	1			
Wild cherry (*Prunus avium*)	6	1			
Common walnut (*Juglans Regia*)	4	2			
Hickory (*Carya cordiformis*)	5	2			
Sweet chestnut (*Castanea sativa*)	3	1			

technique which could be suitable to assess the standard quality parameters of woody biomass, directly in production line to support production process monitoring and tuning. It could be also a useful method to verify the compliance of producer declarations about wood origin and source as defined and requested by fuel quality assurance standards for solid biofuel production chain [9] and by wood pellet specification standard [10].

In literature there are several works that use infrared spectroscopy for wood analysis with classification purposes. Fourier transform Raman and Fourier transform infrared (FTIR) spectroscopies have successfully been used by Evans to discriminate between six softwood and six hardwood [11].

In other studies FTIR measurements were carried out in order to recognize different types of lignin [12]. Other authors [13] used FTIR spectroscopy and X-ray diffraction to evidence the differences which appear in structure of components between hardwood and softwood. A research was carried out by coupling FTIR spectroscopy with thermogravimetric analysis, for evaluating the cellulose and hemicelluloses to lignin ratios in wooden pieces of different species [14].

Several works have also been carried out using near and mid-infrared spectroscopy coupled with chemometric tools to perform classification [15,16] and quantitative analyses of the biomass [17,18].

To our knowledge, chemometrics with FTIR spectroscopy have been already used to discriminate hardwood and softwood, but the application is limited by the small number of samples. Furthermore few paper of this application can be found for solid biofuel sector.

Lestander et al. [19,20] have employed Near Infrared Spectroscopy (NIRS) in a pelletizing process in order to monitor on-line parameters among which the composition of the wood blends, i.e. softwood (spruce, pine) and hardwood (birch). In another paper [21] were able to identify ten different hardwood species by means of NIRS coupled with Principal Component Analysis (PCA). Yang et al. [22] have used NIRS with Partial Least Square (PLS) - discrimination analysis for identifying softwood and hardwood samples. Gulotta et al. [23] have successfully discriminated among species belonging to softwoods and hardwoods by using NIRS and PCA in conservation of cultural heritage sector. Chen et al. [24] have used multivariate analysis, i.e. PCA and Hierarchical Class Analysis, to discriminate between softwoods and hardwoods by their infrared spectral data. Bjarnestad and Olof [25] have developed a procedure based on photoacoustic FTIR and PLS able to recognize hardwood and softwood sample in chemical pulps.

In a recent paper of the authors [26] a FTIR-PLS model was developed to predict the amount of hardwood or softwood in ground wood.

In chemometrics two different approaches are possible for object classification: supervised or unsupervised techniques.

Supervised pattern recognition analyses find multivariate classification rules by using objects with known classes (training set), contrary to the exploratory analysis (or unsupervised techniques) such as Principal Component Analysis (PCA) [27] which do not require a-priori information at all. One of the most used is the Soft Independent Modelling of Class Analogy (SIMCA) introduced by Wold in 1976 [28]. SIMCA performs a PCA for each class and builds a multidimensional space around them using the principal components (PCs) calculated. Then the classification of unknown samples is made evaluating the distance between sample and models; if lower than the critical distance calculated for a specific class at suitable significance levels, then the sample belongs to that class. As class modelling technique SIMCA can also associate samples to more than one class or with no class.

SIMCA is becoming a technique more and more used for quality control of wood [29,30] and it could have a great potential in the field of biomass traceability, too.

This statistical method has been already used for the discrimination between hardwood and softwood employing a method based on thermogravimetric analysis [31], but no studies have been carried out using infrared spectroscopy.

The aim of this study is the discrimination between softwood and hardwood samples by means of FTIR and SIMCA computation. A large number of wood samples belonging to 12 species was analysed by the Biomass Lab of Polytechnic University of Marche in order to build and validate the classification technique. In addition, some blends were also analysed to test the discrimination performance of the tool.

Considering the high number of biomass samples analysed in this study the technique is very reliable and the classification very efficient, none of the previous methodologies has reached a stage of development that allows its use up to this point.

2. Materials and methods

2.1. Sample collection and preparation

A total of 110 samples, divided in 75 hardwoods of nine different species and 35 softwoods of three species, have been collected for subsequently analyses. Considering the aim of the study, only wood species common in the energy pellet sector were selected.

In order to have samples with known origin and non chemically treated, only whole pieces of wood like beams or boards and debarked tree log disks wood slices were taken.

After a reduction in smaller pieces, all the samples were ground by means of a cutting mill (mod. SM 2000, RETSCH) and were sieved under 0.25 mm particle size to collect the samples subsequently analysed in mid-infrared.

The total dataset of samples (n = 110) were divided into two sets: 82 samples (74.5%) were used as training set for multivariate analysis and model building while the remaining 28 samples (25.5%) were used as test set for validation purposes (Table 1).

The test set was chosen randomly in order to have more realistic

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