FTIR spectroscopy for determination of the raw materials used in wood pellet production

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1 # FTIR SPECTROSCOPY FOR DETERMINATION OF THE RAW MATERIALS

2 # USED IN WOOD PELLET PRODUCTION

3 Giuseppe Toscano[1, *], Vittorio Maceratesi[1], Elena Leoni[1], Pierluigi Stipa[2], Emiliano Laudadio[2]

4 Simona Sabbatini[2]

5 [1] Department of Agricultural, Food and Environmental Sciences - D3A, Marche Polytechnic University, Via

6 Brecce Bianche, I- 60131 Ancona, Italy;

7 [2] Department of Science and Engineering of Materials, Environment and Urban Planning - SIMAU, Marche

8 polytechnic University, Via Brecce Bianche 12, I- 60131 Ancona, Italy.

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Abstract

13 The research investigates the possibility of distinguishing pellet wood <u>fuels</u> using infrared spectroscopy. It has been

14 applied Attenuated Total Reflectance - Fourier Transform InfraRed (ATR-FTIR) spectroscopy, deemed an analysis

15 technique appropriate for detecting the chemical composition of materials, to determine woody components make up

16 a wood pellet. Considering the large quantities of this solid biofuel on the market, it is necessary to guarantee the safety

17 and traceability aspects of the product, as its origin and source, in addition to the quality parameters indicated by the

18 ISO 17225-2 standards. In this study, a total of 98 samples of the most common wood used for European pellet

19 production, have been selected and collected. This set of samples include also materials coming from the wood industry,

20 as residues of the chemical process. Through clustering analysis Principal component Analysis (PCA) and Hierarchical

21 Cluster Analysis (HCA) techniques applied to spectroscopic analyzer data, it has been seen that ATR-FTIR results provide

22 information on wood pellet typology, hardwood and softwood, and pellet made by woody materials containing high

23 quantity of chemical products like glues.

24

1. Introduction

26

The European Union has recently proposed a new and stricter package of proposals fit for reducing net greenhouse gas emissions by at least 55% by 2030 and aspires to world leadership in the fight against climate change. This recent decision has also produced effects on the new targets for the share of renewable energy established by the Renewable Energy Directive II, moving them from 32 to 40% by 2030 [1]. To fulfil the EU's climate and energy goals, the heating, and cooling sector must sharply reduce its energy consumption and cut its use of fossil fuels [2]. Currently, this energy sector contributes over 70% of energy consumption [3]. Half of the heat produced from biomass is consumed by the residential and service sector (school, hospitals, hotels…) and this is also evident from the great number of small and medium installations producing bioheat in Europe [3]. The contribution of solid biomass to the achievement of EU objectives can be significant, especially from wood pellets whose production [4] has increased from about 12 million metric tons in 2008 to 56 million metric tons in 2018. More than 27 million metric tons of wood pellets were consumed in Europe in 2018, more than 45% of them for industrial purposes [5] mainly to produce heat for heating or process heat [3]. The global pellet market is expanding and by 2050, is likely to exceed 60 Mt yr^{-1} [6]. Primarily produced with the residues of the wood processing chain, wood pellets are regulated by ISO 17225-2 and which currently identifies three quality classes for domestic heating, A1, A2, B, and three industrial-type classes (I1, I2, and I3). In Italy, class I4 has recently been defined by means of UNI / TS 11765: 2019. It is important to point out how the regulations have helped to support the growth of this fuel in the past years by ensuring the achievement of high-quality levels also through certification schemes and correct environmental performance [7]. The quality classes defined in the technical specifications establish the maximum and minimum levels of physico-chemical parameters and the origin and source of the material based on hierarchical classification made in ISO 17225-1. The latter, in addition to implicitly conditioning the quality level of the pellet, differentiating the type of wood (eg wood from whole trees with our without roots, wood with or without bark, wood from gardens vineyard, and so on …) also signals the presence of matrices not allowed by the legislation of some countries. For example, in Italy the chemically treated wood by-products, residues, fibers and wood constituents (1.2.2), referring to the residues of wood processing residues, namely glue-laminated wood, oriented strand board (OSB), plywood, and chipboard [8], and used wood (1.3) are not allowed as fuel biomass for powering domestic systems. Finally, the knowledge of the origin and source of the solid biomass contributes on evaluating the traceability and sustainability of the pellet or in general of solid biofuel. Currently, this information is always declared by the pellet manufacturer and cannot be detected through a simple visual evaluation as the starting raw material is deconstructed through grinding during the pelletizing process, thus losing the macroscopic characteristics of the original

55 product. Traditional methods of recognizing woody materials are based on costly and time-consuming analysis

56 techniques, such as scanning electronic microscopy (SEM). However, methods of analysis using infrared spectroscopy

57 have been proposed for some years [8, 9 and 10]. Particularly, NIR spectroscopy has been introduced as a promising

58 method for the prediction of wood properties. This technique is a sample task with no chemical destruction and easy

59 preparation of samples [11-13] Many studies have already suggested the use of time-saving analytical models based on

60 NIR spectroscopy. This allows moisture, cellulose, lignin, and extractive contents in wood or biomass to be measured

61 automatically [14-15] It was demonstrated that the FT-NIR is sensitive enough to detect minor differences in the

62 chemical composition in trees of the same species, but growing in various locations [16] FT-NIR spectroscopy can be

63 considered valid pre-screening tool for analyzing large volumes of biomass, overcoming the sampling problem and

64 allowing a total quality control if used for industrial in-line applications [17]. Near infrared energy is strongly absorbed

65 by water molecules and corresponding OH bonds. NIR spectra of biomaterials may be dominated therefore by "water

66 peaks" at ~1450 nm and ~1890 nm, which is especially noticeable in samples with higher moisture contents. In that case

67 the spectrum contains only a few strong, broad absorption bands, masking peaks due to other wood constituents [18].

68 Band assignments can be of particular importance in quality control, because control charting methods can be utilized

69 to determine when the process is out of control based on shifts in functional groups or principal components that

70 represent key functional groups [19-20]. Any shift in the functional groups could be an indication of a change in

71 feedstock quality. Mid Infrared Spectroscopy (MIR-FTIR) is now receiving increased attention because of the availability

72 of the ATR-diamond reflectance method that has made it possible for the spectra of solid samples to be acquired in

73 seconds. In addition, the peaks of FTIR spectra are more interpretable, resulting in easier qualitative analysis [21]. One

74 hurdle for using FTIR spectroscopy on solid wood samples could be the cellular orientation in wood and its effect on

75 reflectance, absorbance, and/or transmission of light [22]. Attenuated Total Reflectance - Fourier Transform InfraRed

76 (ATR-FTIR) spectroscopy is a valuable analytical tool, which let achieve relevant information on the chemical

77 composition of organic, inorganic and biological compounds, by analyzing the interaction between matter and the

78 electromagnetic radiation in the infrared range. This technique needs a very small amount of pure sample powder

79 (usually < 1 mg); it is based on the study of the evanescent wave generated by the incoming IR radiation at the

80 interface between the sample and a crystal with high refractive index. Unlike the other common techniques, FTIR-

81 ATR spectroscopy does not require laborious sample preparation steps and the interpretation of the analysis is

82 rather simple [23].

83   The results of these works seem to be so comforting that a technical specification has been drawn up in Italy - UNI/TS

84   11765:2019 (Solid biofuels - Guidelines for the determination of the quality by near infrared spectrometry) - which

85   allows the determination of some properties of solid biofuels by NIRS. However, the application of near infrared

86   spectroscopy still has room for improvement and requires the experience of specific applications. Therefore, the authors

87   found it useful to evaluate the possibility of using mid-infrared spectroscopic analysis to recognize and characterize the

88   woody raw material of pellets compliant with UNI EN ISO 17225-2. The research objectives concern the possibility of

89   distinguishing softwood from hardwood pellets and untreated and chemically treated wood pellets.

90

91   2. Materials and Methods

92   *2.1 Sample Collection and Preparation*

93   In this study, a total of 98 samples of hardwood and softwood of the most common European species in the energy

94   pellet sector, have been selected and collected. The wood samples were classified as following: 23 virgin softwood,

95   3 laminated softwood, 61 virgin hardwood, 3 laminated hardwood and 8 chemically treated blends (Table 1).

96   The material was first of all stabilized at 45 °C for 24 h; in this way, all of the samples have reached the same

97   moisture content of 6−7%. Subsequently, the samples were ground by means of a cutting mill (model SM 2000,

98   Retsch), sieved below 0.25 mm, stored in plastic containers, and analyzed in medium infrared wavelength.

99

100   **Table 1**. Dataset of virgin (V), laminated (L) and chemically treated wood samples

Softwood	Data set	Hardwood	Data set	Blends of chemically treated woods	Data set
Pine (*Pinus spp.*) (V)	11	Beech (*Fagus sylvatica*) (V)	10	Medium-density fibreboard (MDF)	2
Fir (*Abies spp.*) (V)	11	Valnuts (*Luglans regia*) (V)	10	Oriented Strand Board (OSB)	4
Larch (*Larix decidua*) (V)	1	Oak (*Quercus petraea*) (V)	10	Chipboard	2
Pine (*Pinus spp.*) (L)	3	Chestnut (*Castanea sativa*) (V)	10		
		Ash (*Fraxinus excelsior*) (V)	10		
		Maple (*Acer pseudoplatanus*) (V)	10		
		Turkey oak (*Quercus cerris*) (V)	1		
		Ash (*Fraxinus excelsior*) (L)	3		

101

102   *2.2 ATR-FTIR analysis*

103 All samples were analyzed by using a PerkinElmer Spectrum GX1 spectrometer (PerkinElmer, Inc, Waltham, MA,

104 USA), equipped with the ATR accessory consisting of a crystal of ZnSe (FTIR-ATR) (Pike Technologies Miracle Universal

105 ATR) for the analysis of solid samples in reflectance mode. On each wood sample, ten IR spectra were acquired (64

106 scans, spectral resolution 4 cm^{-1}) in reflectance mode in the 4000-500 cm^{-1} spectral range and the average spectrum

107 was reported. To perform the analysis, a small amount of sample (0.2–0.3 g) was pressed onto the crystal using a

108 high-pressure tower. A background spectrum was collected before each sample acquisition to exclude the signals not

109 associated with the sample (e.g., environment).

110 All raw IR spectra were converted in absorbance, interpolated in the 1800-900 cm^{-1} spectral interval, baseline

111 corrected, and vector normalized in the same range (Spectrum 10.6.1 software, Perkin-Elmer).

112 The above-described spectral parameters of each sample were submitted to Hierarchical Component Analysis (HCA)

113 and Principal Component Analysis (PCA), to evaluate similarities among samples.

114

115 2.3 *Clustering Analysis*

116 Principal component Analysis (PCA) is an algorithm used to extract relevant information from confusing data sets

117 highlighting similarities and differences. The original data set of variables is reduced to smaller number of orthogonal

118 variables called the principal components (PCs). In the analysis of FTIR spectra by PCA the scores report the contribution

119 of each spectrum related to the selected principal component and the corresponding loadings show which wavenumber

120 is responsible for such contribution. In this work the PCA was carried out on all spectra of wood samples extracted by

121 Hierarchical Cluster Analysis (HCA) in order to discriminate between the two classes of virgin wood (hardwood and

122 softwood) and between the virgin samples and the corresponding chemically treated ones.

123 Before PCA computation, the spectral database was subjected to different pretreatments, such as Hierarchical Cluster

124 Analysis (HCA), Standard Normal Variate (SNV) to reduce the scatter effect and spectral average of replicates in the

125 same spectral range (1100-1800 cm^{-1}) because it contained high spectral information. PCA and all pretreatments were

126 performed by means Pirouette 4.5.

127 The various clusters generated by the chemometric analysis were used as a reference model to test unknown samples.

128 The goal was to place the unknown samples in their respective clusters. Woods with a marked chemical treatment

129 would have to be placed in the treated woods clusters, while untreated samples would have to be placed in the virgin

130 woods clusters. To validate the PCA model, six new wood samples were added to those of the full data and subjected

131 to chemometric analysis again. New samples were the following: n°1 oriented strand board (OSB), n°1 chipboard, n°1

132 medium density fiberboard, n°1 laminated pine, n°1 virgin Turkey oak, n°1 virgin ash.

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135 # 3. RESULTS AND DISCUSSION

136

137 ## *3.1.   ATR-FTIR spectral analysis*

138

139 The average spectra of pure softwood and hardwood samples together with the corresponding laminated and the

140 chemically treated ones are compared in Figure 1. Spectral differences between hardwood and softwood are evident.

141 The discriminating elements between the two groups are mainly due to lignin, cellulose and hemicellulose, which are

142 different in chemical composition. The most relevant wavenumbers are reported in Table 2, according to literature [24].

143 There are no significant spectral differences between pure woods and the corresponding laminated samples. Due to the

144 high quantity of glue, however, some spectral differences are evident in the chemically treated samples (MDF, OSB and

145 chipboard) in the range between 1450 and 1800 cm^{-1}. Table 2 shows the band allocations in the ATR-FTIR continuum of

146 wood together with the corresponding types of vibration.

147 **Table 2.** Infrared bands and related molecular bond assignments

| Band (cm^{-1})	Band assignments
805	Vibration of mannan in hemicellulose and CH out of plane bending in phenyl rings
825	CH out of plane bending in guaiacyl units
860	C–H out of plane in position 2, 5, and 6 of guaiacyl units
900	CH deformation of beta-glycosidic linkages in cellulose
945	O–H out of plane deformation in carboxylic acid
960	CH out of plane deformation in lignin
985	CO stretching in cellulose
1005	C–O stretch in cellulose
1025	C–O stretching in primary alcohols in cellulose
1055	C–O stretching of secondary alcohols
1105	C–O–C stretching in cellulose and hemicellulose
1155	C–O–C asymmetric stretching in cellulose and hemicellulose
1185	C–O stretching in cellulose
1225	OH vibration in guaiacyl ring, C–C, C–O, and C=O stretches in lignin
1265	C–O vibration in guaiacyl rings

1315	CH$_2$ wagging in crystalline cellulose
1335	CH of methyl groups in methoxy in amorphous cellulose
1360	C–H deformation in cellulose and hemicelluloses
1385	C–O stretching in cellulose and hemicellulose
1405	C=O in carboxylic groups in carboxylic acid, ester
1425	C–H asymmetric deformation in methoxyl, aromatic skeletal vibrations, lignin
1465	C–H asymmetric deformation in methoxyl for lignin, asymmetric in—CH$_3$ and CH$_2$ in pyran for hemicellulose
1510	C=C stretching of the aromatic ring, C=O bond vibrations in extractive compounds
1590	Skeletal vibrations from the C–C
1610	C=O stretching conjugated to the aromatic ring, and in carboxylic groups in lignin, carboxylic acid, ester compounds
1635	Absorbed O–H and conjugated C–O in polysaccharides
1655	Absorbed O–H and conjugated C–O in polysaccharides
1690	C=O vibration in carboxylic group in resin acid
1730	C=O carbonyls in ester groups and acetyl groups in xylan

All samples show a broadband to $\simeq$3300 cm^{-1} corresponding to O-H stretching vibration from water, cellulose, hemicellulose and lignin and two peaks at 2919 and 2858 cm^{-1} assigned to C-H vibrations of polysaccharides (glucomannans and arabinogalactan) and lignin [25]. In particular, lignin has a different composition in the two plant species: in softwood, is a polymer composed mostly of guaiacyl units, with a small amount of *p*-hydroxyphenyl and syringil units, while in hardwood is composed of syringil and guaiacyl units, with a small amount of *p*-hydroxyphenil units. Absorption bands referred to syringil units (1594, 1326 and 1234 cm^{-1}) are easily detected in hardwood spectra, while the characteristic guaiacyl unit absorption band at 1264 cm^{-1} is more evident in softwood. The bands at about 1740 (stretching of acetyl group) and 1240 cm^{-1} (stretching of C-O group) assigned to hemicelluloses, are more intense in hardwoods than softwoods, due to their different chemical composition. A shift of carbonyl spectral absorption to a lower wavenumber could be primarily due to the higher concentration of lignin in softwood than hardwood [25]. Furthermore, softwood shows a characteristic peak at 807 cm^{-1} (glucomann absorption) which is not evident in hardwood spectra [27]. The band at 1509 cm^{-1} is referred to stretching C=C of aromatic ring and is well evident both in softwood and hardwood samples [28]. As mentioned above, the spectra of laminated softwood and hardwood does not show meaningful spectral differences compared to the corresponding virgin samples. On the contrary, the wood samples treated with a high quantity of glue (MDF, OSB and chipboard) show evident spectral differences especially in the region between 1500 and 1800 cm^{-1}, due to the presence of higher concentration of glue.

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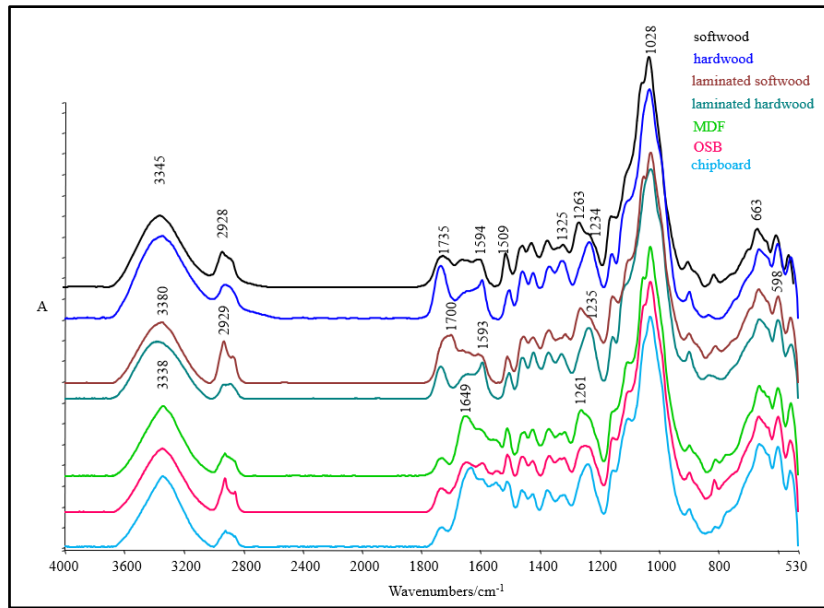
Figure 1. Infrared average spectra of hardwood, softwood, laminated hardwood, laminated softwood and chemically treated woods: MDF, OSB, chipboard in the spectral range 4000-530 cm^{-1}

170

3.2 Chemometrics analysis

PCA was performed on the spectral database to investigate the possibility of discriminating both between virgin hardwood and softwood (Figure 2) and between virgin species and the corresponding chemically treated ones with small (laminated softwood and hardwood) or very significant quantities of glue (MDF, OSB and chipboard) (Figure 3).
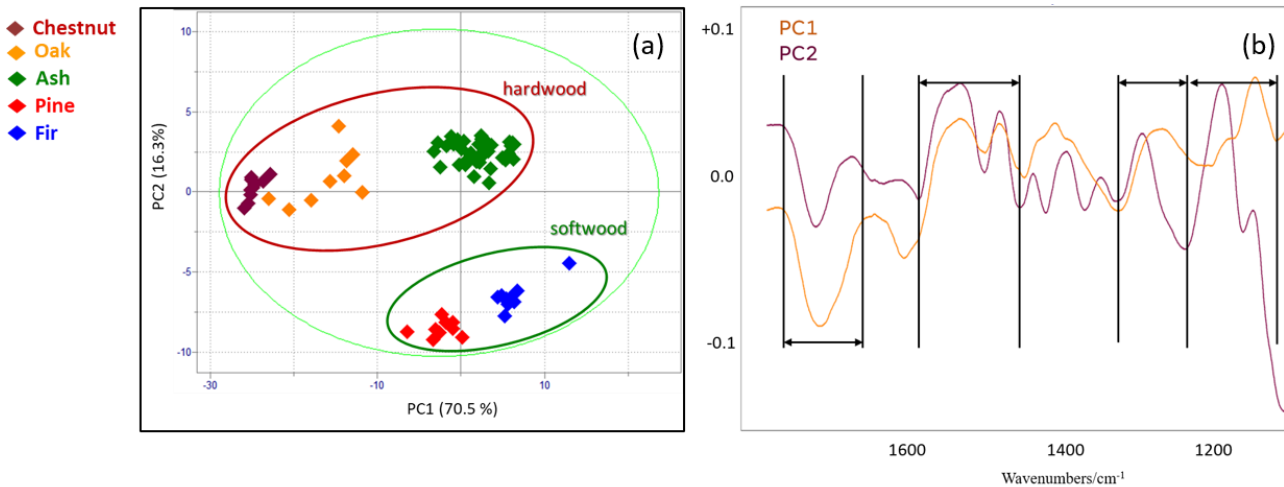
Figure 2. (a) Score plots of the two first PCs about hardwood and softwood; (b) Loading spectra for principal components PC1 and PC2

178   The spectra from all the wood samples analyzed by ATR-FTIR and divided into clusters through the HCA were subjected

179   to chemometric analysis. The spectral region between 1100-1800 cm^{-1} was selected. Accounting for 86.8% of the total

180   initial variability, the two first principal components (PCs) were considered for further elaborations. The scores plot of

181   the first two PCs shows that hardwood and softwood samples are well-separated along PC2; softwoods have negative

182   PC2 scores, whereas hardwoods have positive PC2 scores. It is also possible to observe a separation between the

183   different species along PC1. In softwood fir woods have positive PC1 scores while pine woods have negative PC1 scores

184   (Figure 2a). The methodology guaranteed an excellent separation between the different species both along PC1 and

185   PC2, even within the same group, and could be effective in laying the foundations for the development of a laboratory

186   method for an early assessment of the type of biomass used in the production of pellets. For this reason, could be used

187   in any quality control application when the same hardwood and softwood species are used. The loading spectra of PC1

188   and PC2 showed that the PCA plots were strongly contributed by the ranges of 1680–1780 cm^{-1}, 1600–1450 cm^{-1} and

189   1350–1150 cm^{-1} (Figure 2b). In these wavenumber ranges, absorption caused by C=O stretching vibrations (ca. 1740 cm^{-1}

190   ), C=O stretching conjugated to the aromatic ring (ca. 1610 cm^{-1}), the breathing of the aromatic ring. (ca. 1510 cm^{-1})

191   and the stretching of the C-O group (ca. 1223 cm^{-1}) were reported. This measuring procedure might be helpful in

192   confirming that differences in the composition and structures of cellulose, hemicellulose and lignin are well recognized

193   between softwood and hardwood and that the vibrations of the chemical bonding from these chemical compositions

194   are possibly the key to the separation of softwood and hardwood PCs. The same method was applied to investigate the

195   separation between virgin and chemically treated woods. In this case, HCA classified the spectra of all samples in four

196   clusters, where separation between hardwood and softwood was still maintained. Samples with higher amount of glue

197   (chipboard, MDF and OSB) were isolated in a single cluster. The method was not able to separate the laminated woods

198   from the virgin ones for the low glue content (<0,5% w/w). So, this type of analysis considers virgin wood equal to

199   laminated wood. The results were confirmed by the PCA (Figure 3).
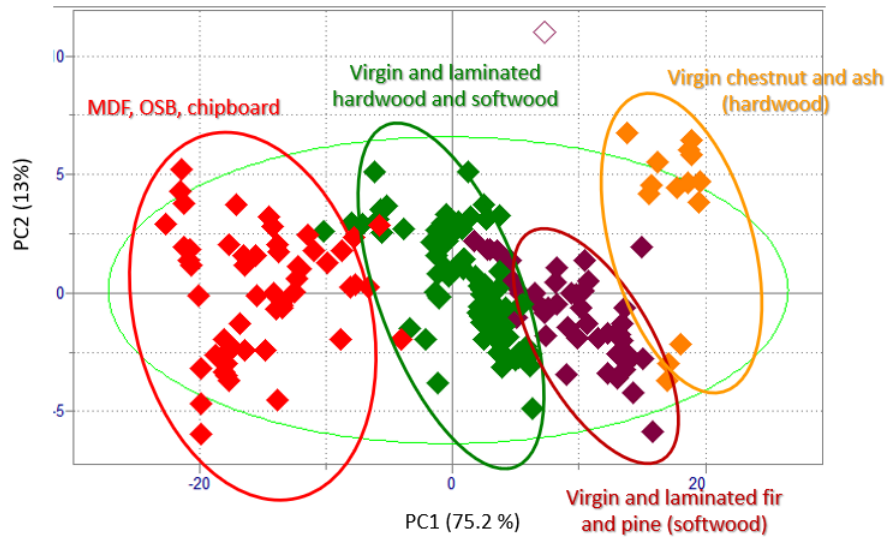
Figure 3. Score plots of the two first PCs about virgin and chemically treated woods

Figure 3 highlights a good separation along PC1, probably resulting from the presence of chemicals. PC1 scores are negative in chemically treated woods while they are positive in most virgin ones. Virgin and laminated woods are not well separated. This model could be used in any quality control application to distinguish samples with a high glue content. It allows you to evaluate the presence of materials unrelated to the woody nature, thus providing more details for their origin and provenance. It could also be used for the analysis of woods with preservatives or other chemical components. The six new wood samples, used to validate the PCA model, were analyzed in triplicate and subjected to the same pretreatments as above. As expected, using the PCA model developed from the reference samples, the spectra of the new wood samples were correctly entered into the corresponding clusters confirming the validity of the pattern. (Figure 4).

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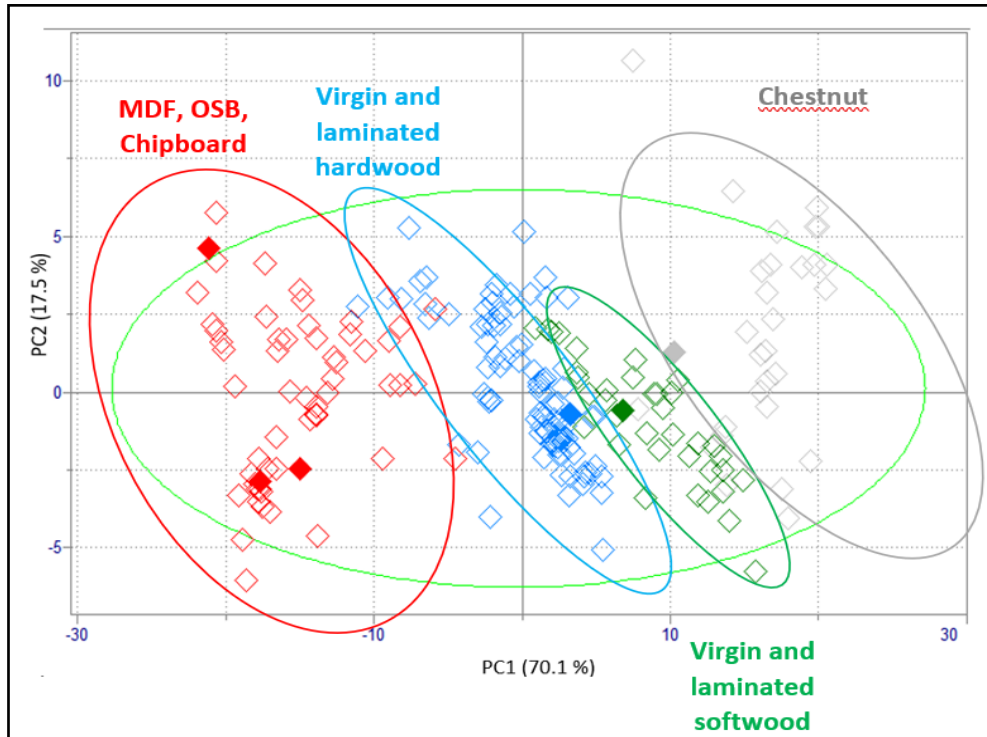
Figure 4. Score plots of the two first PCs about virgin wood and chemically treated wood. The colored symbols

represent the 6 new samples used for validation of the model and placed inside the different clusters (red: MDF, OSB

and chipboard; blue: virgin turkey oak; green: laminated pine ang; grey: virgin ash).

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4. CONCLUSIONS

The FTIR technique together with the development of suitable chemometric models provides useful information able

to differentiate the woody materials with which the pellet is made. Because IR spectra contain robust information about

chemical bonds, compositional information is not directly available from their results [29]; thus, chemometric

techniques, such as multivariate models, are necessary for spectra analysis. By reducing the large amount of spectral

data in several latent variables, the statistical methods/models could build a relationship between spectral features and

chemical components/bonds [13]. The analysis can be applied to any solid sample and does not require pre-treatment

except drying for samples with a high moisture content. This allows to evaluate the origin and the species, as required

228   by the specifications on solid biofuels, by means of a rapid and economical laboratory analytical method and potentially

229   implementable in a qualitative pre-screening system for wood pellets.

230   The application of FTIR can support commodity problems through the distinction between woody groups, such as

231   softwoods and hardwoods, with good prospects also for identifying specific wood species. Furthermore, the ability to

232   recognize wood materials subject to chemical treatment, such as residues of the wood industry, is of interest to assess

233   the compliance of the product with the law and increase the guarantee of compliance with environmental aspects

234   related to the use of wood pellets in combustion. However, it should be noted that this analysis technique fails to detect

235   the presence of low quantities of chemically treated wood, as in the case of laminated ones, whose physical and

236   chemical properties are potentially in line with the ISO 17225-2 class A1 specifications and A2. This aspect leads us to

237   reflect on the possibility of considering this specific wood raw material for producing pellets for domestic use.

238   Considering the prospects deriving from mid-infrared spectroscopy, it could be useful to strengthen the research by

239   expanding the wood materials casuistry of interest to the wood pellet sector, as mixtures consisting of different

240   essences. Thus, providing the basis for proposing a specific technique to supplement the existing one - UNI / TS 11765 -

241   which applies near infrared spectroscopy.

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