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CHARACTERISTICS OF THE HEAT TREATED WOOD PACKAGING MATERIALS ACCORDING TO INTERNATIONAL STANDARDS FOR PHYTOSANITARY MEASURES AND VERIFIABILITY OF HEAT TREATMENT

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ABSTRACT

We aimed to develop a control method to assess compliance with International Standards for Phytosanitary Measures No.15 (ISPM 15) heat treatment wood packaging materials by physico-chemical properties and chemo-metrical approach. *Larix leptolepis* (Siebold & Zucc.) (Gordon larch), *Pseudotsuga menziesii* (Douglas fir) and *Picea jezoensis* (spruce) as wood samples were used in this study. The ISPM 15 processing of wood did not alter the physico-chemical characteristics. By the ISPM 15 processing of wood, the core moisture content of timber was approximately 6%, regardless of the wood species, whereas that before heat treatment was 10-12%. Among the different parameters of wood, the moisture content can be classified by the PCA according to the ISPM 15 processing, which can be easily changed by mild heat treatment. Furthermore, the changes in chemical properties occurring after the ISPM 15 processing were clearly distinguished by using the ATR-PCA system.

KEYWORDS: ISPM 15, wood packaging materials, heat treatment, wood core moisture content, principal component analysis.

INTRODUCTION

In international trade, many goods are being packaged and transported across borders. Wood packaging materials are used in many cases for the packaging of goods. Therefore, they are inevitably transported between countries and countries by nature. At same time, many kinds of organisms, including wood decaying organisms, are attached to wood packaging materials and are likely to cross borders. Organisms that are attached to wood packaging materials and crossed the border can sometimes have a serious adverse effect on the ecosystem of that country (Brockerhoff et al. 2006, Haack et al. 2014, Zahid et al. 2008).

To reduce the biosecurity risk of wood packaging materials, the International Plant Protection Convention has developed an International Standards for Phytosanitary Measures No.15 (ISPM 15), which has been adopted by several countries. This standard specifies that if the width of the wood packaging material (WPM) is < 3 cm, the bark should be re-applied to reduce secondary damage. The wood packaging materials of width > 6 mm may be treated with heat or methyl bromide for fumigation. The heat treatment requires that the core of the timber be maintained for at least 30 minutes at 56°C. These regulatory guidelines are applied in more than 78 countries as of October 2013 (Zahid et al. 2008).

The wood packaging materials that are heat-treated according to the ISPM 15 will be marked with an easily identifiable mark. The National Plant Protection Organizations (NPPOs) are responsible for permitting the disinfection operator to use marks and for regularly inspecting and monitoring the disinfection procedures. However, the ISPM 15 mark occasionally can be found on displayed on wood packaging materials that do not satisfy or are not sufficiently processed. Therefore, there is a need for a diagnostic method that can verify heat-treated wood packaging material according to the ISPM 15. The diagnostics that can verify whether wood packaging is heat-treated according to the ISPM 15 are needed to prevent the expected technical and legal controversies between manufacturers, between manufacturers and countries, and between countries. To develop reliable diagnostics, one or more characteristics that change consistently between untreated and treated wood should be identified. Several studies have focused on detecting various enzymes and sugars in wood samples (Sagisaka 1972, Leonowicz et al. 1997a, 1997b), but only a few studies have analyzed enzymes and sugars in ISPM 15-treated wood.

Henin et al. (2008) used microwaves for the ISPM 15 treatment of wood. The wood was heated by microwaves for up to 7 min. It has been reported that some of the larvae of *Hylotrupes bajulus* survive when the core temperature of the wood reaches 50-55°C, but they die at higher temperatures. It has also been reported that the treatment of wood at 56°C generally does not result in complete inactivation of the enzyme (malate dehydrogenase), but might reduce its activity (Iline et al. 2014). Haack et al. (2014) conducted a study on the rate of infestation of ISPM 15-treated WPM entering the USA, and the types of insects found. Although the wood packaging material is marked with the ISPM 15 processing mark, the infestation of insects or fungi may be suspected. In this milieu, if there is a way to verify ISPM 15 processing, it can aid in decisively deciphering the cause of the problem.

To facilitate the use of eco-friendly wood packaging materials in international trade, there is a need for a reliable method to confirm the ISPM 15 treatment of wood.

Globally, many heat-treated timber manufacturers produce ISPM 15 wood packaging materials, there are about 700 companies registered in South Korea since January 2002. Due to the nature of wood packaging materials, heat treatment cannot be confirmed with the naked eye. Therefore, there is a possibility that untreated wood is distributed.

The confirmation of heat treatment of wood depends only on the disinfection mark (Fig. 1) of the manufacturer approved by the Agriculture, Forestry and Livestock Quarantine Headquarters in Korea. The use of fake-labeled wood packaging material produced by an unqualified company can lead to serious damages. There is a need for a reliable method to verify the heat treatment of wood packaging materials in order to prevent such damage.

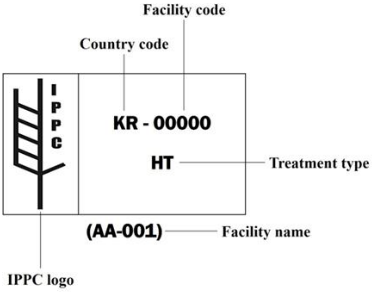


Fig. 1: Mark on heat-treated wood packaging materials according to the ISPM 15.

Kim et al. (2018) suggested the possibility of discriminating wood that is heat-treated at relatively low temperatures by spectroscopic means. The surface IR spectra of the heat-treated wood and control wood were measured. As it is difficult to distinguish heat treatment by IR spectra alone, the IR data were differentiated and subjected to PCA (principal component analysis) system. The results revealed that longer the heat treatment, the more clearly the PCA score plot is separated. Therefore, we presumed that this method can be used to determine whether the wood is heat-treated. In this study, we aimed to develop a control method to assess compliance with ISPM 15 heat treatment. The physico-chemical properties of the heat-treated wood according to ISPM 15 were evaluated. Furthermore, the spectroscopic properties of the surface of wood and the core of timber were compared.

MATERIAL AND METHODS

Materials

Three types of conifers, mainly used for wooden pallets, were kindly provided from heating wood producers (A Timber Co. Ltd, Gyeongsangbuk-do, Korea). Three species of softwood were used in this study: larch is from Korea, but Douglas fir and spruce was imported from the United States and Europe, respectively. The wood was cut into sample of 30 cm (length) × 8 cm (width) × 8 cm (thickness) pieces for heat treatment according to the ISPM 15. Tab. 1 shows the basic characteristics of three species wood samples.

Tab. 1: Characteristics of wood packaging materials treated in compliance with ISPM 15-heat treatment.

Name	Botanical (scientific) name	Country	Information
Larch	*Larix leptolepis* (Siebold & Zucc.) Gordon	Republic of Korea	- Green wood - Moisture content : 25%↑
Douglas fir	*Pseudotsuga menziesii*	USA	- Fungicide treatment Moisture content: about 25%
Spruce	*Picea jezoensis*	EU (unknown country)	- Kiln drying treatment - Moisture content: 15%↓

Heat treatment of wood packaging materials according to ISPM 15 and storage

The three kinds of wood were treated according to the ISPM 15 in wood disinfection equipment in A Timber Co. Ltd. The temperature in the wood disinfection facility were maintained at 70°C to ensure that the wood core can be treated at 56°C for more than 30 min. A probe temperature sensor (SDT25, SUMMIT, Korea) was inserted into the wood core to monitor the temperature of the wood core in real time. The heat-treated wood and the control wood sample were keep in incubator (HB-103L, Hanbaek Co., Korea) at moisture content of 50% and temperature 23 ± 1°C.

Measurement

The moisture content of the wood surface was measured randomly at 10 points using a wood moisture meter (HM-520, KETT, Japan) to determine the surface moisture content of the heat-treated wood and the control wood. The weight and volume of the heat-treated wood and the control wood were also measured every week to confirm the dimension changes of the wood sample. Ten microliters of water were dropped on the surface of wood sample using a contact angle analyzer (Phoenix 150, Surface Electro Optics, Korea) to measure the contact angle. This procedure was repeated 10 times. The water absorption time was measured by the time of the water droplets were completely absorbed into the wood. Two holes were drilled 4 cm deep from the wood surface to measure the core moisture content of the wood after the ISPM 15 treatment. The moisture content of the wood core was measured by inserting a probe-type moisture meter (MD 7820, Shenzhen Sanpo Instrument Co. Ltd., China) into the perforated area. For chemical analysis of the heat-treated wood according to the ISPM 15 and the control wood were chipped and milled through a Willy mill (MF 10 basic microfine grinder, IKA Werke GmbH & Co., Germany). Cold water extract (T 207 cm-08), hot water extract (T 207 cm-08), 1% NaOH extract (T 212 om-12), organic solvent extract (T 204 cm-07), and Klason lignin (T 222 om-11) were measured by TAPPI Standard method. The reducing sugar analysis of the wood samples was carried out in accordance with study of Miller (1959).

Three millilitres of cold water extractives of wood sample and six millilitres of DNS reagent were mixed. The mixture was incubated in a water bath at 100°C for 10 min. Subsequently, 3 mL of reaction mixture was placed in a UV cell and the absorbance was measured at 540 nm using a UV-Vis spectrophotometer (Optizen 3220 UV Bio, Mecasys, Korea).

Alkaline nitrobenzene oxidation was performed to evaluate the chemical structure of lignin (Eom et al. 1996, Takada et al. 2004). Tab. 2 shows the GC conditions to analyze the nitrobenzene oxidation of lignin.

Tab. 2: Gas chromatography operating conditions for analysis of alkaline nitrobenzene oxide.

Column	Rxi®-5ms (30 m × 0.25 mm × 0.25 μm)
Carrier gas	N2, 1.4 mL·min^{-1}.
Injection temperature	250°C
Split ratio	50 : 1
Column condition	
Initial temperature (hold time)	150°C (1 min.)
Program rate (hold time)	200°C, 5°C.min. (5 min^{-1}.)
Post run temperature (hold time)	300°C (5 min.)
Detector temperature	270°C
Detector	FID (flame ionization detector)

The IR spectra of wood were obtained using an attenuated total reflection infrared spectroscopy (ATR-IR, Bruker Optics, Germany). The PCA was performed using the measured ATR-IR spectrum data. The data for the PCA were collected using the ATR-IR, and the spectral data of 10 replicates were collected for each sample. All collected spectral data were subjected to the second derivative by the fifth-order polynomial using the Savitzky-Golay algorithm (Savitzky and Golay 1964). The spectral data of the samples pretreated with the second-order derivative was obtained using Unscrambler® Ver. 9.8 (CAMO Software Inc., Norway). The PCA was carried out at 1.650-1.350 cm^{-1}, the area representing the molecular vibration of lignin and holocellulose.

RESULTS AND DISCUSSION

Changes in the physical properties of wood

Changes in the physical properties of ISPM 15 treated wood with elapsed time after treatment are presented in Tab. 3. The initial moisture content of the ISPM 15 treated wood was lower than that of the untreated wood. To maintain the temperature of the wood core at 56°C for more than 30 minutes, the heat treatment is carried out at 70°C for ≥ 2 hours to ensure that moisture is removed and the surface moisture content of the wood is lowered.

A comparison of surface moisture content of different species revealed that spruce had lower initial moisture content due to kiln drying, which is unlike other wood types, and the difference between before and after heat treatment was not significant. As the woods were maintained under atmospheric humidity at constant temperature condition, all three kinds of wood showed similar moisture content after four weeks regardless of heat treatment. Storage at constant temperature resulted in wood shrinkage as the moisture content of the wood decreased, resulting in a negligible reduction in the volume of wood and a marginal reduction in the weight of wood. As the wood surface became hydrophobic by heat treatment, the contact angle of the wood surface increased with time, and the contact angle of ISPM 15 treated wood was higher than that of the untreated wood. According to the heat treatment, the water absorption time of the wood surface changed proportionately with the contact angle. These results indicate that these dimensional changes are caused by storage at constant temperature rather than heat treatment. Therefore, it is difficult to observe the irreversible changes in the physical properties of wood on ISPM 15 processing.

Tab. 3: Dimensional changes in ISPM 15 treated wood during storage time of four weeks.

Storage time		0 week		1 week		2 weeks		3 weeks		4 weeks	
Content		Control	ISPM 15	Control	ISPM 15	Control	Control	ISPM 15	Control	ISPM 15	Control
Surface moisture content (%)	Larch	38.4 (5.0)[a]	28.5 (3.7)	35.2 (5.4)	26.9 (3.7)	30.7 (3.2)	27.4 (4.4)	28.2 (4.3)	27.4 (3.3)	27.2 (3.7)	25.6 (3.6)
	Douglas fir	52.8 (9.9)	44.2 (7.6)	53.3 (9.0)	43.4 (7.2)	31.2 (5.5)	22.3 (6.1)	29.8 (6.0)	27.6 (5.0)	27.3 (3.9)	26.0 (3.6)
	Spruce	21.1 (4.1)	17.7 (2.7)	18.9 (3.1)	16.5 (3.4)	23.0 (4.9)	24.6 (6.1)	23.3 (3.3)	23.8 (3.9)	22.1 (2.5)	22.8 (3.8)
Total volume [b] (cm^3)	Larch	1481.1	1458.5	1479.4	1442.8	1464.8	1442.4	1460.9	1437.5	1455.5	1433.7
	Douglas fir	1487.3	1487.9	1490.3	1500.3	1481.4	1487.4	1474.0	1482.0	1469.1	1477.7
	Spruce	1384.7	1367.7	1379.5	1362.9	1368.8	1357.3	1365.1	1356.2	1368.8	1354.8
Total weight (g)	Larch	1012.4 (53.9)	957.6 (41.6)	1007.5 (31.1)	943.7 (40.1)	988.1 (22.4)	935.0 (31.6)	979.3 (21.6)	934.5 (33.3)	968.6 (19.8)	929.1 (34.3)
	Douglas fir	1031.5 (85.3)	1008.1 (77.7)	1001.0 (82.1)	976.6 (74.4)	872.4 (69.8)	876.4 (76.6)	799.2 (59.9)	819.3 (78.0)	794.7 (59.7)	806.7 (76.0)
	Spruce	712.9 (37.3)	622.7 (61.0)	703.6 (36.5)	618.4 (61.0)	737.7 (41.0)	640.0 (58.4)	740.1 (40.8)	640.2 (66.5)	735.7 (40.7)	639.9 (63.8)
Contact angle (∡)	Larch	29.8 (8.9)	36.1 (16.9)	26.6 (11.9)	36.6 (4.5)	41.9(6.7)	52.3 (6.1)	39.5 (6.5)	54.1 (8.5)	44.4 (11.1)	57.8 (10.8)
	Douglas fir	51.8 (13.2)	58.9 (9.9)	47.3 (9.1)	46.5 (4.5)	48.8 (2.2)	64.9 (7.3)	54.4 (9.4)	71.9 (3.6)	64.7 (6.1)	79.5 (13.8)
	Spruce	47.2 (5.8)	60.5 (11.0)	60.4 (4.2)	64.6 7.5)	66.9 (8.8)	73.5 (4.5)	72.6 (3.5)	74.9 (2.0)	72.1 (9.8)	85.8 (16.9)
Water absorption (sec.)	Larch	4.0 (3.0)	5.0 (1.8)	3.2 (1.3)	7.0 (1.4)	17.3 (5.5)	12.0 (8.7)	12.4 (12.3)	23.5 (17.2)	16.2 (11.8)	41.1 (8.0)
	Douglas fir	2.1 (0.7)	2.5 (1.3)	2.3 (1.0)	3.4 (1.4)	3.4 (1.8)	61.3 (1.4)	4.2 (1.8)	N/A[c]	4.2 (0.4)	N/A
	Spruce	5.1 (2.1)	5.5 (1.5)	6.3 (2.7)	34.2 (5.6)	23.2 (14.5)	N/A	24.5 (5.6)	N/A	25.5 (6.0)	N/A

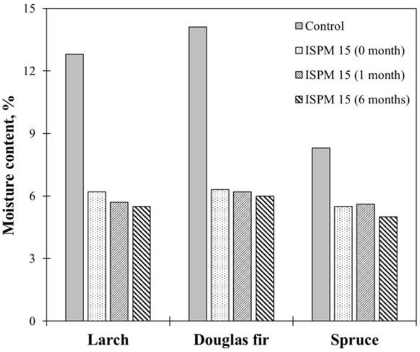


Fig. 2: Moisture content of the wood core after the ISPM 15 processing.

Fig. 2 shows the core moisture content of wood treated according to the ISPM 15. The core moisture content of larch and Douglas fir before the ISPM 15 processing was found to be more than 12%. However, the spruce had a low moisture content of less than 10% before the ISPM 15 processing. This is because the spruce has already been kiln dried. The kiln drying condition of the spruce is assumed to be milder than the ISPM 15 condition. With the ISPM 15 processing, these timbers presented a core moisture content of around 6% regardless of the species. The core moisture contents of ISPM 15 treated woods remained almost unchanged after 6 months. The tracheid is used as a translocation pathway for moisture and nutrient in coniferous wood types. As there are holes between the tracheid, moisture can be supplied not only in the tangential direction, but also in the radial direction (Petty 1972). However, with heat treatment, the pit membrane (i.e., torus and margo) blocks the pores in the cell walls. This process is called “torus closes the pit” (Fuhr et al. 2011). It is difficult for the wood with clogged walls to remove moisture. This might be the reason for no change in moisture content between the wood core and wood surface observed in this study.

Changes in the chemical properties of wood

Tab. 4 shows the changes in the chemical properties of three types of softwood before and after the ISPM 15 processing.

Tab. 4: Chemical composition and the result of alkaline nitrobenzene oxide of woods (unit: %).

Wood species / Content		KOREA Larch		USA Douglas fir		EU Spruce	
		Control	ISPM 15	Control	ISPM 15	Control	ISPM 15
Cold water extracts		11.0	11.4	7.7	6.9	5.9	4.2
Hot water extracts		16.0	13.0	12.0	10.9	5.3	4.2
1% NaOH extracts		15.5	15.7	14.5	14.8	8.7	8.5
Organic solvent extracts		7.9	6.9	9.6	6.6	5.0	5.0
Lignin	Klason lignin (KL)	30.4	31.0	30.0	30.7	28.1	28.3
	Vanillin / KL	17.5	16.6	20.3	21.1	19.4	19.2

The chemical composition of the untreated wood of Douglas fir and spruce showed to be similar to that previously reported (Kim et al. 2018). Although it was expected that there would be a change in the chemical composition of wood after heat treatment, no unusual change was found in the chemical composition before and after the ISPM 15 processing. Lignin has the lowest heat softening point among the main components of wood. Therefore, to investigate the possibility of chemical modification of lignin in wood by the ISPM 15 processing, the nitrobenzene oxidation of wood was analyzed. The results showed that the yield of vanillin before and after heat treatment remained unchanged. This indicates that lignin was not chemically altered by the ISPM 15 processing. Generally, the temperature for drying or reforming wood is more than 170°C and therefore considerable chemical changes can occur. However, the ISPM 15 processing is carried out at temperatures < 100°C for insecticidal and germicidal activities and therefore the chemical composition of wood is not significantly affected. Fig. 3 presents the result of free reducing sugar content in the wood treated according to the ISPM 15.

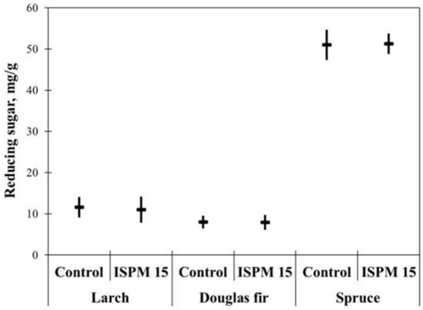
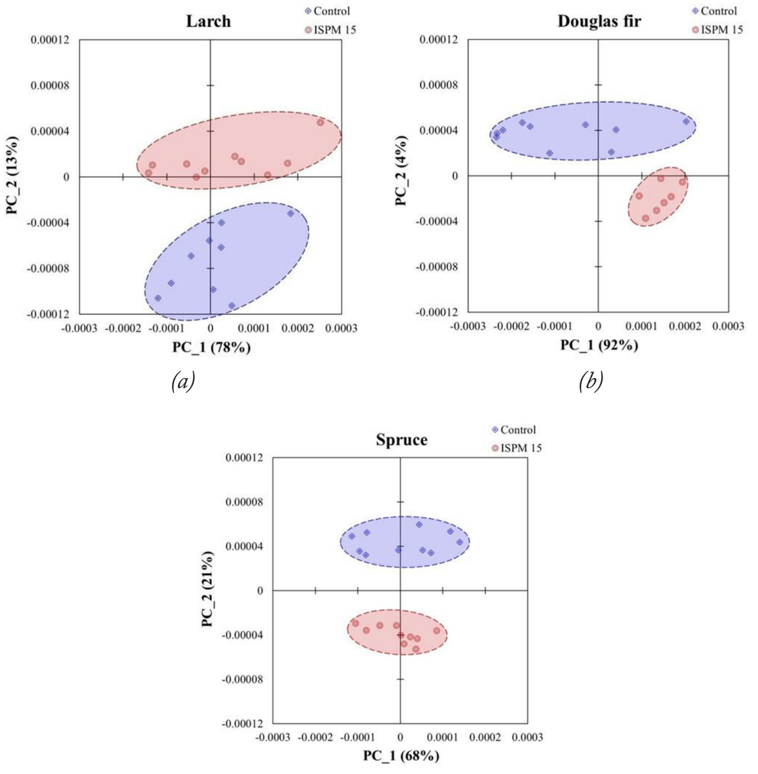


Fig. 3: Reducing sugar content of ISPM 15 treated wood packaging materials.

There was a difference in the free reducing sugar content between species, but there was no change in the reducing sugar content after heat treatment (Tab. 4). Iline et al. (2014) measured enzyme activity and sugar content after heat treating wood according to the ISPM 15. They observed changes in specific enzyme activity and sugar content during the early stage of heat treatment, but it was difficult to distinguish the difference in enzyme activity and sugar content after a storage period of two months. The results observed in this study were consistent with their observations. That is, it was difficult to confirm the changes in the content of free reducing sugar after the ISPM 15 processing at a relatively low temperature.

Chemometrical properties of wood

Principal component analysis (PCA) is one of the dimensional reduction techniques that reduce the difficulties in interpreting high- and low-dimensional signals. When the data are distributed in a multidimensional space, it is possible to find an axis that can reduce the dimension most efficiently and to reduce the dimension of that axis. In other words, it is essential to find the optimal axis among several axes, and the exact axis can be obtained by the PCA (Wold et al. 1987, Matthias 2006). Fig. 4 shows the PCA score plot of the three types of wood before and after the ISPM 15 processing.



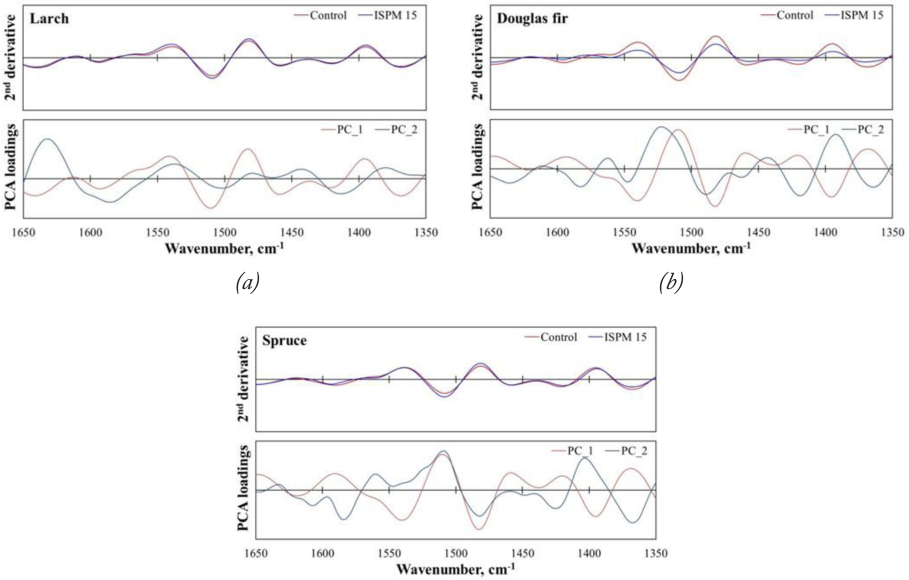
(a) *(b)*

(c)

Fig. 4: Principal component analysis (PCA) score plots of wood analyzed using the ATR-IR spectra.

The IR spectrum data obtained for each wood were collected and analyzed statistically. The results of the PCA score plot differed with species. Both Douglas fir and spruce were not classified based on the principal component 1 (PC_1) of the x axis. However, the three types of wood were classified according to heat treatment (i.e., control and ISPM 15) centering on 0 of the y axis (PC_2).

The PCA score plots showed the untreated larch samples as not heat-treated with negative values (-) around 0 of the y-axis; the positive values (+) showed ISPM 15 treated wood clusters. On the contrary, the PCA score plots of Douglas fir and spruce showed the untreated wood with positive values (0) on the y-axis, and the clusters of heat-treated wood with negative values. As all the three kinds of wood showed a classification pattern that can discriminate whether heat treatment was performed based on the (+) and (-) values of y axis, second derivative IR spectra and PCA loading data were compared to identify the components corresponding to PC_2. The second derivative IR spectra and PCA loading data were compared to track the components of PC_2 of the three woods. The results are shown in Fig. 5.



(a) *(b)*

(c)

Fig. 5: Second derivative spectra and the PCA loading data of wood packaging materials.

The PCA loading data show how the IR spectra contribute to the new principal component obtained by the PCA (Hwang et al. 2016, Kim and Eom 2016). The PCA score plot of all three kinds of wood can be classified by PC_1 / PC_2. As shown in Fig. 4, all wood were classified by PC_2 more accurately than by PC_1 according to the ISPM 15 treatment. Therefore, we tried to identify the characteristic IR peaks in the PC_2 loading data. The region with the most characteristic value in the loading data of larch was 1,632 cm^{-1}, which corresponds to the adsorbed water (Fengel 1993). The changes in the chemical and biochemical properties of wood treated according to the ISPM 15 processing could not be confirmed, but the clusters (Fig. 4) were divided according to the heat treatment of larch due to the changes in moisture content in the IR spectra. The specific factor of the PC_2 classifiable by the ISPM 15 heat treatment of Douglas fir is the CH2 scissoring vibration of cellulose with adsorbed water at 1.632 cm^{-1} and cellulose at 1.419 cm^{-1} (Fengel 1993, Schwanninger et al. 2004). In the case of spruce, the specific factor of PC_2 is different from that of other wood. This is because spruce has already been kiln dried. It means that the peak intensities of 1.482 cm^{-1} and 1.367 cm^{-1} (OH plane deformation vibration) on the loading data and the second derivative spectrum become stronger by ISPM 15 processing (Fengel 1993).

In general, among the different parameters of wood, the moisture content can be classified by the PCA according to the ISPM 15 processing, which can be easily changed by mild heat treatment. The results of this study showed that the classification of wood after the ISPM 15 processing can be confirmed by the moisture content of the wood; however, the moisture content of wood surface becomes ambiguous depending on the storage environment and period. The changes in the chemical and physical characteristics of the wood were not observed either before or after the heat treatment. However, the changes in the chemical properties occurring after the ISPM 15 processing were clearly distinguished using the ATR-PCA system.

CONCLUSIONS

The physico-chemical properties of the heat-treated wood according to the ISPM 15 were evaluated. Furthermore, the spectroscopic properties of the surface of wood and the core of timber were compared and analyzed. The results revealed that the ISPM 15 treatment of wood did not alter the physico-chemical characteristics of wood. However, after the ISPM 15 processing of wood, the core moisture of timber was around 6% regardless of the wood species. The changes in the chemical and biochemical characteristics were not observed either before or after heat treatment. However, the changes in chemical properties occurring after the ISPM 15 processing were clearly distinguished by using the ATR-PCA system.

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