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Evaluation of dried-wood odors: comparison between analytical and sensory data on odors from dried sugi (*Cryptomeria japonica*) wood

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Abstract The emissions of volatile organic compounds from air-dried, conventionally dried, and high-temperature-dried sugi wood were compared by gas chromatography-mass spectrometry. Terpenes were clearly the main compound group in the air-dried wood samples, whereas acetic acid was only detected in the high-temperature-dried wood samples, indicating that considerable changes occurred in the volatile compound emission profile during high-temperature processing. The most abundant compounds in the air-dried wood and conventionally dried wood were δ-cadinene, α-muurolene, and β-cadinene (sesquiterpenes) for all specimens, and α-pinene and D-limonene (monoterpenes) for conventionally dried wood and air-dried wood. In contrast, acetic acid was detected only in the high-temperature-dried wood. Sensory evaluation of volatile organic compounds was performed by 18 male university students. Volatile compounds of air-dried wood and conventionally dried wood were assessed as being significantly more soothing than those from high-temperature-dried wood.

Key words Dried wood · Odor · Sugi · Sensory evaluation · Drying process

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Introduction

Heat treatment is used to modify the properties of lumber to resist dimensional changes due to humidity to provide better heat insulation, to improve resistance to decay and weather, and to reduce moisture deformation. With the increasing demand for dried lumber in Japan, several drying methods have been applied to sugi (*Cryptomeria japonica* D.Don), which is the main species used in Japan.[1–4]

The main constituents of wood are carbohydrates, namely cellulose, hemicellulose, and lignin, with minor proportions of extractives and inorganic compounds. Much research has been conducted on the volatile organic compounds (VOCs) that are emitted from wood during the drying process and on the effect of temperature and humidity on emissions from lumber during drying. There have also been analyses of the chemical changes that occur during heat treatment of wood. There are some reports about the biological activities of the VOCs from lumber during drying. For example, the termite resistance of sugi heartwood samples, which were kiln-dried under several temperatures and processes, was investigated in relation to wood extractives.[5] On the other hand, physiological and psychological parameters in humans are known to change concomitantly with the inhalation of essential oils, such as the wood oil of Taiwan hinoki (*Chamaecyparis taiwanensis* Matsum. et Suzuki) or its components.[6,7] However, there is no report on the sensory data for the VOCs from dried wood.

In this study, sugi wood was treated by air drying, conventional drying, or high-temperature drying, after which gas chromatographic analysis and sensory evaluations were conducted on VOCs that were emitted from the dried sugi wood. The relationship between analytical data and sensory evaluation was evaluated for these three types of dried wood.

Experimental

Sample preparation

Green sugi (*Cryptomeria japonica* D.Don) boxed-heart square timber grown in Tokushima, Japan, was used. Three pieces of lumber with dimensions of 25 × 12 × 400 cm were divided into three samples, one for each of the three drying methods, as shown in Fig. 1. The initial moisture content (MC) of the drying samples was calculated as the average of the two MC specimens cut from each end of the samples. The moisture content of the specimens was obtained after oven drying at 105°C. Trees were felled in Naka town, Tokushima Prefecture, in 2006, and transpirational drying was allowed to take place for 120 days. The lumber was arranged in layers with separating stickers, and air-dried in a climate-controlled room at 20°C and 45% relative humidity (RH) for 80 days (air drying).

Conventional drying and high-temperature drying were carried out with SK Automatic wood-drying equipment (Shimshiba) and high-frequency heating drying equipment (Yasujima), respectively. The schedules of the conventional drying and high-temperature drying are shown in Tables 1 and 2, respectively.

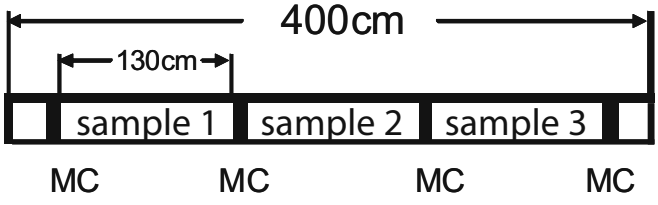

Fig. 1. Sample division for drying. *MC*, Moisture content

Table 1. High-temperature drying schedule

Time (h)	Dry bulb temperature (°C)	Wet bulb temperature (°C)
24	95	95
72	120	90

Table 2. Conventional drying schedule

Time (h)	Drying temperature (°C)	Exhaust temperature (°C)	Steaming temperature (°C)
2	50	49	47
2	60	59	57
2	70	69	67
2	80	79	77
48	80	78	76
48	80	77	75
48	80	76	74
48	80	76	74
48	80	75	73
48	80	75	73
48	80	73	71
48	80	71	69
48	80	71	69
6	80	78	76

Measurement of volatile organic compounds

Volatile organic compounds emitted from air-dried and heat-dried wood flakes (10 mm wide × 10–20 mm long × 1 mm thick) were collected into PEJ-02 tubes (Supelco) by weighing two randomly selected wood flakes and enclosing them inside a 1.0-l glass container. The air flow rate through the container was about 100 ml/min, and the temperature of the container was 25° ± 2.0°C. The collected volatiles were removed from the PEJ-02 tube by heating the trap with an Automatic Thermal Desorption System (ATD400, Perkin Elmer) at 280°C for 15 min. The compounds were cryofocused in a cold trap (air-monitoring trap, Supelco) at 0°C. While heating the cold trap, volatiles were transferred to an HP-5MS capillary column (30 m × 0.25 mm i.d. × 0.25 μm film thickness, Agilent) and analyzed by gas chromatography-mass spectrometry (GC-MS; Hewlett-Packard GC type 6890, MSD 5973). The temperature program began at 40°C where it was held for 15 min, after which it was raised at 4°C/min to 180°C where it was held for 15 min. Then it was raised at 5°C/min to 280°C, where it was held for 15 min.

All mass numbers between *m/z* 15 and 550 were recorded (SCAN technique). Individual compounds were identified by comparing mass spectra with data from the NIST Library, from the literature,[8] and from nuclear magnetic resonance spectra, the details of which are given in a previous report.[9] The total emission of VOCs was first calculated by combining the peak areas of all identified compounds, after which the relative proportions of individual compounds from the total emission were calculated.

Sensory evaluation

A total of 18 male university students (mean age 22.0 ± 1.0 years) were the subjects in this experiment. After giving the subjects sufficient information in the anteroom, including the purpose of the experiment, informed consent was obtained from them. The subjects inhaled volatile compounds of dried wood with their eyes closed in an artificial climate chamber controlled at 23°C, 50% RH, and a light level of 100 lx. Each of the sugi wood samples treated with one of three different drying processes was prepared in the form of wood flake. A 300-g amount of each sample was placed into a 24-l smell bag and saturated. A smell-emitting apparatus was used to generate the volatile compounds of each sample at a flow rate of 3 l/min, which was inhaled by the subjects at 15 cm below their noses for 90 s. Sensory evaluation of volatile compounds after inhalation was made of the "soothing feeling" and "intensity of sensation." The soothing feeling was assessed using a 13-rank scale from 1 (extremely soothing) to 13 (extremely awakening). The intensity of sensation was assessed using a modified 16-rank scale based on the 6-rank scale of odor intensity consisting of "no odor," "very weak odor," "weak odor," "moderate odor," "strong odor," and "overpowering odor."

The soothing feeling and the intensity of sensation were analyzed using the Friedman test. If a significance level of

$P < 0.05$ was observed, the Wilcoxon matched-pairs signed-ranks test was performed. For this test, $P < 0.05$ was also considered statistically significant. The study was performed under the regulations of the Institutional Ethical Committee of the Forestry and Forest Products Research Institute of Japan.

Results and discussion

Volatile organic compounds

Volatile organic compounds released from the dried specimens differed depending on the drying method as shown in Table 3. The total VOC emissions from conventionally dried and high-temperature-dried wood were about 1.2 times higher than the corresponding emissions from air-dried wood.

The highest relative area percentage of the "other" group (see Table 3) was for the high-temperature-dried wood, while relative area percentage of the monoterpene group was the lowest in each of the tested samples. The highest relative area percentage for the monoterpene group was for air-dried wood.

Sesquiterpenes comprised the main group of compounds in the VOC emissions profile of all tested samples. The second most abundant group of compounds was labeled as "others," and the third most abundant group was the monoterpenes. The predominant compounds were δ-cadinene,

Table 3. Volatile compounds emitted from dried sugi wood

Group	Identifiled compounds	Air-dried wood		Conventionally dried wood		High-temperature dried wood	
		Emission (μg/m^2)[a]	Relative area (%)	Emission (μg/m^2)[a]	Relative area (%)	Emission (μg/m^2)[a]	Relative area (%)
Others	Acetic acid	–	–	–	–	28.2	0.12
	1-Methylene-1H-indene	–	–	–	–	2.2	0.01
	Naphthalene	3.0	0.01	0.6	0.01	2.1	0.01
	2-Iaopropyl-1-methoxy-4-methylbenzene	3.0	0.01	–	–	4.0	0.02
	1,3-Di-isopropyl-5-methylbenzene	–	–	–	–	4.7	0.02
	3-*tert*-Butyl-5-ethyltoluene	–	–	–	–	1.2	0.01
	3,3,7,7-Tetramethyl(2-methyl-1-propenyl)tricycle-heptane	95.2	0.46	71.0	0.30	109.7	0.45
Monoterpenes	α-Piene	32.7	0.16	22.7	0.10	3.6	0.01
	Tricyclene	3.0	0.01	2.8	0.01	–	–
	α-Terpinene	–	–	–	–	2.7	0.01
	o-Oymene	3.0	0.01	2.8	0.01	6.9	0.03
	D-Limnene	17.9	0.09	14.2	0.06	13.2	0.05
	δ-3-Carene	6.0	0.03	2.8	0.01	1.6	0.01
	1,4-Terpinolene	6.0	0.03	–	–	3.5	0.01
	Cymenene	3.0	0.01	0.6	0.00	4.1	0.02
	Bornyl acetate	–	–	–	–	2.1	0.01
Sesquiterpenes	α-Gurjuene	–	–	–	–	1.1	0.01
	Thujopsene	3.0	0.01	–	–	6.0	0.02
	δ-Elernene	62.5	0.30	312.3	1.33	955.6	3.96
	β-Cubebene	101.2	0.49	17.0	0.07	18.8	0.08
	α-Cubebene	725.9	3.50	85.2	0.36	37.2	0.15
	Clovene	35.7	0.17	39.8	0.17	80.0	0.33
	Alloaromadendrene	41.7	0.20	51.1	0.22	62.3	0.26
	Aromadendrene	556.3	2.68	1098.9	4.68	1257.3	5.21
	Eremophilene	17.9	0.09	176.0	0.75	191.9	0.80
	β-Caryophyllene	1883.2	9.08	2138.1	9.11	823.0	3.41
	Valencene	–	–	59.6	0.25	105.6	0.44
	α-Caryophyllene	1377.4	6.64	1380.0	5.88	1152.7	4.78
	α-Elemene	895.5	4.32	2180.7	9.29	2166.9	8.98
	β-Cadinene	3061.3	14.76	2390.8	10.19	2716.3	11.26
	Calarene	1431.0	6.90	550.8	2.35	526.0	2.18
	Copaene	202.3	0.98	1680.9	7.16	1084.2	4.49
	α-Muurolene	4081.7	19.68	3980.9	16.96	5079.6	21.05
	β-Pachoulene	–	–	–	–	26.1	0.11
	γ-Muurolene	14.9	0.07	–	–	18.4	0.08
	δ-Cadinene	6783.0	32.70	8333.7	35.51	8151.9	33.79
	α-Cubobene	–	–	–	–	97.3	0.40
	1,4-Cadinadiene	–	–	–	–	506.3	2.10
	β-Himachalene	181.5	0.87	2.8	0.01	4.1	0.02
	Cadina-1(10), 6,8-triene	59.5	0.29	19.9	0.08	55.0	0.23
	α-Gurjuene	68.4	0.33	5.7	0.02	2.3	0.01
	Guiazulene	20.8	0.10	19.9	0.08	17.9	0.07
	Total	21777.1	100.0	24641.6	100.0	25333.4	100.0

–, Not detected
[a]Values standardized against toluene

α-muurolene, and β-cadinene in the sesquiterpenes for all specimens, and α-pinene and D-limonene in the monoterpenes for conventionally dried wood and air-dried wood. Acetic acid was found only in low quantities in high-temperature-dried wood.

In the drying process used in commercial kilns, a high monoterpene emission rate has been found with Douglas fir and scots pine lumber. Boiling points for monoterpenes (e.g., 156°C for α-pinene), are lower than the temperature used in the heat-treatment process.[10,11] Thus, the monoterpenes had already boiled off and a great reduction in the emission of the terpenes from heat-treated wood was detected. For the quality of indoor air, our results indicate that emissions of monoterpenes are lower from heat-treated wood than from air-dried wood at room temperature.

The contents of α-pinene and D-limonene were not high. However, the flavor threshold values were lower than for other terpenes,[12] and α-pinene and D-limonene are used as treatments for asthma. In addition, it is reported that the inhalation of α-pinene and D-limonene is associated with a significant decrease in blood pressure, resulting in physiological relaxation.[13]

On the other hand, in the emissions of our air-dried wood samples, acetic acid was no longer detected. During the heating of wood material, wood constituents start to degrade, eventually leading to remarkable chemical changes. Thermal stability of hemicellulose, especially xylan, is known to be even lower than that of cellulose.[14] In the heat treatment, acetyl groups of hemicellulose are hydrolyzed and acetic acid is formed.[15] In the present study, acetic acid was one of the compounds in the VOC emitted from heat-treated wood, suggesting that acetyl groups of hemicellulose had already hydrolyzed. Acetic acid is also the major compound that is collected in the water phase during heat treatment of Norway spruce boards and it is known to pre-exist during wood drying at elevated temperatures.[16]

The content of acetic acid was not particularly high, but the flavor threshold value is very low,[12] and, as a volatile compound in heat-treated wood emissions, is a strong irritant that may irritate the respiratory system. In addition, acetic acid can have an unpleasant odor that induces negative emotions in humans.[17] Some of the minor identified compounds in the present wood, such as 1-methylene-1H-indene, naphthalene,2-isopropyl-1-methoxy-4-methylbenzene, 1,3-di-isopropyl-5-methylbenzene,3,3,7,7-tetramethyl(2-methyl-1-propenyl)tricycle-heptane, could be regarded as originating from the used adsorbent tubes.[18]

Regarding the "soothing feeling" in the sensory evaluation, as shown in Fig. 2, the volatile compounds of air-dried wood and conventionally dried wood were assessed as inducing a "slightly soothing feeling" while the volatile compounds of high-temperature-dried wood were assessed as inducing a "slightly awakening feeling." The volatile compounds of air-dried wood and conventionally dried wood were assessed as significantly more soothing than those of high-temperature-dried wood. Regarding the intensity of sensation, as shown in Fig. 3, the volatile compounds of the three different wood samples were similarly assessed as having a "moderate odor."

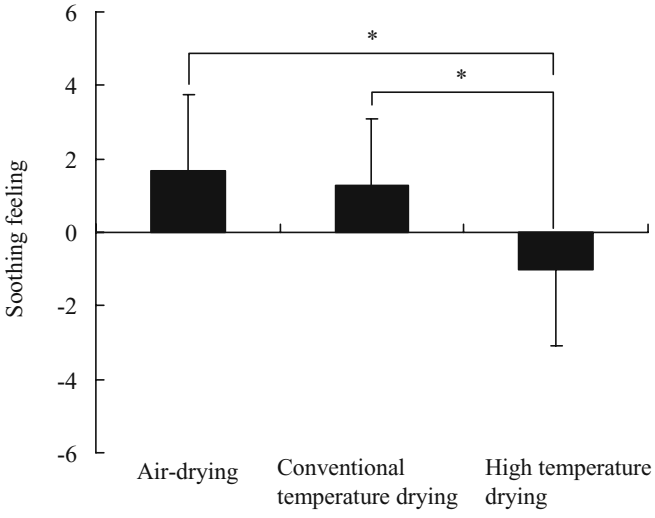

Fig. 2. Scores of soothing feeling in sensory evaluation on odors. Scores from –6 to 6 correspond to descriptive ratings: –6, extremely awakening; 0, neutral; 6, extremely soothing. Data given as mean ± standard deviation (n = 18). *Asterisk*, $P < 0.05$ by Wilcoxon matched-pairs signed-ranks test

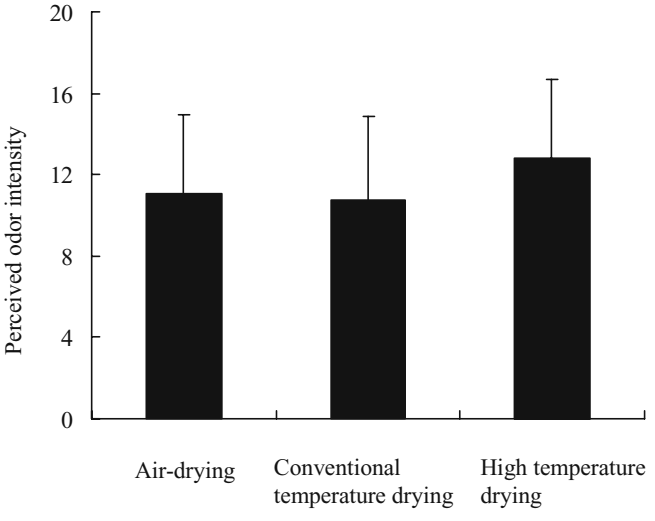

Fig. 3. Scores of perceived odor intensity. Scores from 0 to 20 correspond to descriptive ratings: 0, no odor; 4, very weak odor; 8, weak odor; 12, moderate odor; 16, strong odor; 20, overpowering odor. Data given as mean ± standard deviation (n = 18)

In conclusion, the present study has shown:

1. The VOCs in air-dried wood are composed of terpenes with the predominant compounds of δ-cadinene, α-muurolene, and β-cadinene in the sesquiterpene groups for all specimens, and α-pinene and D-limonene in the monoterpene groups for conventionally dried wood and air-dried wood. High-temperature-dried wood also contained terpenes, and contained high levels of acetic acid.

2. The volatile compounds inhaled from air-dried wood were assessed as being significantly more soothing than those from high-temperature-dried wood.

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