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# Characterization of volatile organic compound emissions from self-bonded boards resulting from a coriander biorefinery

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## A B S T R A C T

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*Coriandrum sativum* L.  
Essential oil  
Linalool  
VOC emissions  
Binderless boards

In this study, the VOC emissions from a self-bonded coriander board originating from a coriander biorefinery, were investigated. These emissions mainly result from the presence of essential oil in the manufactured materials. Firstly, an extensive analysis of the essential oil obtained from French coriander fruits showed the presence of linalool as the major component (72%), with an absolute concentration in the essential oil of 412 g/L. A characterization of the enantiomeric distribution of linalool, which is important in terms of its bioactivity, resulted in an enantiomeric excess of (*S*)-linalool of 77%. Further, the presence of this volatile oil in the coriander boards was confirmed through identification of the terpenoid compounds in the VOC emission profile. The area specific emission rate of linalool and camphor was determined at 125 and 25  $\mu\text{g m}^{-2} \text{h}^{-1}$ , respectively, at 25 °C and 50% RH, while their emission was found to increase by a factor of 3 with an increase in temperature of 10 °C. The renewable self-bonded boards could thus present potentially interesting alternatives for less sustainable materials in the construction or agricultural industry, where they could provide a significant added value in terms of indoor air quality or storage of food and agricultural products.

## 1. Introduction

*Coriandrum sativum* L. presents an annual herb belonging to the Apiaceae (Umbelliferae) family that originates from the Near East and Mediterranean area. It is currently mostly produced in India with an annual production of around 500 000 t and next to its main use as a spice, it has found applications in perfumery and cosmetics (Sharma et al., 2014). Coriander fruits are marked by the presence of both a vegetable oil and an essential oil fraction, the former constituting between 20 and 28% of the fruit, while the latter is present in typically less than 1% (Sahib et al., 2013). The vegetable oil contains petroselinic acid as a key component (around 70%), which presents a rather rare positional isomer of oleic acid and shows anti-inflammatory and anti-aging properties, leading to a recently growing interest in coriander vegetable oil from the food, cosmetic and chemical industry (Alaluf et al., 2002, 2005; Uitterhaegen et al., 2016b). Furthermore, coriander vegetable oil has recently been approved as a Novel Food Ingredient (NFI) and can be obtained efficiently and in high quality through thermomechanical pressing using a twin-screw extruder (European Food Safety Authority (EFSA), 2013; Uitterhaegen et al., 2015). The

essential oil fraction is defined by a high linalool content, typically ranging between 60 and 80%, while other important compounds include  $\alpha$ -pinene,  $\gamma$ -terpinene, camphor, limonene and geranyl acetate (Sahib et al., 2013). The fragrant oil, exhibiting a characteristic odor of linalool, is widely used in perfumery and the cosmetic industry, and in the food and pharmaceutical industry as a flavoring agent and adjuvant (Burdock and Carabin, 2009). Further, coriander essential oil has been shown to exhibit antioxidant and anti-inflammatory activity and inhibit a broad spectrum of micro-organisms (Beikert et al., 2013; Darughe et al., 2012; Silva et al., 2011).

In view of the increasing industrial interest in coriander vegetable oil, by-products from the oil extraction process will become more important in years to come and their valorization will present a key challenge in the light of sustainable processing and industrial viability. Recently, it has been demonstrated that the press cake resulting from the thermomechanical pressing of coriander fruits can be used for the production of renewable and environmentally friendly materials with a high cost-performance ratio (Uitterhaegen et al., 2016a, 2017a, 2017b). In particular, the deoiled press cake was transformed into binderless boards through thermopressing without the addition of any chemical

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adhesives, which are often toxic and can lead to harmful emissions such as formaldehyde. The self-bonding character of the press cake was mainly attributed to the protein fraction (Uitterhaegen et al., 2017b). These materials were proven viable alternatives for current commercial wood-based and resin-bonded materials such as particleboard (PB), medium-density fiberboard (MDF) and oriented strand board (OSB), the use of which will become increasingly unacceptable considering the depletion of wood resources and recent regulations concerning indoor air quality. Next to this, there is an increasing trend involving the addition of essential oils or essential oil compounds as active agents with a view to obtaining functional materials with antimicrobial, pesticidal and/or antioxidant activities (Hashim et al., 2009; Singh and Chittenden, 2010; Yingprasert et al., 2015). With regard to this, coriander essential oil could present an interesting solution for pest management, the control of bacterial and fungal growth in food or agricultural products or in housing and furniture applications, where mold growth implies significant health risks. Furthermore, its inherent presence in the press cake obtained from thermomechanical oil extraction from coriander fruits would designate an important added value for the materials resulting from the thermopressing of the cake. These materials could then show potential for high-value applications such as functional food packaging for prolonged shelf-life, storage of agricultural products or indoor construction materials for enhanced air quality.

This study presents a full characterization of the essential oil obtained from coriander fruits of French origin, with a clear focus on linalool as the key component. Next to this, a comparison with literature data on coriander essential oil is made in order to assess the variability of the oil composition resulting from the culture region and between different harvesting years. Most research studies on coriander essential oil only report the linalool content. However, as chirality presents a key factor in flavor chemistry and as it has been shown that the chirality of linalool determines not only its sensory profile, but also its biological activities (Aprotosoie et al., 2014), the enantiomeric distribution of linalool in the coriander essential oil was characterized. This report further aims to evaluate the presence and influence of this essential oil in self-bonded boards produced from the press cake of the twin-screw thermomechanical pressing process of coriander fruits. In particular, it presents a critical analysis of the potential of coriander essential oil for providing an added value to the renewable materials resulting from the biorefinery of coriander fruits. Further, a qualitative determination of volatile organic compound (VOC) emissions from the binderless boards allows justification of their use as furniture or building materials with respect to the indoor air quality. The importance of this research is pronounced by the development of a true coriander biorefinery which includes the sustainable production of coriander vegetable oil and extensive valorization of the process by-products. Therefore, these results could present significant industrial value with a view to recent environmental concerns and the current critical pressure towards sustainability and green processing.

## 2. Materials and methods

### 2.1. Materials

Coriander fruits were supplied by GSN Semences (Le Houga, France), consisted of the GSN maintenaire variety and were cultivated in the southwestern part of France. The fruit moisture content was determined according to ISO 665:2000 and was  $8.56 \pm 0.10\%$  (ISO, 2000). The press cake that was used for the purpose of this study resulted from the vegetable oil extraction process from coriander fruits. For this, a Clextral (Firminy, France) BC 21 twin-screw extruder was used with a screw configuration that was optimized for coriander fruits in a previous study (Uitterhaegen et al., 2015). Further deoiling of the press cake was carried out through a solvent extraction of 5 h with a 1 L Soxhlet apparatus and cyclohexane as the extracting solvent.

### 2.2. Chemicals

*n*-Butylbenzene (99.8%), (*R/S*)-linalool (> 99.0%), (*R*)-linalool (> 97.5%), camphor (> 96%), linalyl acetate (> 97%), geranyl acetate (> 99%), limonene (> 99%), nonane (> 99.8%), decane (> 99.8%), undecane (> 99.8%), dodecane (> 99.8%), tridecane (> 99.8%), tetradecane (> 99.8%) and pentadecane (> 99.8%) were obtained from Sigma-Aldrich (Saint Louis, MO, USA).  $\gamma$ -Terpinene (> 97%), *p*-cymene (> 99%) and  $\alpha$ -pinene (> 98%) were from Extrasynthèse (Genay, France). All solvents used in this study were purchased from Sigma-Aldrich (Saint Louis, MO, USA) or Merck (Darmstadt, Germany) and were of analytical grade.

### 2.3. Thermopressing

Self-bonded boards consisting solely of 200 g of deoiled coriander press cake were manufactured through thermopressing. For this, a Pinette Emidecau Industries (Chalon-sur-Saône, France) heated hydraulic press with a 400-ton capacity and a 150 mm  $\times$  150 mm aluminum mold was used. All raw materials were dried in a ventilated oven at 50 °C to a moisture content between 3 and 4% prior to thermopressing in order to avoid delamination of the panels. The applied operating conditions were optimized in a previous study (Uitterhaegen et al., 2017b) and include an applied pressure of 21.6 MPa, a mold temperature of 205 °C and a molding time of 300 s. All manufactured materials were conditioned in a climatic chamber (25 °C, 60% RH) for four weeks prior to characterization. The dimensions of the test specimens were 130  $\times$  30  $\times$  6 mm. Before testing, all samples were stored in hermetically sealed aluminum foil bags.

### 2.4. Essential oil extraction

Essential oils were extracted from 200 g of coriander fruits by hydrodistillation in a Clevenger-type apparatus for 5 h. Milled coriander fruits were introduced to a round bottom flask with 2 L of distilled water to recover the essential oil (1:10 ratio). The distillate was extracted with pentane, which was evaporated with N<sub>2</sub>. The oil samples were stored in airtight glassware at 4 °C.

### 2.5. Essential oil analysis

Gas chromatography analyses of the essential oils were performed with a Thermo Scientific (Thermo Fisher Scientific, Waltham, MA, USA) Trace 1300 gas chromatograph equipped with a Restek (Bellefonte, PA, USA) Rtx-5 capillary column (30 m  $\times$  0.25 mm; 0.25  $\mu$ m film thickness). A Thermo Scientific (Thermo Fisher Scientific, Waltham, MA, USA) ISQ QD mass selective (MS) detector was applied for qualitative analysis, while a flame ionization detector (FID) was used for quantitative analysis. The flow of the carrier gas (helium) was 1.5 mL/min. The oven temperature was programmed from 80 °C (hold 3 min) to 220 °C (hold 2 min) at 4 °C/min. The injector and FID temperature was maintained at 250 °C. Temperatures of the quadrupole and the ion source were 150 and 230 °C, respectively. The MS detector was run in electron impact mode with an electron energy of 70 eV. The mass scan ranged from 35 to 300 *m/z*. For each sample, 1  $\mu$ L was injected under split mode with a 100:1 ratio. Essential oil components were identified by comparison of their retention indices with those from literature and authentic standards available in our laboratory. The retention indices were calculated by the use of a series of *n*-alkanes (C<sub>9</sub>–C<sub>15</sub>) according to Eq. (1). Further identification was carried out through comparison with library mass spectra (NIST Version 2.0).

$$RI = 100 \cdot \frac{t_R(i) - t_R(n)}{t_R(n+1) - t_R(n)} + 100 \cdot n \quad (1)$$

*RI* retention index,

*t<sub>R</sub>(i)* retention time of compound *i*,

$t_R(n)$  retention time of alkane  $n$ ,  
 $t_R(n + 1)$  retention time of alkane  $n + 1$ ,  
 $n$  number of carbon atoms in alkane  $n$ .

Chiral separation of linalool was performed on a Restek (Bellefonte, PA, USA) enantioselective column (Rt- $\beta$ DEX; 30 m  $\times$  0.32 mm, 0.25  $\mu$ m film thickness) coupled with a flame ionization detector (FID). The flow of the carrier gas (helium) was 1.5 mL/min. The oven temperature was programmed from 80  $^{\circ}$ C (hold 1 min) to 220  $^{\circ}$ C at 2  $^{\circ}$ C/min. The injection volume was 1  $\mu$ L (splitless mode).

The enantiomeric content was expressed as an enantiomeric excess and calculated as  $ee\% = (a - b)/(a + b) \times 100$ , where  $ee\%$  is the percentage of enantiomeric excess,  $a$  is the major enantiomer, and  $b$  is the minor enantiomer. Chromatograms and mass spectra were evaluated using Xcalibur™ (Thermo Fisher Scientific, Waltham, MA, USA) software.

## 2.6. VOC emission analysis

### 2.6.1. Qualitative study

Emissions of volatile organic compounds (VOCs) from coriander materials were characterized through solid-phase microextraction (SPME) coupled with gas chromatography-mass spectrometry (GC-MS). Supelco (Sigma-Aldrich, Saint Louis, MO, USA) 100  $\mu$ m polydimethylsiloxane (PDMS), 75  $\mu$ m Carboxen/PDMS and 85  $\mu$ m polyacrylate fibers were applied for the adsorption of volatiles. Prior to the analyses, the SPME fibers were conditioned at 250, 300 and 270  $^{\circ}$ C, respectively, for 1 h in the injection port of the GC-MS. Test specimens of the materials to be analyzed were placed in a 600 mL glass cell and sampling was carried out through exposure of the SPME fibers to the cell headspace for 30 min at  $23 \pm 1$   $^{\circ}$ C. Blank analyses were conducted of the SPME fiber and the glass cell. The analytical conditions for GC-MS were according to those described in Section 2.5 for the analysis of essential oil, with the addition of a Supelco (Sigma-Aldrich, Saint Louis, MO, USA) 0.75 mm *i.d.* injection liner fitted to the injection port. The identification of compounds was carried out according to the method described in Section 2.5.

### 2.6.2. Quantitative study

Quantitative determination of the VOC emissions from coriander materials was conducted by the use of dynamic headspace analysis coupled to gas chromatography and was based on the method described by ISO 16000-6:2011 and ISO 16000-10:2006 (ISO, 2006a, 2006b, 2011). The experimental setup, incorporating the flow-through glass cell containing the material to be tested, is schematically represented in Fig. 1. It consists of a ZAG7001 Zero Air Generator system (Environnement S.A., Poissy, France) and a bubble humidifier equipped with mass flow controllers (type F-201CV, Bronkhorst, Ruurlo, The Netherlands) to supply the cell with clean air with a temperature of  $25 \pm 1$   $^{\circ}$ C,  $30 \pm 1$   $^{\circ}$ C or  $35 \pm 1$   $^{\circ}$ C and a relative humidity (RH) of  $50 \pm 5\%$  at a constant flow rate of 300 mL<sub>N</sub>/min. Emissions of VOCs were collected using sorbent tubes packed with Tenax TA (300 mg; 60–80 mesh; TERA environnement, Crolles, France). A volume of air

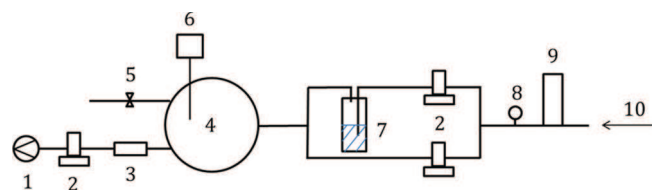


Fig. 1. Schematic representation of the experimental setup for the quantitative determination of VOC emissions from coriander materials through dynamic headspace analysis.

(1) pump; (2) thermal mass flow controller; (3) sorbent tube; (4) emission cell; (5) valve; (6) temperature and relative humidity probes; (7) humidity generator; (8) manometer; (9) active charcoal filter; (10) compressed air.

Table 1

Operating parameters of automated thermal desorption (ATD) and gas chromatography (GC) for quantitative VOC analyses.

Step	Parameter	Value
Tube desorption	Desorption temperature	280 $^{\circ}$ C
	Desorption time	10 min
	Desorption flow	26 mL min <sup>-1</sup>
Trap desorption	Desorption temperature	320 $^{\circ}$ C
	Focusing trap temperature	-30 $^{\circ}$ C
	Desorption time	15 min
	Transfer line temperature	230 $^{\circ}$ C
	Outlet split flow	12 mL min <sup>-1</sup>
GC separation	Capillary column	Agilent J&W DB-5(30 m $\times$ 0.25 mm; 0.25 $\mu$ m)
	Carrier gas	He; 2 mL min <sup>-1</sup>
	Oven temperature profile	35 $^{\circ}$ C (16 min), 35–240 $^{\circ}$ C at 6 $^{\circ}$ C min <sup>-1</sup> , 240 $^{\circ}$ C (7 min)
Flame ionization detection (FID)	Temperature	250 $^{\circ}$ C
	Hydrogen flow	50 mL min <sup>-1</sup>
	Air flow	450 mL min <sup>-1</sup>

varying from 0.5 to 3 L was sampled through the sorbent at a constant flow rate of 100 mL<sub>N</sub>/min by the use of a pump coupled to a mass flow controller (type 5890 TR, Brooks Instrument, Hatfield, PA, USA). The sampled volume was selected in such a way as to optimize the signal/threshold ratio without exceeding the breakthrough volumes of each compound (Simon et al., 1995; Moukhtar et al., 2005). Thermal desorption and analysis of the compounds was carried out using a Perkin Elmer (Waltham, MA, USA) Autosystem XL gas chromatograph equipped with a flame ionization detector (FID) and a TurboMatrix ATD thermal desorber. The analytical conditions are reported in Table 1. Qualitative analysis was made by injection of authentic standards of the compounds identified through the procedure described in Section 2.6.1. Quantification was based on external calibration curves obtained from liquid standards. Area specific emission rates (*SER*) were calculated according to Eq. (2).

$$SER = \frac{(m - m_0) \cdot q}{V \cdot S} \quad (2)$$

*SER* area specific emission rate ( $\mu$ g m<sup>-2</sup> h<sup>-1</sup>),

$m$  mass of VOC in the sorbent tube ( $\mu$ g),

$m_0$  reference value of the glass cell ( $\mu$ g),

$V$  sampled volume (m<sup>3</sup>),

$q$  air flow (m<sup>3</sup> h<sup>-1</sup>),

$S$  sample surface (m<sup>2</sup>).

All analyses were carried out through five repetitions and results are presented as mean  $\pm$  standard deviation (SD).

## 3. Results and discussion

### 3.1. Essential oil from coriander fruits

Essential oil was obtained in a yield of  $0.71 \pm 0.02\%$  on a dry basis from milled coriander fruits of French origin through hydrodistillation. The resulting yield complies with literature data reporting common essential oil contents of 0.3–0.9% in coriander fruits and reveals the coriander fruits of French origin to be a relatively rich source of essential oil (Mandal and Mandal, 2015; Sahib et al., 2013). A comparative overview of the main identified compounds in coriander essential oils from this study and from literature data is presented in Table 2. When comparing coriander fruits of different origins, it is clear that important differences can be found in the essential oil composition

Table 2

Comparative essential oil composition (relative% of peak area) of coriander fruits.

Compounds	$Rt_{exp}$	$Rt_{th}$	This study	France <sup>a</sup>	Italy <sup>b</sup>	Poland <sup>c</sup>	Romania <sup>d</sup>	Tunisia <sup>e</sup>	Pakistan <sup>f</sup>	India <sup>g</sup>	Bangladesh <sup>h</sup>	Canada <sup>i</sup>	Brazil <sup>j</sup>
$\alpha$ -Pinene	938	939	1.4	5.6	5.0	4.0	5.5	1.7	7.1	4.1	<i>n.d.</i>	4.5	4.0
Camphene	946	954	0.5	0.8	1.0	1.5	1.2	tr.	1.8	0.1	0.1	0.5	0.3
$\beta$ -Pinene	982	979	0.8	1.1	0.8	0.9	1.7	tr.	0.7	0.9	1.8	<i>n.d.</i>	0.4
Myrcene	994	991	1.2	0.6	0.1	1.2	1.4	0.1	1.0	0.3	0.6	1.3	0.8
<i>p</i> -Cymene	1027	1025	2.8	1.6	2.8	0.8	<i>n.d.</i>	0.6	0.9	0.5	<i>n.d.</i>	2.1	2.2
Limonene	1031	1029	3.4	1.9	2.6	1.3	4.7	0.3	4.5	0.6	0.4	2.6	1.3
1,8-Cineole	–	1033	<i>n.d.</i>	<i>n.d.</i>	0.1	<i>n.d.</i>	<i>n.d.</i>	0.1	0.2	0.4	<i>n.d.</i>	<i>n.d.</i>	<i>n.d.</i>
$\gamma$ -Terpinene	1060	1060	7.9	5.0	2.7	2.2	9.2	2.2	7.5	0.7	14.4	8.9	4.6
Linalool oxide	–	1070	<i>n.d.</i>	<i>n.d.</i>	0.7	<i>n.d.</i>	<i>n.d.</i>	0.3	<i>n.d.</i>	0.5	<i>n.d.</i>	0.6	<i>n.d.</i>
Terpinolene	–	1091	<i>n.d.</i>	<i>n.d.</i>	0.5	0.3	1.0	0.1	<i>n.d.</i>	0.2	<i>n.d.</i>	<i>n.d.</i>	<i>n.d.</i>
Linalool	1102	1097	71.9	71.7	67.8	71.2	50.7	86.1	55.5	75.3	37.7	57.0	77.5
Camphor	1149	1146	4.8	4.2	5.0	5.8	5.7	2.6	5.6	0.1	<i>n.d.</i>	6.4	2.6
Borneol	1170	1169	0.8	1.2	0.3	0.1	0.3	0.7	0.4	0.3	0.3	0.1	0.2
Terpinen-4-ol	–	1179	<i>n.d.</i>	<i>n.d.</i>	0.3	0.2	0.6	0.4	0.4	0.2	0.1	0.5	0.2
$\alpha$ -Terpineol	1195	1189	tr.	0.4	0.6	0.6	0.9	tr.	0.4	0.4	<i>n.d.</i>	0.1	<i>n.d.</i>
Decanal	–	1209	<i>n.d.</i>	<i>n.d.</i>	<i>n.d.</i>	0.6	0.1	tr.	4.7	<i>n.d.</i>	0.1	<i>n.d.</i>	0.5
Linalyl acetate	1256	1257	1.5	2.9	<i>n.d.</i>	<i>n.d.</i>	1.8	tr.	<i>n.d.</i>	<i>n.d.</i>	<i>n.d.</i>	7.1	<i>n.d.</i>
Geraniol	1268	1270	tr.	<i>n.d.</i>	2.0	3.4	3.3	1.6	2.2	0.8	1.9	0.1	0.6
Geranyl acetate	1386	1387	3.1	3.0	3.7	1.6	6.2	0.6	4.2	8.1	17.6	2.0	1.1

*n.d.* Not detected. tr. Traces.<sup>a</sup> (Uitterhaegen et al., 2015).<sup>b</sup> (Baratta et al., 1998; Caputo et al., 2016).<sup>c</sup> (Zawislak, 2011).<sup>d</sup> (Tsagkli et al., 2012).<sup>e</sup> (Sriti et al., 2009).<sup>f</sup> (Shahwar et al., 2012).<sup>g</sup> (Singh et al., 2006).<sup>h</sup> (Bhuiyan et al., 2009).<sup>i</sup> (Sriti et al., 2011).<sup>j</sup> (de Figueiredo et al., 2004).

(Table 2), which may result from several factors. On the one hand, the specific crop variety, as well as cultivating and climatic conditions may exert an important impact on the fruit and essential oil composition (Gil et al., 2002; Telci et al., 2006). On the other hand, coriander fruits have been shown to undergo significant changes during ripening (Msaada et al., 2007), leading to a key influence from the fruit maturity at harvest, while changes in the oil composition may also occur due to differences in extraction methods (Msaada et al., 2012). However, linalool always represents the major compound of the essential oil fraction and is most often situated in the typical range of 55–80%. The linalool content of the essential oil obtained from hydrodistillation of the coriander fruits used during the course of this study is 72%, which represents an intermediate value. Other major compounds include  $\gamma$ -terpinene (8%), camphor (5%), limonene (3%), geranyl acetate (3%) and *p*-cymene (3%). The low linalool content (38%) in the oil from Bangladesh (Bhuiyan et al., 2009) most likely resulted from immature coriander fruits, as it has been reported that the fraction of linalool in the essential oil increases during ripening of the fruits, together with a decrease of the geranyl acetate fraction (Msaada et al., 2007), which coincides with the high geranyl acetate fraction (18%) in the oil from Bangladesh. These fruits also displayed a relatively poor essential oil content of 0.42%, which further implies their immature nature (Bhuiyan et al., 2009; Msaada et al., 2007). When comparing the composition of the essential oil from this study and the one obtained from the same coriander variety and origin but from a different production year, i.e. 2014 (Uitterhaegen et al., 2015) vs. 2015 for the fruits used in this study, it is clear that only minor differences are present (Table 2). This suggests that climatic conditions show a relatively low impact on the essential oil composition, which is important in terms of the industrial capability of ensuring a stable product composition and providing a continuous product quality throughout different production years.

As linalool is the principal compound in coriander essential oil and as it is of great interest as a fragrant molecule with low toxicity and

significant therapeutic potential owing to its neurological, anti-inflammatory, antimicrobial and insecticidal properties (Aprotosoae et al., 2014), its absolute concentration in the coriander oil was determined through the setup of a calibration curve with *n*-butylbenzene as an internal standard. This rendered a linalool content of  $412 \pm 11$  g/L of coriander essential oil, which corresponds to 478 mg/g when taking into account an essential oil density of 0.86 g/mL. Pavlić et al. (2015) have reported an absolute linalool content of 835 mg/g of coriander essential oil obtained by hydrodistillation of Serbian fruits, although other commonly reported major compounds, such as  $\gamma$ -terpinene, camphor and geranyl acetate were either not detected or detected at relatively low contents. Further, the enantiomeric distribution of linalool was assessed through chiral capillary gas chromatography. Chiral GC analysis allows the separation of enantiomeric compounds and presents an important element when analyzing essential oils. Different enantiomers often display different biological activities and organoleptic properties (Marriott et al., 2001), and this has also been shown in the case of linalool. While (*S*)-linalool, which is predominantly found in coriander essential oil, is perceived as sweet and floral with citrus and fruity notes, (*R*)-linalool, the major enantiomer in lavender essential oil, has a woody aroma profile (Aprotosoae et al., 2014). Furthermore, it has been shown that the chirality of linalool not only influences its sensory characteristics, but also the biological properties, including neurological activity (de Sousa et al., 2010; Sugawara et al., 2000). The coriander essential oil obtained during the course of this study was found to have an enantiomeric ratio of  $88.5 \pm 0.5\%$  of (*S*)-linalool and  $11.6 \pm 0.5\%$  of (*R*)-linalool, which corresponds to an enantiomeric excess of (*S*)-linalool of  $76.9 \pm 1.0\%$ . This is in agreement with reported data on coriander essential oil from literature, which commonly ranges between 85 and 90% of (*S*)-linalool content (Parthasarathy and Zachariah, 2008). Moreover, the enantiomeric distribution is largely independent from the fruit origin and was proven a valuable tool for essential oil quality determination and the detection of adulteration (Aprotosoae et al., 2014). In view of this,



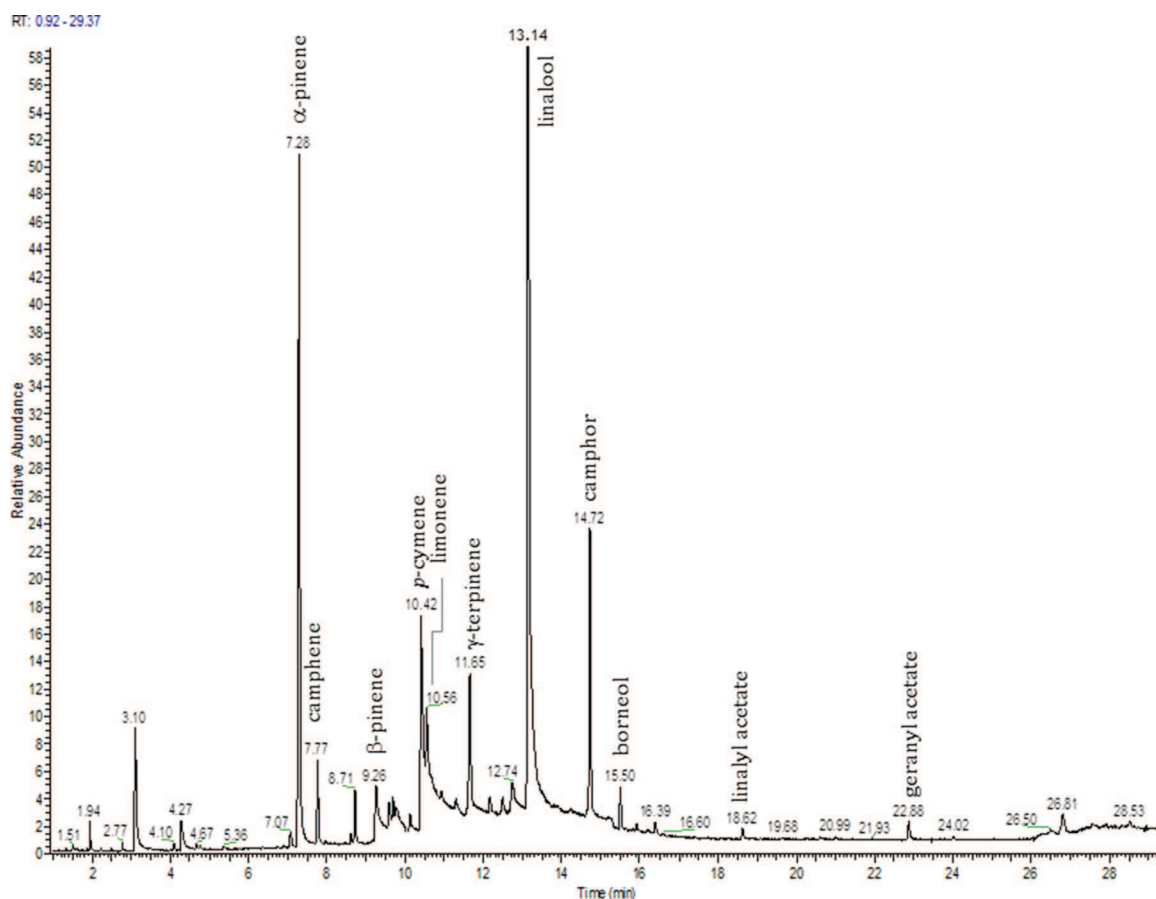


Fig. 2. Chromatogram resulting from the SPME-GC-MS analysis of the self-bonded coriander board using a 75  $\mu\text{m}$  Carboxen/PDMS fiber coating (T, 23  $^{\circ}\text{C}$ ;  $t_{\text{adsorption}}$ , 30 min).

the European Pharmacopoeia reports a maximum level of (*R*)-linalool of 14% in the standard for coriander essential oil (European Directorate for the Quality of Medicines and HealthCare, 2010).

### 3.2. VOC emissions from self-bonded coriander boards

Aiming at the valorization of process by-products and the development of a coriander biorefinery platform, the press cake resulting from coriander vegetable oil extraction through twin-screw extrusion was applied as a raw material for the production of renewable materials. The press cake was further deoiled through a cyclohexane solvent extraction prior to its use as the residual oil would create defects inside the boards due to its expression during thermopressing, which would show a detrimental effect on the mechanical performance of the materials (Uitterhaegen et al., 2016a, 2017b). Thermopressing of the deoiled press cake under optimized conditions provided cohesive panels, free of synthetic binding resins, with good mechanical performance characteristics (Uitterhaegen et al., 2017b). More specifically, the binderless boards showed a density of 1323  $\text{kg}/\text{m}^3$ , a flexural strength of 23 MPa, a modulus of elasticity of 4.4 GPa, and a thickness swelling of 31%. These mechanical properties are comparable to those of commercial boards such as OSB and PB, although they display a significantly higher density (typically around 650  $\text{kg}/\text{m}^3$  for commercial OSB or PB panels). This indirectly results from the absence of synthetic resins in the boards, as high temperature and pressure conditions need to be applied during the thermopressing process in order to mobilize the protein fraction, which acts as a natural binder (Tajuddin et al., 2016). As these materials could find applications in the building and furniture industry, it is important to also consider their impact on the indoor air quality. Due to numerous health implications such as sick

building syndrome and multichemical sensitivity, public awareness has recently been drawn towards indoor air quality and emission regulations have been implemented concerning commonly applied building materials such as MDF, OSB and PB (Jensen et al., 2001; World Health Organization, 2010). As a result, most studies have focused on the negative impact of toxic VOC substances such as formaldehyde, benzene and toluene emitted from building materials on human health and environment. However, the main source of such hazardous emissions, in particular formaldehyde, has been reported to be the glue used to bind the materials, which often consists of urea or phenol formaldehyde resins (Guo et al., 2002; Que et al., 2013). In this respect, the materials produced from coriander press cake present major advantages in terms of indoor air quality and environmental impact, as they do not involve the use of any synthetic resins owing to the natural binding capacity of the protein fraction of the cake. Next to this, VOCs emitted from natural materials such as wood and herbs may predominantly consist of terpenes which have been shown capable of delivering beneficial effects owing to their antimicrobial, analgesic, and neurological activities (Son et al., 2013). Care must be taken, however, as some terpenes have been shown to induce allergic symptoms and may cause problems for patients with chemical sensitivity (Rea et al., 2015). In this respect, coriander essential oil has shown no toxicity and produced no sensitization reaction throughout a series of studies and while some skin irritation has been reported for rabbits, this was not the case for human subjects (Burdock and Carabin, 2009). As a previous study has shown that part of this essential oil was maintained within the press cake after thermomechanical pressing of the fruits and that furthermore, the composition of this essential oil was similar to the one obtained directly from the fruits, it can be anticipated that materials produced from this press cake could lead to the emission of terpene compounds, in particular

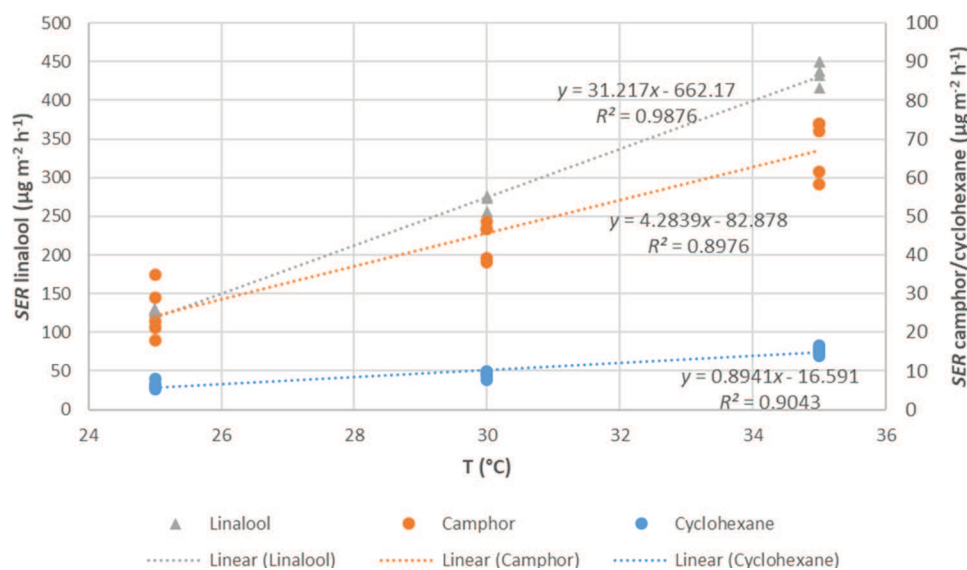


Fig. 3. Area specific emission rates (SER) of linalool, camphor and cyclohexane from the coriander self-bonded material as a function of temperature.

linalool (Uitterhaegen et al., 2015).

Emissions of VOCs from the renewable coriander self-bonded materials were characterized by the use of coupled SPME-GC-MS analysis. Three different fiber coatings (100 µm polydimethylsiloxane (PDMS), 75 µm Carboxen/PDMS and 85 µm polyacrylate fibers) were evaluated for the adsorption of volatiles from the material headspace. The chromatogram obtained using the Carboxen/PDMS fiber is presented in Fig. 2. Here, most of the compounds that were identified in the essential oil obtained directly from the coriander fruits were detected in the emissions from the coriander board. This confirms the presence of essential oil in the coriander press cake resulting from twin-screw extrusion and further its conservation throughout the hot pressing process for the production of materials. Next to this, a quantitative determination of the volatiles emitted by the materials was conducted through a dynamic headspace analysis using a glass test cell and coupled to thermal desorption and gas chromatography. Linalool was identified as the most important emitted compound with an area specific emission rate (SER) of  $125 \mu\text{g m}^{-2} \text{h}^{-1}$  at 25 °C and 50% RH, followed by camphor at  $25 \mu\text{g m}^{-2} \text{h}^{-1}$ . It is important to note that these values represent maximal emission rates, given that they were determined immediately after climatic conditioning of the panels, while several studies have reported a notable decrease of the emission of VOCs with time (Guo et al., 2002; Son et al., 2013). The emission rates of other compounds identified in the coriander essential oil and listed in Table 2, were below the limit of quantification (LOQ) estimated at  $1 \mu\text{g m}^{-2} \text{h}^{-1}$ , considering an air sampling volume of 1 L.

The predominance of linalool in the VOC emissions from the material is in accordance with the obtained composition of the essential oil, where it represents 72%, while camphor was also identified as a significant compound at 5% of the essential oil (Table 2). However, the second most important volatile compound in the essential oil, i.e.  $\gamma$ -terpinene at 8%, was emitted by the material in a minor amount below the limit of quantification. The limited presence of  $\gamma$ -terpinene in the VOC emissions from the coriander boards may be attributed to two distinct phenomena. Firstly, a considerable fraction of the essential oil was most likely lost through volatilization during thermomechanical pressing of the fruits as well as thermopressing of the press cake. Here, the compounds with a lower molecular weight, which are thus more volatile, will predominantly be lost and thus become relatively less significant in the remaining essential oil fraction in the material. This is the case for  $\gamma$ -terpinene, which shows a lower boiling point (183 °C) compared to linalool (199 °C) or camphor (209 °C). This phenomenon has been demonstrated in a previous study, where a decreasing

essential oil content of the press cake with a concurrent decreasing relative amount of  $\gamma$ -terpinene was observed with increasing extrusion temperature (Uitterhaegen et al., 2015). Another explanation lies within the relatively high lability of  $\gamma$ -terpinene when exposed to unfavorable temperature and light conditions. A study by Misharina (2001) reported the rapid loss of  $\gamma$ -terpinene through oxidation and identified it as the most unstable compound during storage of coriander essential oil in the dark and in daylight. Both phenomena, i.e. the relatively high volatility and instability of  $\gamma$ -terpinene as compared to the other major compounds of the essential oil, will probably contribute to the substantial loss of  $\gamma$ -terpinene during the processing of the coriander fruits that lead to the production of the self-bonded boards and consequently, to the minor emission of this compound by the material.

Next to the volatiles resulting from the presence of essential oil inside the coriander material, cyclohexane was detected as a VOC emitted from the boards. The origin of this compound lies within the additional deoiling process of the press cake prior to thermopressing, which consists of a cyclohexane solvent extraction. This residual oil extraction is carried out in order to avoid the formation of defects inside the boards during thermopressing due to the expression of oil (Uitterhaegen et al., 2017b). The emission of cyclohexane was quantified and resulted in a minor SER of  $6.3 \pm 1.0 \mu\text{g m}^{-2} \text{h}^{-1}$  at 25 °C and 50% RH. The regulatory reference concentration (RfC) for cyclohexane set by the U.S. EPA is  $6 \text{ mg/m}^3$  (U.S. Environmental Protection Agency (EPA), 2003), and even though this reference concentration may not be directly related to the determined SER for cyclohexane from the coriander boards, such concentration would unlikely be reached with the obtained order of magnitude of the cyclohexane emission rate. The low emission rate of cyclohexane from the boards probably results from its volatilization during the thermopressing process, as it shows a relatively low boiling point (81 °C).

Further, the impact of temperature on the emission rates of the main emitted essential oil compounds (i.e. linalool and camphor) and of cyclohexane was evaluated. The emission rate of the aforementioned compounds was determined at three different temperatures ( $25 \pm 1$  °C,  $30 \pm 1$  °C and  $35 \pm 1$  °C), while maintaining a constant relative humidity (RH) of 50%, and the results are presented in Fig. 3. From this, it is clear that the area specific emission rate (SER) shows an important dependence on temperature. The emissions from self-bonded coriander materials can result from a diffusion or an evaporation process or a combination of both. The diffusion of VOCs within a material is a function of the diffusion and partition coefficient of the specific compound, which are dependent on the compound's physical properties,

**Table 3**  
Comparative area specific emission rates ( $\mu\text{g m}^{-2} \text{h}^{-1}$ ) of terpenes from lumber and engineered wood materials.

	Lumber										Engineered wood						
	Japanese Red Pine <sup>a</sup>	Korean Pine <sup>a</sup>	Japanese Larch <sup>a</sup>	Japanese Cedar <sup>a</sup>	Norway spruce <sup>b</sup>	Scots pine <sup>b</sup>	PB (wood waste) <sup>a</sup>	MDF (wood waste) <sup>a</sup>	Plywood (Radiata Pine) <sup>a</sup>	Plywood (Oceania Timber) <sup>a</sup>	PB (Southern Pine) <sup>c</sup>	PB (Pine) <sup>c</sup>	PB (Douglas-fir) <sup>c</sup>	PB (Hardwood) <sup>c</sup>	MDF (Pine) <sup>c</sup>	MDF (Hardwood) <sup>c</sup>	
$\alpha$ -Pinene	6595	3258	318	130	120	890	7	15	117	1	42	42	11	9	n.d.	n.d.	
Camphene	186	148	18	8	7	20	< 1	2	4	n.d.	3	3	n.d.	n.d.	n.d.	n.d.	
$\beta$ -Pinene	691	286	68	19	66	29	21	65	104	< 1	19	72	1	3	n.d.	n.d.	
$\beta$ -Myrcene	< 1	10	2	9	12	31	< 1	2	< 1	< 1	-	-	-	-	-	-	
$\delta$ -3-Carene	61	9	29	67	19	680	55	96	< 1	< 1	n.d.	78	2	3	2	n.d.	
$\alpha$ -Terpinene	61	27	4	8	-	20	< 1	1	< 1	< 1	-	-	-	-	-	-	
$\beta$ -Phellandrene	-	-	-	-	14	-	-	-	-	-	-	-	-	-	-	-	
<i>p</i> -Cymene	85	70	20	13	-	-	n.d.	1	3	< 1	6	28	4	n.d.	0.5	0.4	
Limonene	1231	221	52	58	140	28	2	8	43	< 1	9	50	9	n.d.	2	5	
$\gamma$ -Terpinene	35	5	1	1	-	-	2	< 1	< 1	-	-	-	-	-	-	-	
Borneol	-	-	-	-	-	-	-	-	-	-	16	11	4	n.d.	7	2	

n.d. Not detected. PB Particleboard; MDF Medium-density fiberboard.

Testing conditions (T, RH): <sup>1</sup>(25 °C, 50%); <sup>2</sup>(23 °C, 50%); <sup>3</sup>(23 °C, 45%). PB and MDF materials were bonded with urea formaldehyde (UF) resin.

<sup>a</sup> (Son et al., 2013).

<sup>b</sup> (Hytinen et al., 2010).

<sup>c</sup> (Baumann et al., 1999).

the temperature and the structure of the material within which the diffusion is occurring (Liu et al., 2014; Zhou et al., 2015). More specifically, emission rates were found to increase with a factor of 3 for all compounds when the temperature was increased by 10 °C (from 25 to 35 °C). These results are in accordance with literature reports, where several studies have demonstrated increasing emission rates with a rise in temperature (Liu and Little, 2012). Lin et al. (2009) observed an increase in VOC emission rates with a factor of 1.3–17.8 when increasing the temperature from 15 to 30 °C, while Liu et al. (2014) reported a 1.5-fold rise in emissions with an increase in temperature from 23 to 30 °C.

For the purpose of comparison and to allow extensive interpretation and evaluation of the obtained results, literature data on the emission of terpenes from wood and wood-based engineered materials is presented in Table 3. Firstly, when considering lumber products, it is clear that these materials can emit considerable amounts of terpenoids, with reported SER values for  $\alpha$ -pinene of up to  $6500 \mu\text{g m}^{-2} \text{h}^{-1}$ . These emissions originate from the inherent presence of wood essential oil, which predominantly consists of monoterpene compounds with  $\alpha$ -pinene,  $\beta$ -pinene, limonene and  $\delta$ -3-carene as the most important constituents. The amount and composition of these wood volatiles and their emission varies with the wood species, as demonstrated in Table 3, and their presence is particularly marked in softwoods such as pine, spruce and fir (Baumann et al., 1999; Hytinen et al., 2010; Roffael, 2006). Further, the emission of terpenes is shown much less prominent from engineered wood materials as compared to lumber (Table 3). The manufacturing process of the engineered materials, which often comprises an intensive fiber pulping operation under high temperature and steam pressure, inevitably leads to the evaporation of an important fraction of the wood essential oil (Baumann et al., 1999; Son et al., 2013). Several studies have demonstrated a reduction of terpene emissions of 98–99% or 93% after a heat treatment of pine or spruce wood, respectively (Hytinen et al., 2010; Manninen et al., 2002). Even though the thermopressing process for the fabrication of the panels in this study will have inevitably led to the loss of an important amount of volatiles, the absence of an intensive steam operation for fiber processing allowed the conservation of a significant fraction of the essential oil in the panels, leading to a considerable emission of particularly linalool and camphor. Next to this, commercial engineered wood products are bonded using synthetic resins, most often urea formaldehyde resins, which may give rise to a considerable emission of non-terpenoid VOCs, notably formaldehyde. Son et al. (2013) has shown that while terpenes represented more than 90% of total VOC emissions for lumber originating from different species of pines, this was less than 30% for resin-bonded engineered wood materials such as PB and MDF.

The emission of monoterpenoids from building and furniture materials could contribute to an enhancement of the indoor air quality owing to their characteristic fragrance leading to physiological relaxation and to their bioactivities. While  $\alpha$ -pinene,  $\beta$ -pinene,  $\delta$ -3-carene and limonene are widespread among wood species, the emission of linalool from construction materials has never been reported. Studies have illustrated the anxiolytic, memory-enhancing and neuroprotective effect of coriander essential oil upon inhalation and have attributed this to the presence of linalool (Cioanca et al., 2013; Emamghoreishi et al., 2005; Gastón et al., 2016). Further, numerous reports have described antibacterial and antifungal activity against a wide range of micro-organisms (Elgayyar et al., 2001), while Silva et al. (2011) have stressed the significance of the *S*-(+)-enantiomer of linalool, which predominance was confirmed in this study, for the inhibitory activity against Gram negative bacteria including important food pathogens. Fumigant toxicity of coriander essential oil, owing to the presence of linalool and camphor, has been observed against different stored grain pests (Khani and Rahdari, 2012; López et al., 2008), while insecticidal and repellent activity was found against mosquito species (Benelli et al., 2013). Finally, linalool as a major compound of coriander essential oil effectively suppressed sprouting of potatoes during cold storage (Singh



et al., 2006). These bioactivities render the manufactured materials from a coriander press cake, with significant emissions of linalool and camphor, potentially interesting for applications in the building industry, as well as for the storage of agricultural and food products, with a view to an enhanced indoor air quality or an extended product shelf life.

Other renewable materials incorporating coriander press cake have been considered recently, including fiberboards from coriander straw, a crop residue, where the added coriander press cake acts as a natural binder inside the boards (Uitterhaegen et al., 2017a). An evaluation of the VOC emissions from these materials could reveal their potential for specific high-value applications. Future research should further include an assessment of the evolution of terpene emissions from different self-bonded coriander materials over time in order to gain insight into the durability of the potential beneficial effects resulting from the emission of essential oil volatiles, which is important in terms of commercialization of the products. Finally, it could be interesting to examine the influence of processing conditions, in particular the hot pressing temperature, on the amount and profile of VOC emissions from the resulting materials. Lowering the thermopressing temperature could lead to a significant rise in terpene emissions and the comprehension of this correlation would allow careful balancing of the mechanical performance of the material and the emission of linalool to the surrounding environment, depending on the envisioned applications and the desired material characteristics.

#### 4. Conclusion

This study comprises a comprehensive evaluation of the role of coriander essential oil in the development of an integrated coriander biorefinery. In particular, the essential oil obtained from coriander fruits of French origin was extensively characterized and (*S*)-linalool was identified as the major component with an enantiomeric excess of 72%. The absolute concentration of linalool was determined in the coriander essential oil and was 412 g/L. Further, the presence of this essential oil was confirmed in materials resulting from the valorization of by-products from the vegetable oil extraction process. Self-bonded boards produced exclusively from the coriander press cake were proven a valuable rare source of (*S*)-linalool and camphor emissions, with area specific emissions rates of  $125 \mu\text{g m}^{-2} \text{h}^{-1}$  and  $25 \mu\text{g m}^{-2} \text{h}^{-1}$ , respectively, at 25 °C and 50% RH. Owing to their characteristic odor and reported various bioactivities, these monoterpene emissions could present a potential added value for the coriander materials in the construction or agricultural industry.

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