Analysis of the Chemical Constituents of Dry-Kiln Condensate and its Technological Recovery – Part 1: Volatile Extractives

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In Central Europe the main species that are used for the production of sawn wood are spruce, pine, and European beech. After the sawing process, the sawn timber is technically dried to a certain moisture content by means of condensation drying. The water movement in the cellular structure, which is caused by the drying process, draws some of the extractives into solution. In the process of kiln drying, hot air evaporates the water and the dissolved extractives. Some of the water condenses on the floor and the walls of the kiln, while the rest is blown out with the steam. Therefore, condensate was taken from the bottom of the kiln as well as from the energy recovery system. A chemical analysis by means of purge-and-trap showed the presence of volatiles that could be classified as typical for the wood materials from which they originated under the conditions of high temperature and high moisture content.

Keywords: Condensate; Extractives utilization; Dissolved VOC; European Beech, VOC; Kiln drying; Pine; Spruce; Purge-and-trap

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INTRODUCTION

Several factors have an influence on the utilization of renewable biomass for material products, energy, and fine and polymeric chemicals. These factors include economic, environmental, and practical interests (Sattler *et al.* 2008). Economically, the forest products industry is in need of new products, as margins have been slashed in the last few years due to global competition and subsidized large-scale factories. From an environmental perspective, wood is a “green” source for solid materials, fuel, and chemical products. The practical interest involves the opportunity to replace petroleum-derived polymers with polymers produced from natural resources. Today, most of these bio-based polymers are produced from starch, leading to competition with food and animal feed markets (Schmidt and Padukone 1997; Parajo *et al.* 1996).

All over the world, governments are mandating stringent restraints on the emission of volatile organic compounds (Zwick *et al.* 1997). These emissions are produced, for example, during timber drying. One possibility for reducing emissions caused by timber drying is vacuum drying technology. Using this technology, the moisture as well as the volatile components can be condensed into liquid effluent (McDonald *et al.* 1999a). The drying process starts a complex reaction chain in the wood structure. Volatilization, steam distillation, thermal degradation, and/or hydrolysis initiate the release of a combination of chemical compounds (Cronn *et al*. 1983; McDonald and Wastney 1995; Fraser and Swan 1972; Roffael 1987). The organic extractives emitted during the kiln drying process are said to include primarily terpenes, methanol, acetic acid, formaldehyde, resin acids, and fatty acids (McDonald *et al*. 1999b). The emitted water vapor mixture may be cause for environmental concern (Cronn *et al*. 1983). McDonald and Wastney conducted a study in 1995 to analyze kiln emissions from conventional kiln drying of Radiata pine, but they did not find hazardous emissions.

Extractives from wood originate mainly in the resin, which dissolves in organic solvents and various hydrocarbons (Kärki and Väätäinen 2004). In coniferous wood, resin of two kinds can be found: physiologic resin of healthy wood and pathologic resin as a consequence of damage or infection. While physiologic resin consists of fats, waxes, and alcohols, pathologic resin contains resin acids and terpenes (Voipo and Laasko 1992). The extractives of wood that dissolve in organic solvents are aliphatic hydrocarbons, alcohols, fatty acids, terpenes, resin acids, sterols, and waxes. Extractives that dissolve in water include phenols, mono- and dimeric sugar derivates, pectin, and tannin. The proportion of extractives in wood most commonly ranges from 2 to 5% (Fengel and Wegener 2003). The extractives are located in specific morphological places in the wood structure. Resin, for instance, is located in resin canals, while waxes and fats are stored in parenchyma cells. Phenols are mainly found in heartwood and in the bark. All of these components are needed to enable the biological functions of the tree. Fats, for example, are a source of energy, while phenols, resins, and terpenes protect the tree from microbiological damage. All these components are washed out from the wood structure and are to some extent evaporated during the wood drying process. For this reason, the extractives are present in the air of the kiln (as well as in the condensed liquid collected in the kiln).

One way to reduce emissions is to condense their volatile parts and to use the liquid-bound extractives for technical purposes. A number of studies have been performed to investigate the composition of the condensates that accumulate during the process of vacuum kiln drying. Bucko *et al*. (1993) identified formic acid, acetic acid, levulinic acid, furfural, hydroxymethyl-furfural, formaldehyde, and acetaldehyde from the kiln-dried condensate of ash (*Fraxinus excelsior*), beech (*Fagus sylvatica*), and oak (*Quercus* spp.). A chemical analysis of the condensate of *Pinus radiata* generated from experimental vacuum kiln-drying was performed by Pervan and Draščić (2008). In their study, the condensate was sampled at regular time intervals throughout the total drying cycle. A chemical analysis of the green timber led to the conclusion that only 10% of the monoterpenes found in the wood structure could be recovered in the kiln condensate. In another study, Bicho *et al*. (1996) partially analyzed the condensates from Douglas fir (*Pseudotsuga menziesii*), Western red-cedar (*Thuja plicata*), and Western hemlock (*Tsuga heterophylla*). The condensates were obtained through radio-frequency-vacuum drying. Kärki and Väätäinen (2004) investigated the extractives from European aspen wood (*Populus tremula*) after high temperature drying. In their study, the extractive content in the wood mass depending on its position in the tree was examined, rather than the extractive content of the condensate. No significant differences between the extractive contents in different sample locations in the tree were found. However, a significant difference was found between the extractive contents of the heartwood and sapwood. The condensate from the steaming process of beech timber was analyzed by Ledig *et al*. (2003). In their study, only cumulative values of the components (*e.g.*, phenols and saccharides) were determined, and these values reflected a higher degree of extractives for indirect steaming than for direct steaming.

Only one study was found that dealt with the condensate analysis of kiln-dried spruce wood (*Picea abies*) (Dejmal and Zejda 2008). This study was performed according to the accredited method SOA-16. However, only spruce wood was analyzed, and the chemical compounds were not investigated precisely. In the present study, the condensates from spruce, beech, and a mixture of spruce and pine (common for packaging material) were obtained and analyzed precisely by means of purge-and-trap followed by gas chromatography-mass spectroscopy (GC/MS) analysis. A technological solution for the recovery of condensate was given. In addition to the chemical compound analysis, the prices of these compounds were researched, and thus, a rough investment cost analysis for the technological recovery of the condensate was generated.

EXPERIMENTAL

Condensate Preparation and Storage

Condensate samples were selected from an Austrian softwood sawmill as well as from a German hardwood sawmill. They included samples of spruce (*Picea abies*), a pine/spruce mixture (*Picea abies* and *Pinus sylvestris*), and European beech (*Fagus sylvatica*). The samples were collected at the first stage of the kiln drying cycle from 3 dryer charges, which had a peak temperature of 77 °C for spruce, 72 °C for pine/spruce, and 65 °C for beech. From each of three dryer charges, two 500-mL samples were taken at the first stage of the kiln drying cycle. The condensates were stored in amber glass bottles at 4 °C and periodically examined for changes in pH value. No change in pH was noted in any of the condensates during storage.

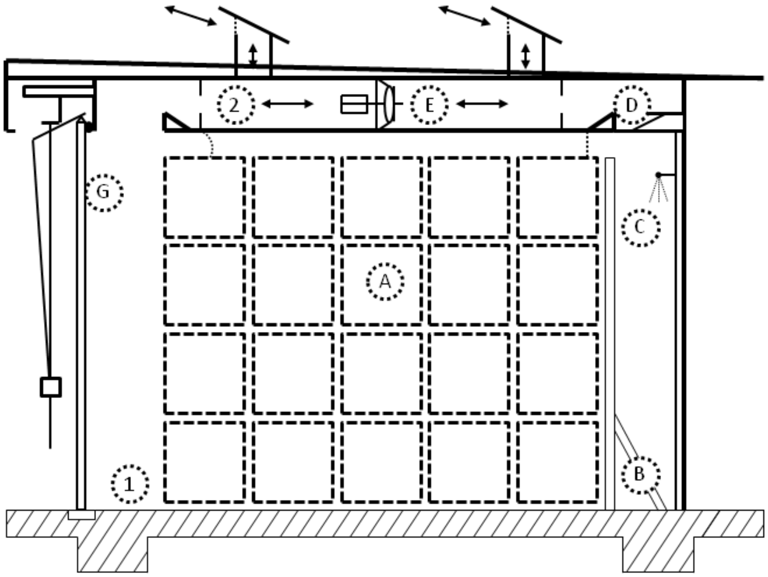

Fig. 1. Sectional drawing of a kiln with timber packages (A), stand (B), water spraying (C), damper register (D), and fan (E). Specimens were taken from the bottom (1) and from the intermediate ceiling (2).

The condensate samples were selected during the timber drying process from the bottom of the kiln (1) and from the intermediate ceiling (2), as can be seen in Fig. 1. The specimens were stored at 4 °C before the analysis process was begun.

Analysis of Condensate

Forty milliliters of each condensate was poured into a 40-mL clear glass screw vial (Sigma) and was subjected to purge-and-trap, by means of a homemade installation (Fig. 2) at 60 °C for 2 h, using technical air (hydrocarbon-free, Linde) as a purge gas and 200 mg of conditioned Tenax TA (60- to 80-mesh, Sigma) in TDAS 2000 glass tubes (Chromtech) as a sorbent. To reduce the temperature of the sorbent, the installation was put into a Binder KB 115 cooled chamber at 23 °C. After the sorption step had taken place, the Tenax tubes were spiked with 1 µL of a 0.373 µg/µL methanol dilution of toluene-d_8 (Sigma) per tube as an internal standard (ISTD) and rinsed for 30 min at 23 °C with dry technical air (Linde gas) to reduce the water content in the sorbent. The air mass flow during the steps described above was set to 6 mL/min. The sorbent tubes were then thermally desorbed using a TDAS 2000 thermal desorption unit (Chromtech) coupled with a programmed temperature vaporizing (PTV) injector of an Agilent 7890A/5975C GC/MS. The analytes were desorbed at 250 °C for 5 min and cryo-focused in the PTV injector at -15 °C. The PTV injector was then heated to 280 °C at its maximum rate, and the analytes were separated on an HP-PONA methyl siloxane column (50 m x 0.2 mm x 0.5 mm, Agilent) using helium as the carrier gas in the velocity range of 25 to 30 cm s^{-1} and using the following oven temperature program: 3 min at 35 °C, then increased to 160 °C at 10 °C min^{-1}, and finally increased to 310 °C at 20 °C min^{-1}. The PTV injector was operated in split mode at a split of 30:1. The MS detector was set to scan over a m/z range of 12 to 400. The individual compounds were identified using a database with a spectra of wood-specific substances (Wood K plus). The concentrations of the identified volatile organic compounds (VOC) were then calculated in toluene equivalents.

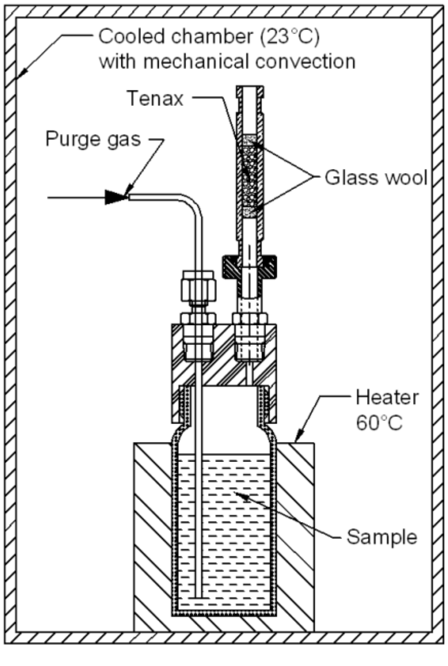

Fig. 2. Homemade purge and-trap installation

Purge-and-Trap Method Evaluation

To evaluate the average recovery rate and the quantification limit of the purge-and-trap method, a C6 to C14 alkane mixture was prepared by injecting 5 µL from each alkane in 955 µL of acetone. Twenty microliters of the solution were diluted with methanol in a ratio of 1:50, and 1 µL of the diluted solution was injected in 35 mL of deionized water. Finally, the mixture was analyzed according to the procedure described in the previous section (Analysis of Condensate). One microliter of the same methanol-acetone alkane mixture and 1 µL of ISTD were directly spiked in a Tenax tube and rinsed with dry technical air at 6 mL/min for 30 min. The areas of the GC/MS peaks of the alkanes, revealed by means of purge-and-trap as well as by direct injection, were divided by the corresponding area of the ISTD peak. These normalized areas were used for calculating the recovery rates in the alkane range of C6 to C14. It should be pointed out that the retention indices (RIs) of the C6 to C14 alkanes to a great extent covered the range of the RIs (not shown) of the detected substances in the condensate. The theoretical limits of quantization (LOQ) were calculated by dividing the known alkane concentrations in the deionized water solution by one tenth of the signal-to-noise ratios (S/N), determined from the Agilent MSD Chemstation software using “total ion concentration” as the signal (TIC).

Technological Implementation of Condensate Recovery

Conventional timber drying uses 60 to 95 °C warm air, which circulates through the timber packages. The timber packages are stacked with spacers to enable airflow. The kiln climate is controlled by venting the hot air and replacing it with ambient air. Thus, a drying program can be adapted according to the characteristics of the kiln load, *e.g.*, species, dimensions, and moisture content. In Fig. 3, a simplified transverse section of a kiln chamber with an energy recovery and dehumidification system is shown. The air, which is heated by the damper register (D) and circulated through the kiln by fans (E), heats the timber (A) and carries away the evaporated moisture and VOCs from the wood.

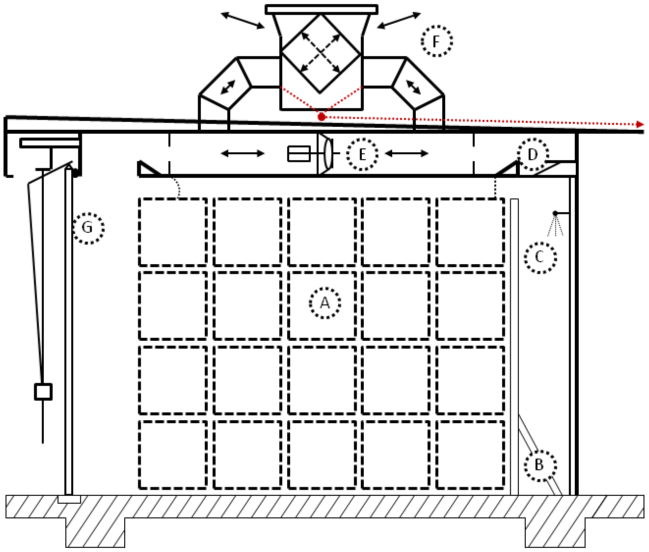

Fig. 3. Sectional drawing of a kiln chamber with timber packages (A), stand (B), water spraying (C), damper register (D), fan (E), and heat exchange device where condensate is ideally recovered (F); see red line.

The dehumidification unit of the kiln dries the wood in a closed system. This means that the dehumidification system condenses only as much moisture from the air as is necessary to generate the required relative humidity needed for the optimal drying process. The heat removed in the condensing procedure is returned to the kiln, with the fresh air coming in through a recuperator. The air flows over the lamella of the heat exchange unit, and the energy is transmitted into the incoming air while the condensed air moisture drops down and is collected at the bottom of the dehumidification unit, where it can be acquired for technical purposes.

The main disadvantages of the dehumidification unit are the high installation costs, as well as the higher operational and maintenance costs. The energy savings can compensate for part of these additional costs. However, in Millar's work (2006), the feasibility of using one dehumidification unit for the condensate recovery was examined. In that study, the VOC concentration in the kiln atmosphere was in the range of 10 ppm and was described as significantly lower than that of conventional kilns. This confirms the possibility of using dehumidification units for the recovery of chemical compounds released from wood during the drying process.

RESULTS AND DISCUSSION

Condensate Characteristics (Volatile Extractives)

Before analysis was performed, the condensate liquids were visually inspected. The condensates of softwood were of various shades of orange to orange-brown, while the beech condensates were brownish in color and contained suspended particles. The cumulative condensate characteristics of European beech, spruce, and the spruce/pine mixture are summarized in Table 1.

Feasibility of Using Condensate for Technological Purposes

In Table 2, the chemical components of the condensates are listed on the basis of Table 1. Here, the volatile extract value per m³ of condensate has been calculated depending on the amount and the price of the volatile organic components found in the liquid. The market prices were acquired from the catalog of one of the largest research chemical suppliers. For the extract value calculation, only 20% of the chemical prices were used due to the fact that processing costs, transportation costs, and sales costs must be included. The recovery rates (Table 4) were also considered when calculating the prices. The individual percentage contributions of the volatile compounds to the total extract value are also listed in Table 2.

The condensate that was collected during the industrial kiln drying processes contained the following volatile organic compounds: (i) terpenoids, (ii) ketones, (iii) alcohols, and (iv) traces of aldehydes and aromatic compounds. Small traces of benzene were found in the spruce condensate, but the concentrations were below the regulated limits. An interesting observation was that the fractions of alcohols and ketones were much higher than their usual fractions detected by direct emission determination of the corresponding dry wood materials (Hyttinen *et al.* 2010). This fact could be explained with the higher solubility of polar ketones and alcohols in water than terpenes which leads to their selective concentration in the condensate. High temperatures and moisture content during the drying process should also contribute for the oxidation of the monoterpenes.

Table 1. Characteristics of Condensates from Three European Species

Substances	Concentration [mg/m³]		
	Beech	**Spruce**	**Spruce/Pine**
Total Volatile Extract	**21.4**	**58.2**	**72.1**
2-Butanone	4.6	1.6	9.9
Ethyl acetate	0.0	0.0	2.0
Butanol	0.0	1.1	0.4
Benzene	0.0	0.8	0.0
Isopropyl nitrate	0.0	2.4	0.0
2-Pentanone	1.9	0.9	5.9
1-Pentanol	0.0	4.7	6.6
Toluene	0.2	0.8	0.9
Hexanal	0.9	0.8	1.9
1-Hexanol	0.1	2.7	2.3
3-Heptanone	1.5	0.0	0.1
2-Heptanone	1.4	1.1	1.4
2-Heptanol	1.5	0.0	0.0
α-Pinene	0.3	11.3	0.3
3-Octanone	0.0	0.1	3.2
2-Octanone	2.3	0.1	1.2
2-Octanol	3.3	0.0	0.0
β-Pinene	0.0	7.7	0.6
2-Ethylhexanol	0.1	0.9	0.0
delta-3-Carene	1.3	6.8	0.4
Limonene	0.0	7.6	0.2
Fenchone	0.0	0.8	3.5
Fenchol	0.0	0.7	3.2
Camphor	0.3	0.4	7.5
Pinocamphone	0.0	0.0	2.1
Isopinocamphone	0.8	0.0	7.7
Terpinen-4-ol	0.1	0.7	2.9
α-Terpineol	0.4	1.9	4.6
Verbenone	0.0	0.1	2.6
Tridecane	0.3	0.2	0.3
Sesquiterpenoids	0.0	2.0	0.5

By using a different analytical technique (SPME) and much shorter condensate-gathering time (only four hours), Dejmal and Zejda (2008) also showed that the fraction of hydroxylated and oxidized terpenoids in condensate is much higher than that of the softwood characteristic monoterpenes like alpha- and beta-Pinene, 3-Carene, and Limonene.

Table 1 shows that there were significant differences between the volatile contents of the three condensates. As expected, the concentrations of monoterpenes (such as alpha-pinene and 3-carene) in the spruce condensate were higher than those detected in the European beech condensate. Unexpectedly, the detected concentrations of monoterpenes in the spruce/pine condensate were much lower compared to spruce. It is possible that monoterpenes emitted during spruce/pine drying were oxidized/ hydroxylated at higher rates to keto- and hydroxy- terpenoids such as fenchone, fenchol, camphor, pinocamphone, isopinocamphone, terpinen-4-ol, and verbenone. These terpenoids were detected in higher concentrations in the spruce/pine- condensate.

Table 2. Prices for Chemical Components

Substances	Substance contribution to the total extract value [%]		
	European Beech	Spruce	Spruce/ Pine
Extractives value [EUR/1000 m³]	**0.59**	**2.13**	**3.61**
2-Butanone	**10.6%**	1.1%	3.8%
Ethyl acetate	0.0%	0.0%	0.6%
Butanol	0.0%	0.8%	0.2%
Benzene	0.0%	0.8%	0.0%
Isopropyl nitrate	0.0%	3.4%	0.0%
2-Pentanone	4.7%	0.6%	2.4%
1-Pentanol	0.0%	6.0%	5.0%
Toluene	0.4%	0.5%	0.3%
Hexanal	2.7%	0.6%	0.9%
1-Hexanol	0.1%	0.9%	0.5%
3-Heptanone	**16.2%**	0.0%	0.2%
2-Heptanone	3.4%	0.8%	0.6%
2-Heptanol	**10.8%**	0.0%	0.0%
α-Pinene	3.2%	**30.6%**	0.5%
3-Octanone	0.0%	0.2%	3.3%
2-Octanone	5.0%	0.1%	0.4%
2-Octanol	5.1%	0.0%	0.0%
β-Pinene	0.0%	4.1%	0.2%
2-Ethylhexanol	0.2%	0.4%	0.0%
delta-3-Carene	8.1%	**11.7%**	0.4%
Limonene	0.0%	**9.5%**	0.2%
Fenchone	0.0%	0.9%	2.3%
Fenchol	0.0%	0.9%	2.4%
Camphor	1.9%	0.6%	7.3%
Pinocamphone	0.0%	0.0%	2.8%
Isopinocamphone	6.1%	0.0%	**9.9%**
Terpinen-4-ol	9.0%	**15.8%**	**41.6%**
α-Terpineol	1.2%	1.3%	2.0%
Verbenone	0.0%	0.4%	**10.0%**
Tridecane	**11.2%**	1.5%	1.5%
Sesquiterpenoids	0.0%	6.5%	0.9%

According to Table 2, a maximum value of 0.59 € per 1000 m³ of condensate could be gained from the beech condensate. For spruce, 2.13 € per 1000 m³ could be gained, while the mixture of spruce and pine was revealed to have the highest potential gains, at 3.61 € per 1000 m³, which is in all likelihood due to the high terpene content in pine. The calculation of the technological feasibility was based on the raw material revenue for each kiln drying cycle. The additional costs for the dehumidification unit were calculated, estimating 10,000 € for each kiln chamber.

The condensate that is generated is typically transported to waste, as no toxicity has been detected. In this study, the profitability of using the condensate as a chemical resource was analyzed.

Table 3 shows a number of parameters needed for the cost calculation, including density, moist weight, water content, and the revenue that would be generated per drying cycle and per year using the chemical compounds in the condensate.

Table 3. Physiological Parameters and Revenue Calculation

Parameter	**European Beech**	**Spruce**	**Spruce/Pine**
Dry density [kg/m³]	740	430	475*
Moist density [kg/m³]	1340	774	855
Water content [kg/m³]	600	344	380
Kiln dry capacity [m³]	180	300	300
Peak dryer temperature [°C]	65	77	72
Condensate pH Value	5	7	7
Total water content [t]	108	103	114
Number of cycles [p.a]	13	70	70
Revenue per cycle [€]	0.06	0.22	0.41
Revenue per year [€]	1	15	29
* Average			

Green timber has an average wood moisture content (calculated based on dry weight) of 80%, which indicates a corresponding water content of approximately 45%. In Central Europe, hardwood is most often dried unedged, while softwood is edged or already sawn into dimensions. In addition to the shape of the boards, the drying time is also quite varied. Normally, it takes about five days to dry softwood timber, while hardwood requires at least four weeks. Assuming 350 production days for the kiln, 13 dying cycles can be accomplished with hardwood timber, and 70 with softwood, due to the longer drying cycles of hardwood (European beech). The two sawmills from which the condensate was obtained operated with kiln capacities of approximately 300 m³ for the softwood kiln and 180 m³ for the hardwood kiln. According to the calculations that were made, the revenue that could be generated from the volatile extractives by drying hardwood is 0.06 € per drying cycle.

Method Evaluation

The theoretical limits of the quantification (Table 4) were higher than the detection limits reported by Rosell *et al.* (2003), but taking into account the potential for further reductions in the split ratio, the overall sensitivity of the current P&T method should be slightly higher.

Table 4. C6 to C14 Recovery Rates and Theoretical Limits of Quantification

Alcane	**Concentration [μg/L]**	**Recovery rate**	**LOQ by split 30:1 [ng/L]**	**S/N (TIC)**
C6	1.87	73%	319	59
C7	1.94	102%	203	96
C8	2.01	94%	98	205
C9	2.05	87%	73	280
C10	2.09	86%	56	371
C11	2.11	76%	60	352
C12	2.23	67%	47	476
C13	2.16	48%	107	202
C14	2.16	54%	296	73
Average	**2.07**	**76%**	**140**	**235**

However, the substances used for the LOQ detection were not identical to those used by Rosell *et al*. (2003). The recovery rates in the range of C7 to C14 decreased, as expected, due to the decrease in the VOC volatility. The recovery rate of hexane was unexpectedly lower than that of heptane, but this could be explained by its loss (due to higher volatility) during the preparation steps before trapping took place.

CONCLUSIONS

According to the findings presented, the recovery of the volatile fraction (of dry-kiln condensate) alone with the help of dehumidification unit is not economically reasonable. Only the combination of extractive recovery combined with energy recovery systems seem to be economically viable. It is of further interest whether the inclusion of nonvolatile condensate extractives in the equation could contribute for making recovery more profitable.

ACKNOWLEDGMENTS

The support from the sawmills Schaffer in Eppenstein, Austria and B. Keck GmbH in Ehningen, Germany as well as the language support by Cornelia Riegler are gratefully acknowledged by the authors.

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Article submitted: July 3, 2013; Peer review completed: August 10, 2013; Revised version received: September 16, 2013; Accepted: September 19, 2013; Published September 26, 2013; Paper updated with corrections: November 8, 2013.