

## Analysis of the Chemical Constituents of Kiln-dry Condensate and its Technological Recovery – Part 2: Semi-volatile and Volatile Extractives

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The demand of the global economy for fossil resources needed for the production of fuel and basic chemicals is expected to exceed supply in the coming decades. Because of its heavy reliance on fossil fuels for increased efficiencies over the 20th century, the chemical industry has been particularly motivated to harness alternative raw materials, such as biomass, that are environmentally and economically sustainable. Biorefineries have provided stable, large-scale means of converting biomass into base chemicals, but until recently the main focus has been on the conversion of the mainly cellulosic fraction of edible plants into biofuels. Second- and third-generation biorefineries are striving to be more economically integrated and sustainable by utilizing raw material fractions to a greater extent and by not competing with the agriculture and food sector. The goal of this study was to evaluate the potential of kiln-dry condensate as a source for production of bio-based chemicals. The condensates of three typical European wood species were analyzed. Part 1 evaluated the volatile extractives; Part 2 concentrates on semi- and non-volatile extractives of kiln-dry condensates.

*Keywords:* Kiln drying; Kiln-dry condensate; Extractives utilization; Dissolved organic compounds; MTBE extraction; Bio-based chemistry

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### INTRODUCTION

As the global economy seeks more sustainable energy sources in the wake of global climate change and the reduction of energy-return-on-energy-investment (EROEI) in fossil fuel extraction, the 21st century chemical industry faces a unique set of challenges. Given its dependence on fossil resources for increased efficiencies throughout the 20th century, the chemical industry must fundamentally change almost all aspects of how it operates, particularly in terms of its raw materials. Bio-based primary materials, or biomass, may provide a powerful alternative source for the production of chemical applications (Ragauskas *et al.* 2006). This includes the use of trees, grasses, plants, and crops, which can be used for the production of both biofuels and chemical raw materials.

An additional motivation within the chemical industry to identify new raw materials is the advent of the REACH (Registration, Evaluation, and Authorization of

Chemicals) certification. For the first time, the entire supply chain of chemical products is analyzed. Because it is not known how many products will fall outside of the REACH certification, it may be necessary to discover, develop, and apply bio-based substitutes for some currently used chemical products.

The term biorefinery is used to describe a facility that produces energy and chemical products from biomass. The original impetus behind biorefineries was to improve the competitiveness of pulp and paper mills and increase revenue (van Heiningen 2006), leading to great interest in the topic by the research community, the forest products industry, and policy makers (Realf and Abbas 2004; Cunningham 2005; Magdzinski 2006; Ragauskas *et al.* 2006). In today's biorefineries, the raw material, such as sugar beet, is converted primarily into bioenergy and sugar derivatives. In the future, biorefineries may fractionate raw materials into many valuable components, *e.g.*, by fermentation, extraction, pyrolysis, and defibration (Clark 2007). The gained chemicals could be further processed by established organic chemistry methods (*e.g.*, Friedel Crafts reaction, reductions, or condensation). Such high-value refined products could build the platform for the formulation of existing chemical products, such as household products, pharmaceuticals, and other products, based on green raw materials.

One of the most critical factors in the success of a biorefinery is the feedstock (Towers *et al.* 2007). The optimum condition in implementing biorefinery processes is the usage of waste streams in existing forest product-based factories (*e.g.*, pulp and paper mills, sawmill, and/or panel factories). One such possibility for obtaining chemical raw materials from the sawmilling industry was described in the first part of this study (Rathke and Stratev 2013). The drying processes of kiln dries, which generates a huge amount of vapor, can be used in combination with a dehumidification unit to gain extractives. In this task, the dehumidification unit is used for energy recovery and, due to condensation of the vapor, produces a condensate that contains extractives. Extractives represent a minor fraction in the wood structure and consist of a wide range of hydrophilic and hydrophobic components (Amidon *et al.* 2011). The extractives consist of short-chain carboxylic acids, aldehydes, terpenoids (mono- as for instance  $\alpha$ -pinene,  $\beta$ -pinene,  $\Delta^3$ -carene and limonene, sesqui- and diterpenoids), sugars (primarily mono- and disaccharides) and sugar alcohols (glycerol, inositol), fatty acids, resin acids, phenolic substances (like simple phenols, lignans, stilbenes, flavonoids, and hydrolysable tannins) sterols and sterol esters, glycerides, glycosides, and proteins (Fengel and Wegener 1983; Amidon *et al.* 2011). The described extractives (if purified) can be easily used as raw materials in the chemical industry.

The first part of this study focused on volatile organic compounds (Rathke and Stratev 2013). In the second part, semi- and non-volatile organic compounds are analyzed and a more comprehensive estimation of extraction feasibility is introduced.

## EXPERIMENTAL

### Condensate Preparation and Storage

Samples from the same condensates used in the first part of this study (Rathke and Stratev 2013) were analyzed by utilizing liquid-liquid extraction and derivatization. The

samples include 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 three dryer charges. From each charge, two 500-mL samples were taken. Condensates were stored in amber glass bottles at 4 °C.

### Analysis of Condensate

Each condensate (250 mL) was used in a three-stage liquid-liquid extraction, with a total amount of 225 mL of MTBE (2-methoxy-2-methylpropane) as a solvent. Ten milliliters from the MTBE extract was then transferred into Pyrex® culture tubes and the solvent was removed through vacuum-drying at ambient temperature. The semi- and non-volatile residues were then derivatized with 80 µL of BSTFA (N,O-bis-trimethylsilyltrifluoroacetamide, Sigma), 20 µL of TMCS (trimethylchlorosilane, Sigma), and 20 µL of pyridine (Sigma) at 70 °C for 45 min. Heneicosanoic acid (Sigma) was used as the internal standard (ISTD). One microliter from each sample was injected into the injector of a gas chromatograph (Agilent 7890a) at 270 °C and separated on a HP5 GC column (30 m, 0.25 mm i.d., 0.25 µm, Agilent). The oven temperature was initially held for 1 min at 100 °C and then ramped up to 325 °C with a rate of 5 °C min<sup>-1</sup>, followed by a hold at 325 °C for 14 min. The mass spectrometer (MS) detector (Agilent 5975) was set to scan over a m/z range of 35 to 750. Approximate quantification was done by multiplying the known quantity of the ISTD with the quotient of the substance peak area and the ISTD peak area, assuming identical linear responses for all analyzed substances in their detection concentrations. The Wiley MS-database was used for qualitative identification of the compounds.

### Technological Implementation of Condensate Recovery

Conventional timber drying is performed in a temperature range of 60 to 95 °C, using air that circulates through the timber packages. Some kiln-dry chambers use recuperator units for energy purposes. The dehumidification unit condenses the moist air and generates the required relative humidity that is needed for the optimal drying process. The heat removed in the condensing procedure is returned, combined with fresh air in the kiln, by using a recuperator. In the recuperator, air flows over the lamellas of the heat exchange unit. The energy is then transmitted into the incoming air while the condensed air moisture drops down and is collected at the bottom of the dehumidification unit as condensate.

The main disadvantages of the dehumidification unit are its high installation, operation, and maintenance costs. The energy savings can compensate for part of these additional costs. However, when Millar (2006) examined the feasibility of using one dehumidification unit for condensate recovery, the VOC concentration in the kiln atmosphere was in the range of 10 ppm, 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 (Rathke and Stratev 2013).

## ESTIMATING FEASIBILITY

Consistent with the first part of the study (Rathke and Stratev 2013), the market prices of the determined chemical substances were used as the basis for estimating the economic feasibility of extractive recovery. The market prices (at the lowest purity offered) were acquired from the online catalogues of the two leading suppliers of chemicals for laboratory use. For price estimations to be closer to a real market situation, the influence of packaging size (purchasing quantity) and chemical purity was also taken into account (Eqs. 1 and 2). An exponential price-quantity function was introduced for calculating the market prices for aliquot packaging sizes of 5 kg (Eq. 1),

$$Price' [EUR/kg] = ax^b \quad (1)$$

where  $x$  is the quantity in grams and  $a$  and  $b$  are substance-specific coefficients.

For chemicals offered by the supplier in three or more packaging sizes,  $a$  and  $b$  were determined by deriving exponential regression functions. The exponential form for describing the dependency of the price on the packaging size was quite appropriate, with mean values of  $R^2$  equal to 0.97 and 0.86 for supplier 1 and 2, respectively. For chemical substances offered only in one or two packaging sizes, the price (in EUR/g) for the quantity closer to 5 kg was chosen for calculating the coefficient  $a$ , while the average value of the already calculated  $b$ -coefficients for the corresponding supplier was used as  $b$ . These average values of  $b$  for supplier 1 and 2 were calculated to be  $-0.442 \pm 44\%$  and  $-0.232 \pm 1\%$ , respectively.

$Price'$  is the chemical's price per kg for a packaging size of 5 kg for the lowest offered purity of the suppliers. The lowest offered purity was mainly in the range of 97% to 99.9%. However, there were cases where the lowest purity was beneath 95% and no other purities were offered by the suppliers. Therefore,  $Price'$  should be assumed to closely correlate with the price for producing substances (by an inexperienced producer) with current technology and in economically reasonable purities. Because the lowest offered purities for almost all substances in Table 1 were higher than 98%, a second price ( $Price''$ ) was introduced to estimate feasibility in the case where the producer would not have the infrastructure or know-how to purify extractives above a certain extent (97% was chosen). This approach lowered the single prices of the majority of the substances. However, it simultaneously drew attention to a couple of extractives with high economic potential, which at present are not offered by the suppliers in high purities or are not offered at all. For calculating  $Price''$ , the following dependency was used:

$$Price'' \left[ \frac{EUR}{kg} \right] = \frac{Price' (1-0.97)^{-2.719}}{(1-purity)^{-2.719}} \quad (2)$$

Equation 2 is the restatement of the function  $Price=f(impurity)$  (3), which was derived from observations of the chemical prices (of supplier 1) with varying contents of impurities (1-purity):

$$Price = c(1 - purity)^{-2.719}, R^2 = 0.99,$$

(3)

where,

$$c = \frac{\text{Price}'}{(1-\text{purity})^{-2.719}} \cdot \quad (4)$$

Price corrections based on Eqs. 1 and 2 were not used in the first part of this study, where only the lowest prices calculated per kg for the lowest available chemical purity were chosen. This correction for the volatiles listed in the first part was done in the current part. Cumulative results are given in Table 2.

## EXTRACTIVES PROCESSING AND ALTERNATIVE SOURCES

The biggest challenge in monetizing the condensate's potential would be the separation, purification and, eventually, storage of extractives. As a first step, solid phase residues like lignin and polysaccharides should be removed from the condensate. This could be done by means of ultrasonic application (Clark 2007) and filtration (Bicho *et al.* 1996). The actual separation could be done by applying different strategies, among them supercritical fluid extraction (Mukhopadhyay 2000) combined with chromatographic and selective membrane techniques. The use of the purge and trap technique (similar to the method presented in the first part of this study), steam extraction, or vacuum distillation could also be used as processing technologies. However, the choice of extractives purification steps would be dependent on the specific focus of the extraction. The authors did not conduct research on profit reduction due to extractives purification, but instead roughly estimated it by reducing the extractives value by 80%. Considering the low extractives concentrations in condensates, this estimation may still be optimistic.

It should be pointed out that feedstock from other productions, such as process water from an ABS<sup>TM</sup>-based biorefinery (Amidon *et al.* 2011) or condensate from the steaming processes for hardwood timber (Ledig *et al.* 2003), could also be suitable as extractive sources. It can be assumed that steaming and timber drying processes can be used for the extraction of natural and high-value chemicals, which can serve as a resource for chemicals currently derived from petroleum, coal, and gas (Amidon *et al.* 2008; Liu 2010; Liu *et al.* 2010).

## RESULTS AND DISCUSSION

### Condensate Characteristics

The analysed condensates (Table 1) contained large numbers of hydroxy-, keto- and dicarboxylic acids; saturated and unsaturated monocarboxylic acids; resin acids; terpenoids; aromatic compounds; and numerous unidentified extractives that to a great extent (mass spectra not shown) also belong to these groups. In addition, spruce condensate contained certain amounts of fenpropimorph (wood protection agent) and tetradecyltrimethylammonium bromide (surfactant), which have anthropogenic origins and are process-specific rather than raw material-specific ingredients of the condensate.

Unidentified compounds contributed from 9% to 47% of the total extractives content and can be considered to increase the feasibility of extractives recovery.

**Table 1.** Characteristics of Kiln-Dry Condensates from Three European Wood Species

Substance	Price' [EUR/kg] à 5 kg	Price'' [EUR/kg] à 5 kg and 97% purity	Concentration in condensate [mg/m <sup>3</sup> ]		
			Beech	Spruce	Spruce/Pine
Cyclohexenol	14.3	0.7	1.2	0.0	1.5
Lactic acid	34.7	916.7	1.7	5.3	0.8
Hexanoic acid	27.9	9.3	5.7	0.6	5.9
Glycolic acid	210.9	210.9	3.6	0.0	0.0
Levulinic acid	39.9	13.3	10.7	0.0	0.0
3-Hydroxypropionic acid	116.7	38.4	4.0	0.0	0.0
3-Hydroxyisobutyric acid	116.7	38.8	7.4	0.0	0.0
2-Ethylhexanoic acid	14.4	0.7	38.5	57.4	1.5
Octanoic acid	25.5	8.5	7.8	0.0	0.0
n.i. RT 5,88	-	-	0.0	0.0	5.9
3-Hydroxy-3-methylbutyric acid	1021.7	7.8	3.8	0.0	0.0
4-Oxohexanoic acid	8847.5	67.8	5.8	0.0	2.9
Benzoic acid	69.5	0.5	111.1	0.5	38.3
Benzeneacetic acid	53.2	53.2	18.3	0.0	11.2
n.i. RT 8,68	-	-	5.7	0.0	0.0
n.i. RT 8,85	-	-	1.4	0.0	63.1
Catechol	51.8	2.6	22.1	0.0	0.0
Methylsuccinic acid	1066.9	53.8	17.1	0.0	0.0
Butoxyethoxyethanol	22.8	1.1	50.5	0.0	0.0
Glutaric acid	178.6	178.6	36.9	3.5	7.3
Myrtenic acid			1.4	0.0	9.4
Phenylpropanoic acid	210.0	10.6	10.5	0.0	1.1
(2-Butoxyethoxy)acetic acid	2213.6	17.0	9.1	0.0	0.0
n.i. RT 11,82	-	-	11.3	0.0	0.0
n.i. RT 11,89	-	-	0.0	0.0	7.3
n.i. RT 12,95	-	-	20.8	17.0	44.3
n.i. RT 13,1	-	-	0.0	0.0	7.7
Adipic acid	5.2	0.3	8.4	2.1	0.0
n.i. RT 13,15	-	-	0.0	0.0	16.1
p-Menthane-1,8-diol	947.8	47.8	0.8	15.6	14.5
n.i. RT 13,63	-	-	11.7	2.1	0.0
m-Hydroxybenzoic acid	14.9	0.7	2.2	0.0	8.1
Tyrosol	777.5	258.2	16.3	0.0	0.0
n.i. RT 14,64	-	-	0.0	1.5	13.2
n.i. RT 14,95	-	-	10.3	0.0	0.0

Pimelic acid	123.5	41.0	15.4	3.7	0.0
p-Hydroxyphenylacetic acid	-	-	0.0	0.0	22.4
N-Tetradecyltrimethylammonium bromide	356.9	356.9	0.0	15.0	0.0
Suberic acid	124.1	41.2	48.6	11.9	7.5
9-Hydroxynonanoic acid	19959.7	152.9	0.0	8.9	0.0
2x n.i. RT 17,75	-	-	18.5	0.0	4.1
3-(3-Hydroxyphenyl)propionic acid	650.3	32.8	19.3	0.0	2.0
Vanillic acid	327.0	327.0	0.0	6.6	0.0
n.i. RT 18,96	-	-	0.0	6.5	0.0
Azelaic acid	21.3	7.1	117.9	64.9	16.4
n.i. RT 19,97	-	-	0.0	7.4	0.0
Protocatechuic acid	207.4	207.4	2.3	0.0	6.9
n.i. RT 20,28	-	-	0.0	9.5	3.8
Myristic acid	11.4	3.8	4.6	5.5	2.0
Sebacic acid	34.6	1.7	17.4	10.9	1.2
1,11-Undecanedioic acid	1874.7	1874.7	9.9	0.0	0.0
Fenpropimorph	-	-	0.0	162.6	1.1
Palmitic acid	35.8	143.7	21.1	50.6	10.0
Heptadecanoic acid	800.6	265.9	0.0	34.3	0.0
n.i. RT 26,01 (Diterpene alcohol)	-	-	0.0	17.3	0.0
n.i. RT 26,52	-	-	0.0	11.4	0.0
n.i. RT 26,61	-	-	0.0	9.4	0.0
Linoleic acid	1949.3	98.3	3.3	29.0	0.9
Oleic acid	3403.4	171.6	3.8	19.8	0.7
Stearic acid	416.8	63.3	8.5	15.6	3.8
n.i. RT 28,97	-	-	0.0	11.0	0.0
n.i. RT 29,31	-	-	3.3	19.0	0.0
Dehydroabietic acid**	222.4	70932.7	13.5	25.4	6.3
7-Oxodehydroabietic acid**	222.4	70932.7	7.7	8.6	1.3
<b>Total extractives [mg/m<sup>3</sup>]</b>			<b>771</b>	<b>671</b>	<b>351</b>
<b>Summ unidentified [mg/m<sup>3</sup>]</b>			<b>72</b>	<b>112</b>	<b>166</b>
<b>Market value<sup>1</sup> of identified compounds [EUR/1000 m<sup>3</sup> condensate]</b>			<b>221</b>	<b>374</b>	<b>56</b>
<b>Technical* value<sup>1</sup> [EUR/1000 m<sup>3</sup> condensate]</b>			<b>44</b>	<b>75</b>	<b>11</b>
<b>Market value<sup>2</sup> of identified compounds [EUR/1000 m<sup>3</sup> condensate]</b>			<b>1546</b>	<b>2447</b>	<b>548</b>
<b>Technical* value<sup>2</sup> [EUR/1000 m<sup>3</sup> condensate]</b>			<b>309</b>	<b>489</b>	<b>110</b>
<b>Weight fraction of unidentified extractives [%]</b>			<b>9%</b>	<b>17%</b>	<b>47%</b>

<sup>1</sup> Prices calculated for packaging sizes of 5 kg and for the lowest available purity of the supplier (usually greater than 98%)

<sup>2</sup> Prices calculated for packaging sizes of 5 kg and 97% purity

\* Consistent with Part 1 of the study (Rathke and Stratev 2013), only 20% of the total value of identified extractives is taken into account, to compensate for processing costs

\*\* Price of abietic acid with 75% purity was used as the basis for calculation

n.i. RT ... – not identified substance (derivate) with retention time of ... min

## Economic Feasibility

Semi- and non-volatile condensate ingredients substantially increase the economic potential of technological extractives recovery (Table 2). The market value of these substances is one to two orders of magnitude higher than the market value of only the volatile extractives. This margin could be even higher when the unidentified extractives are taken into account. Extractives recovery from spruce condensate has the highest profit potential. This is due to the high concentrations of three specific substances that at present have high (theoretical) sell prices at 97% purity: lactic acid, dehydroabietic acid, and 7-oxodehydroabietic acid. The low number of price-determining substances is advantageous from a technical point of view, but is at the same time a risk factor when evaluating profit sustainability. Extractives recovery from beech and spruce/pine condensates has 35% and 75% lower respective profit potential. However it should be considered that the purification of the non-volatile fraction of the kiln-dry condensates could be very challenging in its technical implementation since distillation cannot be used as a method for separation. Thus, the expected costs for purification of the non-volatile extractives are expected to be higher than the purification costs of the volatile extractives.

**Table 2.** Physiological Parameters and Revenue Calculation

Parameter	Beech	Spruce	Spruce/Pine
Dry density [kg/m <sup>3</sup> ]	740	430	475
Moist density [kg/m <sup>3</sup> ]	1340	774	855
Water content [kg/m <sup>3</sup> ]	600	344	380
Klin dry capacity [m <sup>3</sup> ]	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 non-volatiles* [€]	33.38	50.50	12.49
Revenue per cycle volatiles* [€]	0.06	0.23	0.52
Revenue per year [€]	435	3551	910

\* Calculated using *technical value*"

## CONCLUSIONS

Kiln-dry condensates contain a plentiful range of chemical substances that can be used for the needs of the bio-based chemistry of the future. By using Purge and Trap-GC/MS (Part 1) and MTBE-Extraction-GC/MS analytical techniques, the authors detected approximately 100 volatile and non-volatile substances in the analysed condensates. The number of detected/identified substances could be increased if LC/MS analysis were also performed since this technique does not require derivatisation of the extractives and thus substances without hydroxyl groups could also be detected. Despite the qualitative variety of extractives, their recovery from kiln-dry condensate does not seem to be profitable, due to the low concentration of the extractives and the high energy



needed for purification. An additional factor that may hinder the technical implementation of extractives recovery is the (theoretically calculated) low revenue, which does not justify investments in recovery installation and highly qualified staff.

The fraction of unidentified extractives (between 10% and 50%), however, indicates that kiln-dry condensates contain substances that are not conventional for the fossil-based chemical industry. This indicates that the potential of the condensates for the purposes of the biorefineries could be completely evaluated only after identification and consideration of the position of these chemicals in the production chain. Further steps to develop and select cost-effective methods for the isolation and purification of condensate extractives could be of great benefit for the industrial implementation of condensate recovery.

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