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Low temperature fluidized wood chip drying with monoterpene analysis

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Abstract This paper describes the drying of *ponderosa* pine wood chips at low (20°C and 50°C) temperatures using a bench-scale batch pulsed fluidizer to evaluate both volatile pine oils (monoterpenes) and moisture losses during drying. Ten monoterpenes were measured; anecdotal information on inter-tree differences in monoterpene composition indicate that while overall total monoterpene composition is similar for each tree, the ratios of α-pinene, β-pinene, δ-3-carene and limonene differ between individual trees. Results of the drying studies show normal drying curves at 20°C and 50°C; at the air flow rates used, a 20% final moisture content resulted after approximately 45 and 25 min for the two temperatures, respectively. Oil content data were highly variable but indicated that at 50°C, oils start to volatize by approximately 10 min of drying. At 20°C, oil content does not appear to change over drying time.

Monoterpenanalyse bei Niedertemperatur-Wirbelschichttrocknung von Holzspänen

Zusammenfassung Beschrieben wird die Trocknung von Gelbkiefer-Holzspänen bei niedrigen Temperaturen (20°C und 50°C) in einer Labor-Versuchseinrichtung mit pulsierender Wirbelschicht um die flüchtigen Pinienöle (Monoterpene) und den Feuchteverlauf beim Trocknen zu bestimmen. Es wurden zehn Monoterpene gemessen. Im Einzelnen zeigte sich, dass zwar die Monoterpenzusammensetzung der einzelnen Bäume im Großen und Ganzen ähnlich ist, sich jedoch die Verhältnisse von α-Pinen, β-Pinen, δ-3-Caren und Limonen von Baum zu Baum unterscheiden. Die Ergebnisse der Trocknungsversuche weisen normale Trocknungsverläufe bei 20°C und 50°C auf. Bei den verwendeten Luftdurchsätzen wurde bei den beiden Temperaturen eine Endfeuchte von 20 % nach ungefähr 45 bzw. 25 Min. erreicht. Der Ölgehalt variierte stark und es zeigte sich, dass sich die Öle bei 50°C nach ca. 10-minütiger Trocknung zu verflüchtigen begannen, wogegen sich der Ölgehalt bei der Trocknung bei 20°C nicht zu verändern scheint.

1 Introduction

This report presents the results of a study designed to better understand the relationship between moisture and monoterpene (pine oil, volatile organic compounds, VOCs; these terms are used interchangeably) removal from *ponderosa* pine slash (needles, branches, cones, bark) under low temperature (20°C, 50°C) conditions; the ultimate goal of the project is to identify economical methods to dry chipped slash in the forest. Low-temperature drying removes excess surface moisture while minimizing useful monoterpene loss. Once surface moisture is removed, remaining moisture and monoterpenes can be captured in a second, slightly higher temperature drying stage. A pulsed fluidized bed bench-scale experimental dryer was used in this study to dry chipped trunk wood and bark (selected to simulate slash). Also as part of this study, ten monoterpene compounds were analyzed from three different test trees to provide information on inter-tree pine oil composition differences.

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2 Background

2.1 Pine oil and oil extraction

This work follows from several years' study of a process that collects volatile oils (primarily monoterpenes) from *ponderosa* pine forest slash generated during either logging or forest thinning activities in National Forests. Monoterpenes dominate the VOC composition of pine slash (Beakler et al. 2007; Rupar and Sanati 2003; Lavery and Milota 2001) and are the chemical focus of this study. As pine slash consists mostly of small branches, needles and cones, the monoterpene content is slightly higher and in differing chemical proportions than that of trunk wood (Kelkar et al. 2006; Smith 2000). Monoterpenes are 10-carbon compounds that form the basis for the suite of volatile plant fragrances and are used by the plant to discourage predation (Latta et al. 2003), and serve as attractants and antifungals (da Fonseca and de Carvalho 2006). Pine needle oil is considered a more valuable essential oil because it contains more than a dozen additional monoterpenes than those found in the xylem (Kelkar et al. 2006; Krauze-Baranowska et al. 2002; Simon 1990). The most common monoterpenes in *ponderosa* pine xylem include α-pinene, δ-3-carene, limonene, β-pinene, and myrcene (Latta et al. 2003; Smith 2000). Monoterpenes can also serve as "natural" (non-petroleum-based) solvents and polymer substrates, and pharmaceuticals. The investigation of biotransformation of terpenes is an increasing area of research (da Fonseca and de Carvalho 2006). A process that recovered these monoterpene compounds would enhance utilization efforts.

However, to obtain these compounds, slash material must be processed soon after thinning operations occur. Therefore, the reason for this research was to develop a processing unit capable of operating in the forest. Additionally, slash material currently is of little commercial value as cut, costing approximately $50/acre (USDA-FS 2003) to pile and burn, and, in doing so, creating air pollution and increasing the fire risk in the forest while awaiting burning. Removal of such material is costly due to the high cost of transportation of a product that is half water. However, slash materials contain a significant energy content and can contribute to a viable national biofuel program. For example, the material could be pelletized and burned for heat in commercial boilers, or could serve as a feedstock for wood pyrolyzers. Economics of slash utilization would be enhanced if partial drying (at least) and full drying and recovery of the monoterpenes (at best) could occur in the forest.

The desired processing unit was a dryer that collected the VOC emissions (essentially, the monoterpenes) and condensed them. Initially, steam extraction was studied in the lab, as steam extraction is a common technique (personal conversation with Texarome Corp. 2004; Thomas and Schuman 1993) for obtaining valuable essential oils from plants. While steam extraction might be desirable in a forest setting (due to fire minimization), analysis of both the practical aspects of steam generation and the energy requirements for condensing the vapors (VOCs plus steam plus attendant moisture in the wood) indicated that such a unit would not be commercially viable. The next step was to evaluate air drying systems that could be adapted for small-scale, in-forest use.

2.2 Wood drying technologies

The drying process takes considerable energy to vaporize the water contained in lumber, strands and chips. Commercially the most common dryer for wood chips is the rotary-drum dryer. In this process hot ($\sim$300°C) gas enters at the wood-feed end of the drum. Although the tumbling action provides reasonable chip-gas contact, most of the chips lie in the bottom of the dryer, reducing the opportunity for effective heat transfer (O'Hagan and Smith 1986). Thus, exit gas temperatures are still higher (by $\sim$10°C) than exiting wood temperatures. However, many individual chips in the dryer see relatively high surface temperatures ($\sim$70°C) (O'Hagan and Smith 1986); these high local surface temperatures cause oxidation/thermal degradation of the volatile compounds, increasing oxygenated VOC emissions (Manninen et al. 2002; Milota 2003). Thus, high temperature drying releases the monoterpenes as VOC emissions (Lavery and Milota 2000, 2001; Rice and Erich 2006; Makowski and Ohlemeyer 2006; Wu and Milota 1999) as well as contributing to their destruction and transformation, eliminating the possibility of their recovery. In addition to the VOC emission problem, these dryers are large and not easily transportable.

For a drying system to be energy efficient, minimizing heat loss to the atmosphere is required. Low-temperature drying can provide sufficient driving force for drying if low-humidity air is available. Low-temperature and microwave processes have been developed that attempt to minimize such VOC emissions/monoterpene losses (Banerjee et al. 1998; Du et al. 2005). Du et al. (2005) found that significantly fewer VOCs (30% loss versus 90% via conventional drying) are lost during the constant temperature phase of microwave drying (the diffusion-controlled phase) which maintains low surface temperatures. Another example is the belt drier that uses lower temperatures and a very thin bed; however, the minimal contact between the drying air and the bed results in poor heat transfer (Van Loo and Koppejan 2008). Some low-temperature belt processes use a cascade of belts in an attempt to mix the chips as they fall from one belt to allow more even drying. These units, however, are also large and may require large volumes of air if their ability to effectively saturate the air is limited (Nugent 1997).

Fluidized beds are known to have very high heat and mass transfer rates. This is desirable when drying wood

chips because it allows fast, even drying and no individual chip sees the drying air long enough to be overheated. However, standard fluid beds require all the particles to be roughly the same size and weight. Additionally, the minimum gas flow rate for fluidization is higher than necessary for water removal during the diffusion-controlling phase. A patent on drying "Hog Fuel" (O'Hagan and Smith 1986) indicates the possibility of using a fluidized bed for drying wood particles. It uses a high fluidization gas rate, and dries only to about 30% moisture content, which is suitable for burning. For applications such as pelletizing, approximately 10% is required (personal communication with Rob Davis of Forest Energy Corp. 2007).

More recently pulsed-fluidized beds have been studied and indicate the ability to handle a bed of varying sized particles (Milota and Wilson 1990; Zbicinski et al. 2001; Jinescu 2004; Reyes et al. 2008). Pulsed fluidized beds are devices that supply periodic high burst air input that breaks up any bridging or channeling in the bed due to mixed particle sizes. Pulsed fluidized beds have the advantage of lower pressure drop and can use lower air flow rates without affecting the drying rate (Nitz and Taranto 2007; Reyes et al. 2008), especially during the diffusion-controlled drying phase.

Because of its ability to handle mixed, high initial moisture particle sizes, a pulsed-fluidized bed dryer operating at low temperatures (to retain monoterpenes) was selected as the appropriate test unit for this study.

3 Methodology

3.1 Pulsed fluidizer design and set-up

The device used was built from a #1 plastic 1 liter soda bottle. Inverted, the top served as the air inlet; a copper tube was inserted into the neck of the bottle with holes in the sides to direct the air outward. The bottom of the bottle (the top of the reactor) was cut off and a rubber glove was placed over the top to modulate the air pulse. 413.7 kPa laboratory air was fed to a valve system that split the air flow into two: a constant flow rate and a timed pulse via a solenoid valve. The continuous air flow rate was measured via a rotometer (100 $\mathrm{l\,min^{-1}}$). The constant air flow was not sufficient to completely fluidize the bed mass; the timed pulse was required to break any bridging that occurred. The overall effect of the system was more like a pulsed air delivery system rather than a fully fluidized system (i.e., bed mass momentarily settled between pulses). Pulses occurred at a rate of 80-$\mathrm{min^{-1}}$ at a flow rate of $\sim$120 $\mathrm{l\,min^{-1}}$.

Thermocouples were placed at the air inlet and at the top of the bottle (this point is referred to as "bed air temperature") above the maximum height that the wood chips would be thrown by the pulse. A 10.1 cm PVC tube was used as the air outlet which went through a fiberglass particulate filter; a third thermocouple was placed in the exit air line downstream of the filter. Additionally, a relative humidity meter (Kestrel 4000 Pocket Weather Tracker) was used at this location. Figure 1 shows a schematic of the unit.

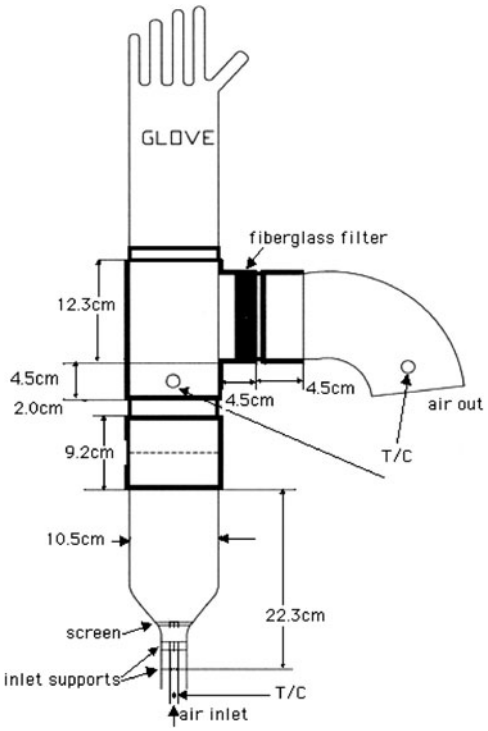


Fig. 1 Reactor schematic
Abb. 1 Schematische Darstellung des Reaktors

When heated air was used, the inlet air was passed through a $\sim$3 m long ($\sim$10 coils of $\sim$15 cm diameter) 10 mm diameter copper tube that was placed in a 4 liter hot water bath (90–100°C) prior to entering the valve system.

3.2 Experimental methods

3.2.1 Tree collection, chipping and preparation

Ponderosa pine trees <10 cm in diameter (at base) and $\sim$3 m high were taken from the NAU Centennial Forest. Trees were cut with a handsaw $\sim$8–10 cm above the ground level, delimbed and were chipped immediately after harvesting. Only bark and trunk (no needles or cones) were used. A specially-modified DR 18 hp wood chipper/shredder was used to reduce final particle size; the chips were collected in a plywood box to minimize chip loss. Chips were then sieved through a 9.5 mm and a 4.75 mm sieve. The chips passing the 9.5 mm sieve were retained for further processing. The coarse (>9.5 mm) and fine (<4.75 mm) fractions were discarded. Chips were bagged and labeled in a sealable bag (Ziploc). Subsamples ($\sim$100 g) of the batch were

Table 1 Experimental matrix
Tab. 1 Versuchsplan

Inlet air T, °C	Time, min	# replicates
20	20	3
	25	3
	33	7
	45	3
50	5	3
	10	3
	18	3
	30	3

placed in tared pans and placed in the oven for moisture content analyses.

3.2.2 Laboratory experiments

All experimental work (unless otherwise noted) was performed in the NAU Environmental Engineering laboratory. For each set of experiments, the following protocol was used.

1. Place 100 +/− 1 g of sample in the reactor.
2. Start the clock timer; open airflow and air pulse valves.
3. Record inlet/bed/outlet air temperatures and outlet humidity at the start of run and at five-minute intervals (first replicate only); record at end of run for other replicates.
4. Upon completion of a run, pulse air through empty chamber twice to remove remaining particles and disassemble and clean fiberglass filter.
5. A portion of the sample is placed in a glass vial (packed full) and placed in the freezer for shipment for oil analysis. Place an additional ∼50 g of sample in a tared pan and place in the oven for moisture content analysis.

Table 1 shows the experimental matrix used; controlled variables were inlet air temperature and run time as shown. All "cold" runs (20°C) were done using two different trees over a two-day period; all "heated" runs (50°C) were done using a third tree over a one-day period.

3.3 Analytical methods

3.3.1 Moisture content of chips

Chip moisture content (MC) analyses were performed in accordance with Sect. 6 (Method B—Oven Drying) of ASTM D 4442-92 (1997) e 1, *Standard Test Methods for Direct Measurement of Wood and Wood-based Materials*.

3.3.2 GC/MS analysis for monoterpenes (oil content)

The University of Idaho (UI) Forest Products Laboratory performed oil analysis. Wood samples were shipped refrigerated or frozen to the UI lab. Analysis consisted of monoterpene extraction followed by GC analysis as follows:

1. Green wood chips (2 g) were accurately weighed into a tared scintillation vial to which 10 ml of extraction solvent (methanol/diethyl ether (1:4) containing naphthalene as an internal standard (50 µg ml^{-1})) was added and the vial orbitally shaken for 4 hr.
2. An aliquot portion of the extract (2 ml) was transferred to a GC vial for analysis by GC-MS (PolarisQ, Thermofinnigan) in the electron impact mode (McDonald et al. 2004). The volatile components were separated on a ZB-1 (30 m × 0.25 mm, Phenomenex) capillary column with a temperature program of 40°C (2 min) to 200°C (10 min) at 5°C min^{-1}. GC-MS data was analyzed using Xcalibur v2 software (Thermo).
3. A synthetic standard solution comprising available standard monoterpenes (α-pinene, β-pinene, limonene, δ-3-carene, γ-terpinene, myrcene, bornyl acetate, terpinolene, α-terpineol, and camphene) was prepared to determine response factors relative to naphthalene. Compound identifications were made by matching mass-spectra, retention time, and comparison with published Kovats Indices (Adams 2004).
4. The MC of the green chips was determined after removal of excess solvent and oven drying at 105°C for 16 h. The yield of monoterpenes was then calculated on a dry wood basis.

4 Results and discussion

Table 2 shows the initial moisture and oil contents of the material used. The greater variation in MC for the 20°C runs is due to the use of two different trees (only one tree was used per day to avoid drying losses overnight; the 20°C runs required two days to complete). Additionally, a procedural error on days 1 and 2 resulted in obtaining only one sample for MC analysis from each tree (thus increasing the variation), and one oil sample from tree 1 The variation in oil content for tree 3 is half that of tree 2.

Figure 2 shows the reduction in MC for the 20°C and 50°C trials. Regression analyses performed on each data set indicate that moisture loss follows trends as reported in the literature (Motta Lima et al. 2004), with good correlation as indicated by the R^2 values.

Figures 3a and 3b show the relationship between inlet air wet bulb temperature, bed temperature, bed air temperature and chip MC during the runs. Wet bulb temperatures

Table 2 Initial conditions of trees used
Tab. 2 Ausgangsbedingungen der untersuchten Bäume

Tree	Temp, °C	N[a]	Chip moisture, % (dry)	N[a]	Oil, g kg^{-1}	CV[b] (oil), %
1	20	1	95.5	1	5.7	
2	20	1	118.6	5	5.2 +/− 2.2	0.42
avg 1&2	20	2	107.1 +/− 16.4	6	5.3 +/− 2.0	0.38
3	50	5	131.9 +/− 1.2	5	5.1 +/− 1.1	0.22

[a] N = number of samples

[b] CV = coefficient of variation

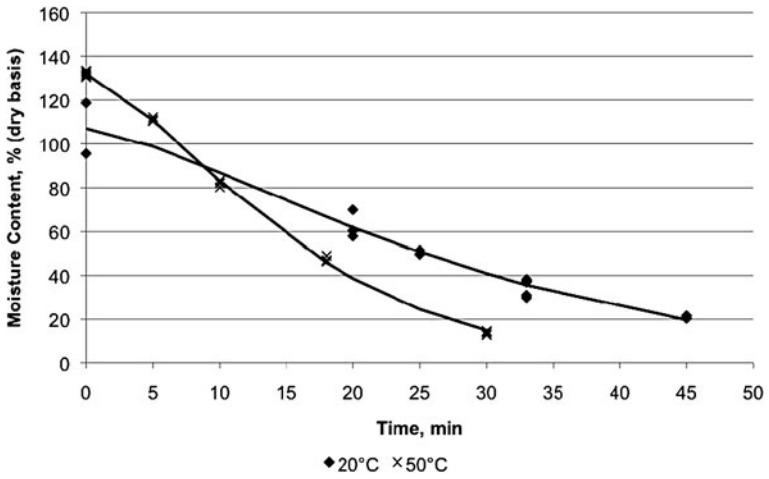


Fig. 2 Reduction of moisture with time for 20°C and 50°C runs
Abb. 2 Feuchteverlauf in Abhängigkeit der Zeit bei einer Trocknung bei 20°C und 50°C

were corrected for barometric conditions in Flagstaff, AZ and are 3.94°C for the 20°C (actually 20.1°C averaged) runs and 14.3°C for the 50°C (actually 46.9°C averaged) runs. For "bed air temperature", the actual temperature measured was the air temperature directly above the bed. "Bed" temperatures were calculated based upon the known excess air (beyond that needed for saturation), as the volume of pulse air was higher than needed (15% and 31% of the total airflow for the cold and heated runs, respectively). In the absence of excess air, the bed temperatures should approach that of the inlet air wet bulb temperature (at least during the convection controlled drying phase).

Figure 4 shows the reduction in oil content (g oil—kg dry wood^{-1}) for the 20°C and 50°C trials. Two data points were eliminated from the data set as outliers due to abnormally high oil contents (far higher than starting oil contents and any reported values in the literature), likely due to analytical errors: one of the 45 min (20°C) runs (22.1 g kg^{-1} oil) and one of the 33 min (20°C) runs (13.0 g kg^{-1}). Two other data points in the replicate set of the 33 min (20°C) runs also had high oil contents, but they were within 1 and 2 standard deviations of the average, and were not removed from the data set. The 33 min (20°C) runs were replicated seven times to determine variation due to the process. However, it must be noted that the 20°C runs utilized 2 different trees; the 33 min (20°C) runs had two samples with tree 1 and four runs with tree 2. Thus, the variation ($CV = 0.39$) i s actually due to the variation due to the tree used rather than to the process itself. (Table 2 shows an oil content average CV of 0.38 for trees 1 and 2; it is 0.22 for tree 3.) Variation in oil content data is much greater than that in the MC data shown in Fig. 2 above.

Despite the variation, trends can be seen. As Fig. 4 shows (the trendline past 30 min is an estimate), with the use of heated (50°C) air, volatile pine oil losses begin to occur by 10 min of drying. A t-test comparing 0 min and 5 min indicated no difference ($t = 0.98$) while comparing 0 min and 10 min indicated a difference ($t = 0.02$). The loss of oil is not linear, but better fits an exponential curve as shown in the figure. The lack of correlation with time for the cold (20°C) air runs (a linear model was the best fit at $R^2 = 0.006$) indicates that substantial pine oil losses are not seen while drying at this temperature.

For the cold trials, the temperature of the chips remained below 14.4°C, with no significant loss of monoterpenes. It can be assumed that at the beginning of the heated trials, the temperature of the chips was near wet bulb (~14.3°C). By ten minutes their temperature had risen to 16.7°C and monoterpenes began to be removed. Similar behaviour of terpene removal occurring only after the cooling effects

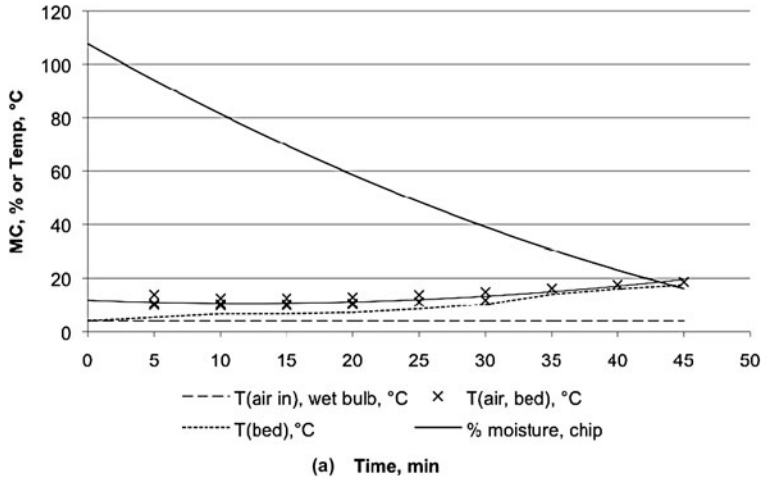
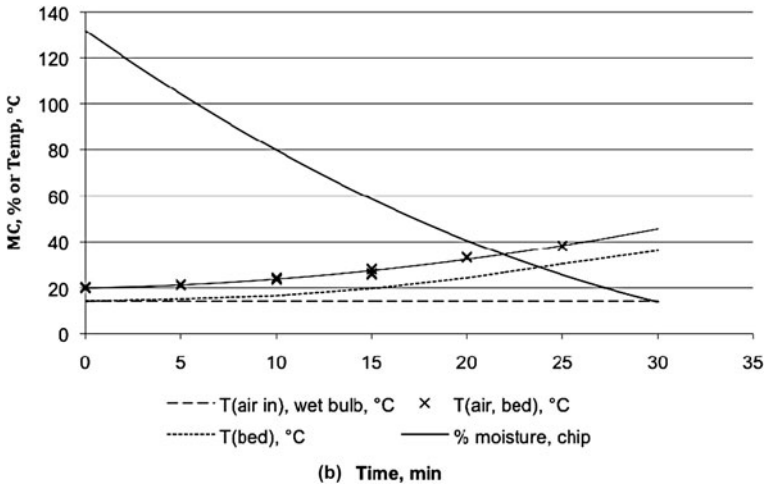


Fig. 3 Change in inlet air wet bulb, bed air, and bed temperatures and moisture content for (**a**) 20°C runs and (**b**) 50°C runs
Abb. 3 Verlauf der Feuchttemperatur der einströmenden Luft, der Lufttemperatur über dem Bett (air, bed) und der Betttemperatur (bed) sowie Verlauf der Holzfeuchte während der Trocknung bei (**a**) 20°C und (**b**) 50°C



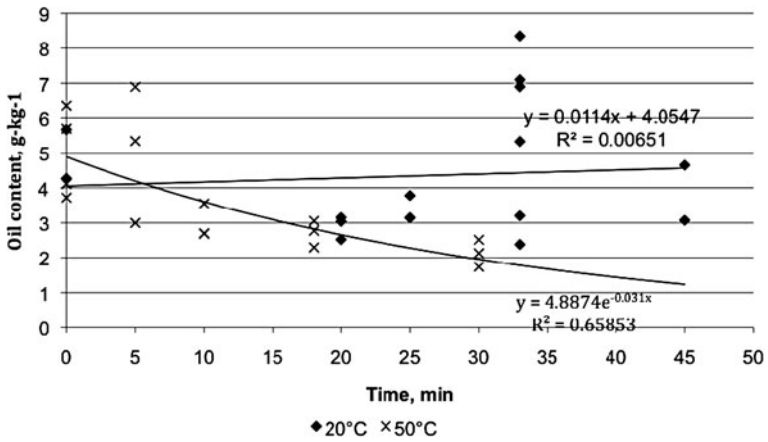


Fig. 4 Reduction of oil content with time
Abb. 4 Abnahme des Ölgehalts in Abhängigkeit der Trocknungsdauer

of surface water evaporation during the convection drying phase has been observed by Banerjee et al. (1995). This phenomenon cannot be explained by the effects of vapor pressure alone, as the ratios of the vapor pressures of the monoterpenes to water differ by less than 0.1% over that temperature range. One possible explanation is that the terpenes are adsorbed to the surface of the wood fibers in the chips, and that desorption is initiated at temperatures between 14.4°C and 16.7°C, for the given conditions.

Even though only three trees were used in this study, the data indicate, if only anecdotally, inter-tree variation of terpene composition. It was observed that terpene ratios did not change during drying, indicating that the various terpene species generally volatilized at similar rates. This is

Table 3 Selected terpene ratios for the test trees
Tab. 3 Ausgewählte Terpenanteile der untersuchten Bäume

	β-pinene/α-pinene	δ-3-carene/α-pinene	limonene/α-pinene
Tree 1 avg	0.008 +/− 0.001	0.002 +/− 0.000	0.202 +/− 0.027
Tree 2 avg	0.878 +/− 0.134	0.510 +/− 0.065	0.209 +/− 0.039
Tree 3 avg	0.029 +/− 0.009	0.336 +/− 0.036	0.033 +/− 0.012

unsurprising given the similar boiling points of the various terpenes (Perry and Green 1984). Table 3 shows the four most prevalent monoterpenes as ratios to α-pinene (the most prevalent). The other six monoterpenes included in the analysis were campene, myrcene, γ-terpinene, terpinolene, α-terpineol, and borneyl acetate; these were present in much lower quantities. All three trees appear to be quite different than one another in composition; tree 1 was from a different part of the forest while trees 2 and 3 were from the same area. However, the overall total concentration of all "pine oils" between the trees was similar (as shown above).

5 Recommendations and conclusion

This work indicates the relationship between moisture and oil losses during wood chip drying. It appears that surface water can be removed while retaining most of the oil (monoterpenes) in the wood; these monoterpenes could be removed/captured in a later processing stage, if desired. Longer time periods are needed to dry at low temperature, which increases equipment size. However, pulsed fluidization allows for lower air flow rates, especially during the diffusion controlled drying phase, suggesting a possible sizing and economic advantage.

One limitation of this work is the unknown actual bed temperatures during the experiments. In this work, actual bed temperatures were estimated as indicated by Figs. 3a and 3b. A second limitation is that the pulsed air flow rate used was higher than the minimum needed for effective fluidization. Additionally, air flow was held constant and was not reduced when surface water was gone (once drying became diffusion limited), so the bed temperature rose very fast as the heat went to heating the wood instead of vaporizing water. It is recommended in future work that bed temperature be controlled and the air pulse rate be adjusted to minimize energy expenditure.

To reduce inter-tree oil content and consistency variation, mixing chips from several trees would be an option. However, to do this would require many more trees, as fresh material is required for each day's runs.

One primary recommendation is the use of pulsed fluidizer drying of ground wood slash material. Slash is defined as needles, cones and buds, small branches and bark. The addition of needles (which are difficult to grind because of the long narrow shape) will substantially change the particle size distribution structure in the woody mix, and, with that, the pertinent pulsed fluidizing conditions. Also, oil removal from slash materials may differ from that of the bore wood and bark used in current processes. In order to make the most of such material, an efficient means of handling/drying needles is required.

A second recommendation is the use of multiple small staged pulsed fluidizers with the capability of removing the completely dried smaller particles while larger undried particles move to further drying stages, as indicated in O'Hagan and Smith (1986). Oil capture can be added at the appropriate stage in the drying process.

Finally, the adsorption-like behavior of the monoterpenes relative to the wood fiber should be more fully investigated to confirm the phenomenon and to determine the appropriate sorption model and ranges for the adsorption constants.

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