Water balance in Hydrosol Production Via Steam Distillation: Case Study using Lavandin (*Lavandula* x *intermedia*).

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Essential oils that derive from aromatic plants are typically obtained by steam distillation (Simon, 1990), a simple and relatively inexpensive process in which the essential oils are removed from the plant by a stream of water vapor, and then both phases are separated easily (Charles and Simon, 1990; Alkire and Simon, 1992; Vieira et al., 2000). During distillation, part of the essential oil components become dissolved in and remain in the distillation water and the 'product' is called hydrosol, which is also known as the distillate water (Rajeswara Rao et al., 2003). At present, there are no legal definitions of a hydrosol per se nor grades and standards set forth by industry and the scientific community, nor are there specifications of natural hydrosols as defined by international standardization boards and associations internationally recognized such as ISO, FEMA or ANFOR. Typically, the distillate water, which includes minor amounts of the essential oil that remains in the water fraction, is highly aromatic and its composition is often different than the prime essential oil, which forms as a separate and distinct layer following distillation and represents the essential oil of commerce from a plant species. The amount of essential oil remaining in the distillate water, and the composition of the oil found in the distillate is a function of many factors including the distillation process and the plant species.

The oxygenated components, among the most valuable components relative to organoleptic properties, are more soluble in the distillation water. The distillate water is typically discarded after the prime oil is removed, unless the commercial operation directs the distillate water into a redistill or undergoes a secondary distillation to recover remaining essential oil in the distillate water. The essential oil that is captured from the secondary distillation process typically has a different composition than the prime oil. In the extraction of some oils, such as in lavender, the resulting hydrosol or distillate water has a very pleasant aroma and is recognized as having commercial applications.

Lavender (*Lavandula angustifolia*) is recognized as producing a very high quality aromatic oil similar yet distinct from the oil of lavandin (*Lavandula x intermedia*). Higher essential oil yields are obtained from lavandin plants compared to lavender, and as such, use of lavandin is found both as a replacement oil to lavender, as an adulterant to lavender oil, and in its own right as a distinct aromatic commercial essential oil (Renaud, Charles and Simon, 2001). International specifications as set forth by the ISO are available for the array of lavender essential oils.

The objective of this research was to evaluate the water balance in the steam distillation process for the production of hydrosols. We sought to examine the relative contribution of the water from the plant material and from the steam that is used in the extraction process. A secondary objective of this research was to examine the composition of lavandin oil and of the hydrosol over time through the entire distillation process and to monitor volatile oil compositional changes over time along with other indicators; that information is presented separately from this report which focuses on the water source of the hydrosol.

Material and methods

The plant material. Fresh lavandin (*Lavandula* x *intermedia*) inflorescences were harvested on September 20th, 2003 by PurpleHaze Lavender (1-888-852-6560, 180 Bell Bottom Road Sequim, WA 98382), ground shipped directly to the New Jersey Agricultural Experiment Station, Snyder Research Farm and received on September 22nd. Lavandin was received in cardboard boxes and kept in the refrigerator (4°C) until distillation and processing. Three sub-samples from each load were used to determine the water content and dry weight biomass of the plant material using a moisture analyzer (Mettler Toledo HB43 Halogen Moisture Analyzer) set at 105°C.

Steam distillation and Lavandin hydrosols. The lavandin inflorescences were subjected to steam distillation (Alkire and Simon, 1992) (Figs. 1-4). For each load, fresh material (44-49 kg) was placed into the distillation vessel (Fig. 2, Table 1-2). The first load was conducted to set-up and establish the procedure and determine the processing parameters, and two replications (second and third loads) were conducted as the main study. The results are presented as the average of these two replications (Tables 1-3, Figs. 5-6).

Steam was supplied through a manifold pipe into the bottom of the vessel from a high-pressure boiler (at the end of the boiler the steam was set at a constant pressure of 8 psi). The steam is routed upward through a false-bottom perforated plate to the plant material being extracted (Fig. 2). The steam removes the essential oils and both leave the vessel near the top via a 7.6 cm diameter flexible pipe and are carried to a water-cooled, parallel-piped multitubular aluminum condenser, that is mounted vertically (Fig. 3). The essential oil (lighter than water) and the water condensate (hydrosol) were then separated in a 10 liter Pyrex florentine flask (Fig. 1) that was cleaned prior to and after each use.

The water pumped into the system (in kg.) was measured using a flow meter (GPI, Great Plains Industries, Inc. Wichita, KA) that was attached at the water inlet (before the boiler); the distillate water (kg of hydrosol), its pH (Accumit AB15 pH meter) and essential oil accumulation (kg) were measured every ten minutes.

For setting up the procedure, the first distillation was allowed to run for 300 min (Fig. 6); however in the main study the distillation was finished at 160 min after no further accumulation of oil was observed (Fig. 5).

At the end of each distillation, the amount of water remaining in the boiler, the distillation vessel, and in the plant material was also weighed and recorded.

The loss due to evaporation was estimated by subtracting the total water that went through the system from that remaining after distillation (Table 2).

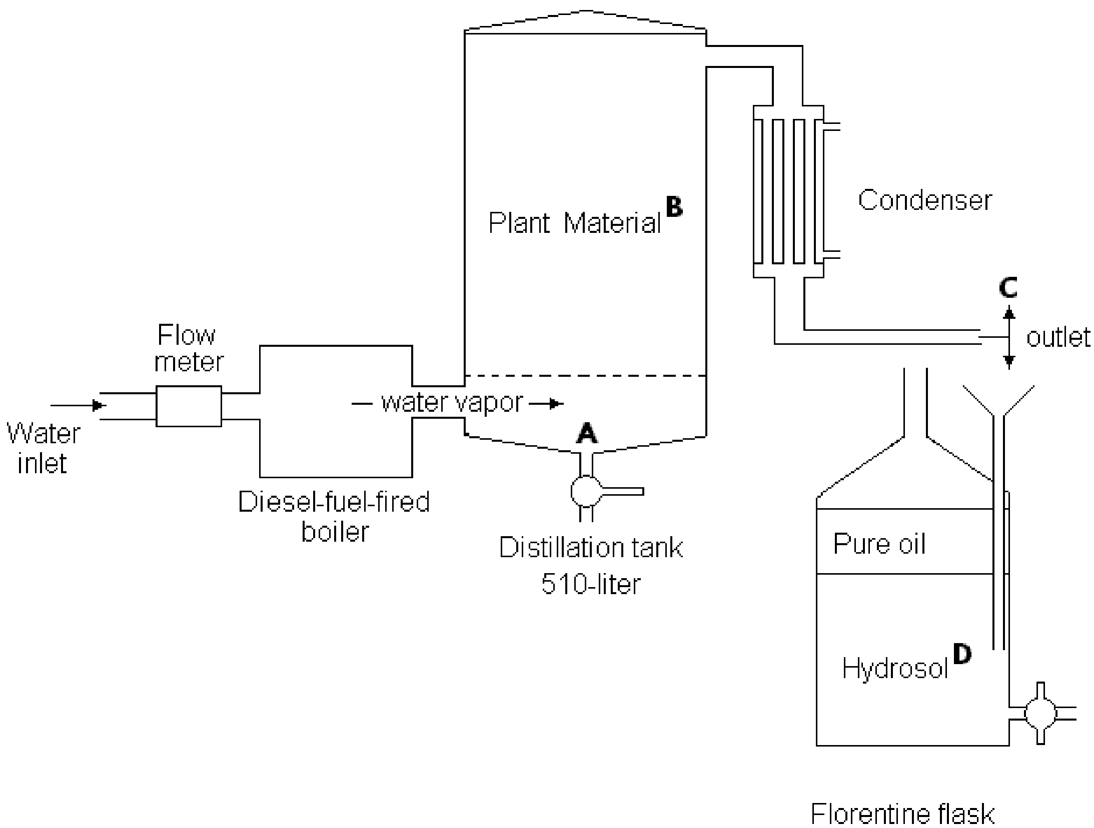

Figure 1 – Schematic diagram of the steam distillation unit used in this work and water flow during and post-distillation. At the termination of the distillation, the water could be accounted for from that amount found in the bottom of the distillation tank (A), in association with the plant material (B), evaporated into the atmosphere (C), and in the hydrosol or distillated water (D).

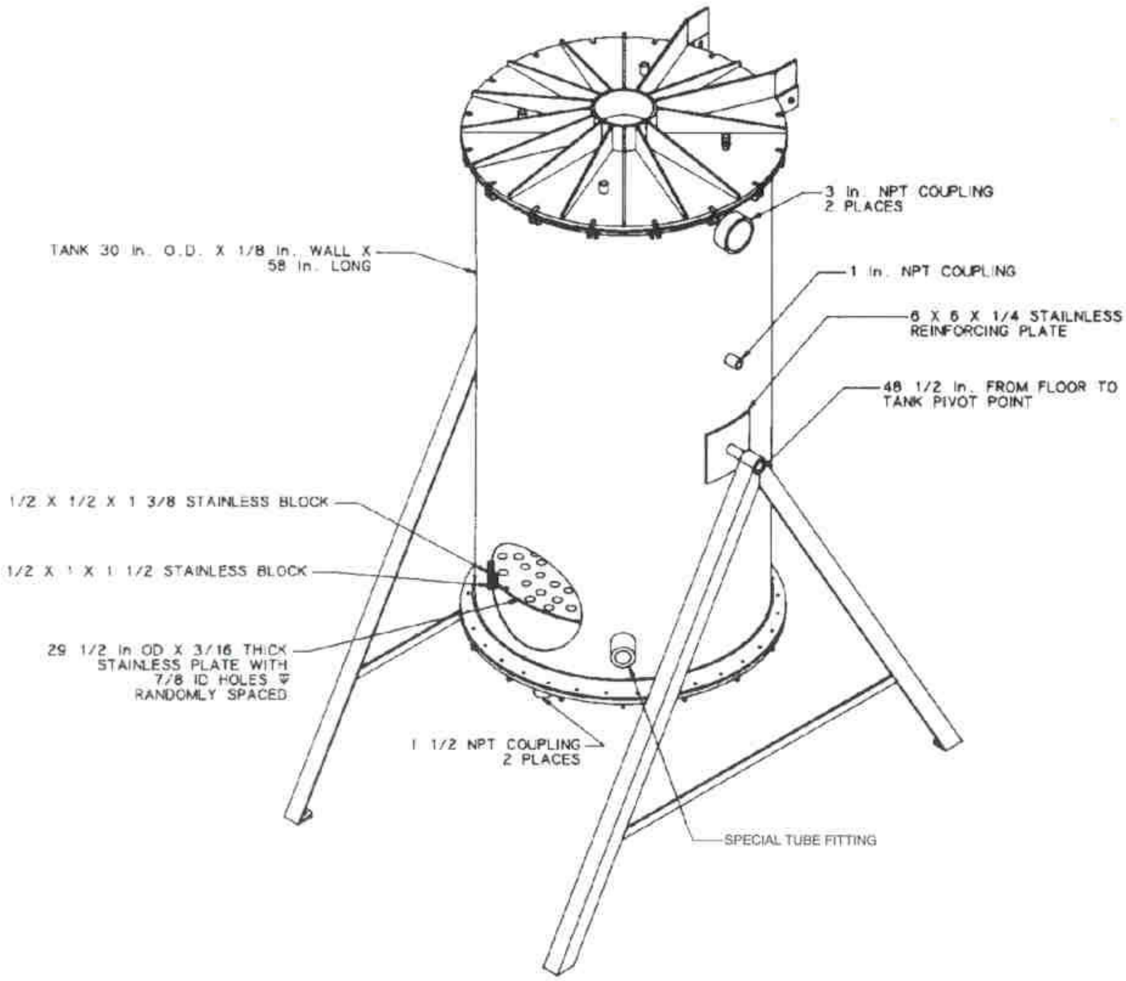

Figure 2 - Assembly drawing of the stainless steel 510-liter steam extraction vessel (from Alkire and Simon, 1992).

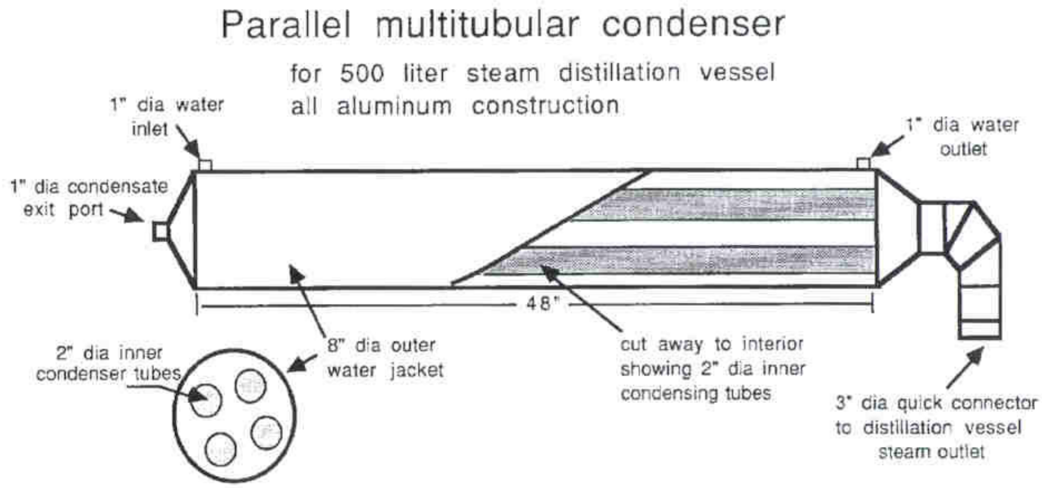

Figure 3 - Design of the aluminum multitubular condenser for the distillation unit, which is mounted vertically on the trailer, adjacent to the steam vessel (from Alkire and Simon, 1992).

Figure 4 - The portable steam distillation unit used in the present work. The distillation tank (left) is tilted to facilitate loading/removal of plant material. The system has a series of pressure regulators to monitor and maintain pressure. Photo taken while preparing the unit for this study, Pittstown, New Jersey.

Results and discussion

The process began with the loading of 50 kg of water into the boiler. The water was then pumped into the system in linear fashion at 0.61 kg per min (Fig. 5). A few minutes after distillation commenced (8 min), the production of the distillate began that was composed of the essential oil plus the distillate water (or together as a hydrosol) that also increased linearly at 0.53 kg per min. This slope was slightly lower compared with the water inlet (0.61 kg/min). The water input and hydrosol production are constant through the process (Fig. 5). According with the regression, the equation for the water inlet and hydrosol production were the following:

water inlet, y (kg) = 0.61(kg/min)x + 50.28(kg), R^2=0.9929

hydrosol outlet, y (kg) = 0.53(kg/min)x - 4.06 (kg), R^2=0.9937

The essential oils began to accumulate and the first measurement was made at 30 min yielding 0.75 kg of essential oil (Fig. 5). The essential oil accumulation also increased linearly until 100 min and then stabilized around 1.59 kg (Fig. 5).

During the first preliminary run during which the parameters of the main study were to be established, we observed that after 140 min of actual distillation no further accumulation of essential oil was detected (Fig. 6). As a result, we terminated the distillations of the main study at 160 min, a period of time after the distillation process with this species was found to be complete because no additional essential oil could be observed coming from the condenser nor was any further essential oil accumulating in the Florentine flask, and because further increases in the hydrosol volume would dilute the essential oil dissolved in the water. The distillation process was almost finished at 110 min during both the first preliminary run (Fig. 6) as well as during the main study run (Fig. 5), when more than 98% of the oil was extracted.

The fresh plant material, composed mainly of water (52%), plant dry biomass (45%), and essential oil (3.4%), contributed 24.1 kg of water to the system (Table 1), while the tap water feeding into the boiler contributed 128 kg (Table 2).

At the termination of distillation, 128 kg of water was pumped into the system, 19.9 kg remained in the boiler and thus was not used, and 82 kg of hydrosol was obtained (at 160 min). The remaining water ended up in the bottom of the distillation vessel (26 kg), at least 12 kg were absorbed by the plant material, and the rest lost due to evaporation (9 kg) (Table 2).

Although it was not possible to determine the absolute contribution of water from the steam and plant distilled into the hydrosol, our results show that a maximum of 29% of the water in the hydrosol could come from the plant material, assuming that all the water from the plant material (24.1 kg) ended up in the hydrosol (82.1 kg) (Table 3).

However, this assumption is likely to be an over-estimate because at the pressure and temperatures used in the distillation, not all water that is in the fresh plant material would be distilled out of the plant by the end of the distillation and re-condensed into the hydrosol.

After loading the system with water (50 kg), the water intake increased constantly at 0.61 kg/ min while the hydrosol outlet increased at 0.53 kg/min. This relationship showed that at every minute, up to 87% (0.53 kg) of the incoming water (0.61 kg) ended up in the hydrosol, suggesting that the water in the hydrosol came chiefly from the inlet water. This observation is also supported by the fact that the plant material absorbed water.

The contribution of the plant water to the hydrosol will also depend on the duration of the distillation and the pressure used in the distillation process. While setting up the process, the system was allowed to continue until 300 min, so hydrosol accumulation increased at the described rate (0.53 l/min) producing 197.3 kg of hydrosol. In this case, the maximum contribution of the plant water to the hydrosol would be only 12%. When producing hydrosols private companies could maximize or continue to increase the production of hydrosol by simply continuing the distillation time beyond the time frame when all volatile aromatic oils have already been exhausted from the original plant materials.

Further studies that determine the absorption of water over time by the plant material and running the unit to further confirm the water balance equation without the plant material will be needed to support these assumptions. In the future, deuterated water could also be used to determine the exact amount of the contribution of the plant material to the hydrosol.

The pH of the hydrosol at the beginning of production was 5.4 and decreased through the process till pH 3.8 at the end of distillation (Fig 5).

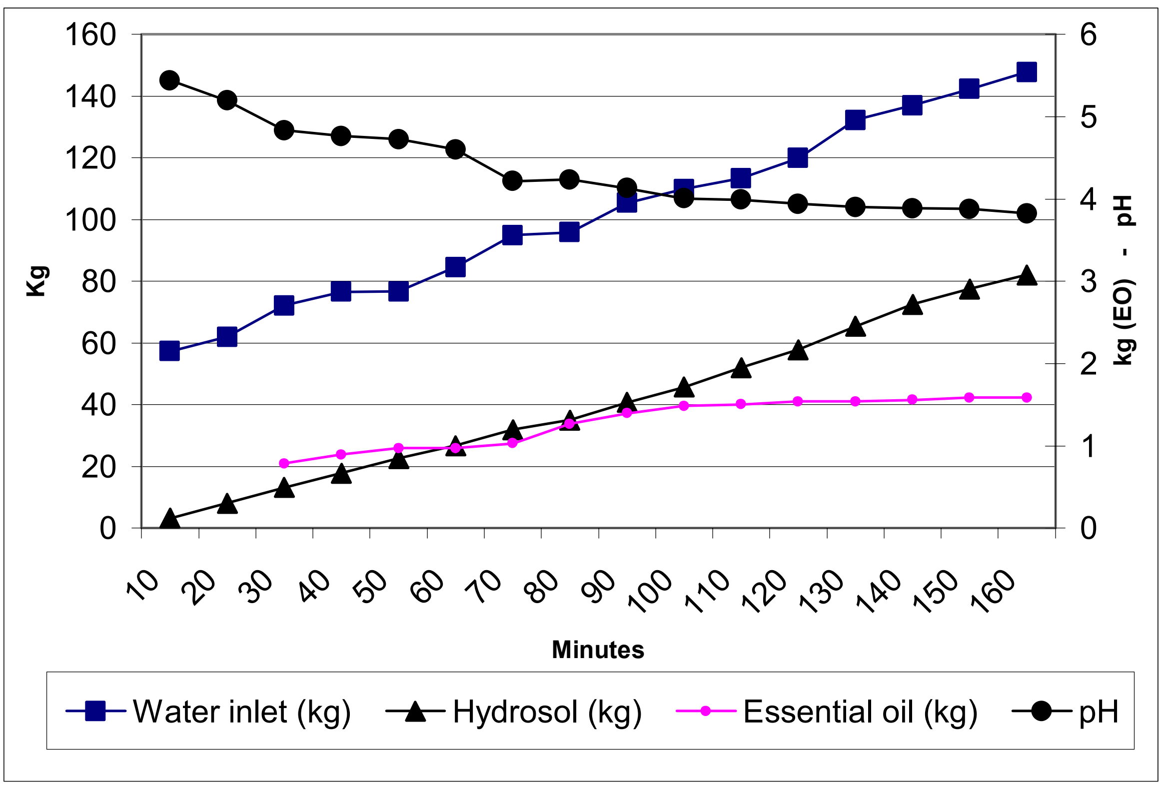

Figure 5 – Time-course of steam distillation of lavandin (*Lavandula* x *intermedia*).

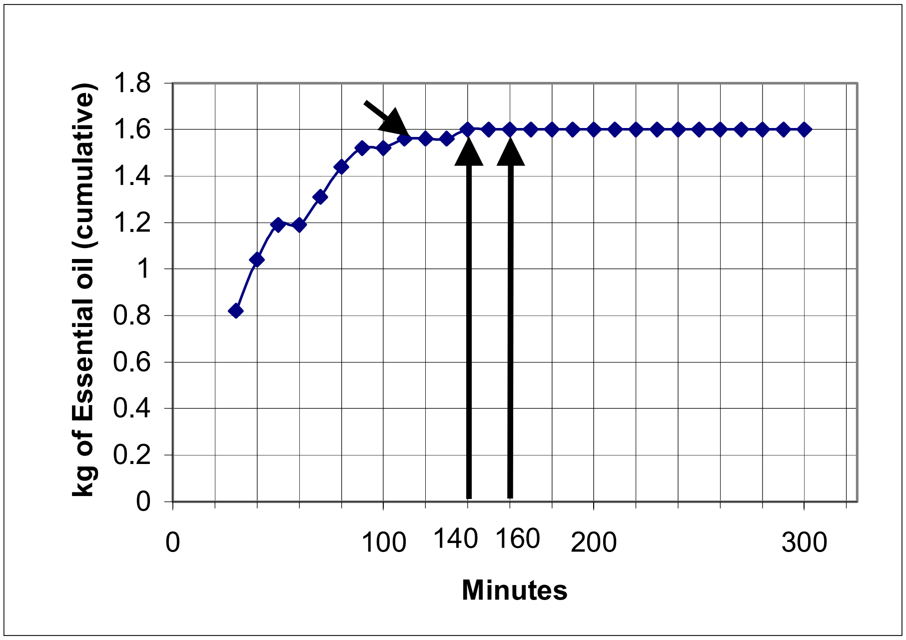

Figure 6 – Time-course of essential oil accumulation during the distillation of lavandin (*Lavandula* x *intermedia*). Note that about 98% of the oil was extracted at 110 min (oblique arrow), and that no further accumulation of oil was observed after 140 min. In these studies, the distillation was terminated 160 min.

Table 1 - Dry weight, essential oil and water content of the fresh load before distillation.

	Absolute (kg)	Relative (%)
Dry weight	20.8 ± 0.9[1]	44.7
Essential oil content	1.59 ± 0.02	3.4
Water	24.1 ± 1.2	51.8
Fresh load (kg)	46.5 ± 2.1	100

[1] - Average of two replications ± standard error.

Table 2 - Gross weights of the fresh load and water at the beginning and at the end of steam distillation.

Gross weight (kg)	Beginning	End
Fresh load	46.5 ± 2.1[1]	56.9 ± 4.7
Tap water used	147.9[2] ± 31.1	0
Boiler	0	19.9 ± 1.4
Bottom of tank	0	26.0 ± 9.5
Essential oil	0	1.59 ± 0.02
Hydrosol	0	82.1 ± 9.5
Total	195.3	186.3[3]

[1] - Average of two replications ± standard error.
[2] - The total water that was effectively used to distill the essential oils was 128 kg since at the end of distillation 19.9 kg of water remained in the boiler.
[3] - The loss of weight due to evaporation was 9 kg.

Table 3 - Contribution of the water from inlet and plant material to the hydrosol.

Distribution of water in the hydrosol	Absolute (kg)	Relative (%)
Coming from plant material (max.)	24.1	29.4
Coming from tap water (min.)	58.0	70.6
Total	82.1	100

Conclusions

Our results showed that the water in the hydrosol came from both the plant material and from externally applied water pumped into the system, which was used as the source of steam in the distillation process. The results with this species showed that the external water contributed a minimum of 71% of the total water volume in the captured hydrosol, while a maximum of 29% came from the lavandin plant. Although it was not possible to determine the absolute amount of the water that came from plant, this study suggests that the contribution from the fresh floral material would account for less than 29% of the total volume since it is unlikely that all of the water from the plant would be distilled out of the plant and into the hydrosol product.

For this study, we removed the prime oil. In future studies, the prime oil could be removed and later added back into the hydrosol to examine the impact on a hydrosol, and also the distillation time could be well extended beyond the time frame used in the distillation of the oil for which only the prime oil is sought coupled with an examination of the aroma impact of the hydrosol. Additional studies that aim to define a hydrosol, as well as distillation runs during which the impact of differential steam pressure, temperature and other parameters of the distillation process can be further examined, are now needed with this and related *Lavandula* species.

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