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Distillation of tea-tree *(Melaleuca alternifolia)* oil. I. Establishment of basic parameters and standard conditions for a test distiller and evaluation of two prototype distillers

[Penyulingan minyak tea-tree *(Melaleuca alternifolia).* I. Pewujudan parameter asas dan syarat piawai bagi penyuling uji dan penilaian dua penyuling prototaip]

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Key words: water-steam distillation, steam distillation, test distiller, prototype distillers

Abstrak

Suatu penyuling uji dengan muatan maksimum 20 kg telah direkabentuk, dibina dan digunakan untuk menilai kecekapan dua penyuling prototaip untuk penyulingan minyak tea-tree. Parameter asas seperti tempoh masa asas, parameter tambahan per unit ketinggian muatan dan pengisi lag telah dianggarkan. Syarat piawai seperti kadar aliran air dan kandungan minyak telah ditentukan dengan menggunakan penyuling uji ini. Hasil daripada kajian tentang tempoh masa keseluruhan pengekstrakan dan minyak yang dihasilkan menunjukkan bahawa penyuling prototaip yang menggunakan wap yang sederhana kering daripada alat penyejat (penjana wap) lebih baik daripada penyuling prototaip yang menggunakan sistem takungan air.

Abstract

A test distiller with a maximum charge capacity of 20 kg was designed, fabricated and used to evaluate the efficiency of two prototype distillers for distilling tea-tree oil. The basic parameters such as the basic time, increment parameter per unit charge height and the lag factor were estimated. Standard conditions such as water flow rate and oil content for the distillation of tea-tree oil were determined using the test distiller. Results of total extraction time and yield of oil indicated that the prototype distiller using fairly dry steam from an evaporator (steam generator) with a steam manifold was superior over the prototype distiller using a water bath system.

Introduction

Tea-tree or *Melaleuca alternifolia* is an essential oil crop currently being planted commercially in Australia. Tea-tree oil is used primarily for its antiseptic and wound cleansing properties, and the therapeutic claims such as antiseptic treatment of cuts and abrasions, stings and bites as well as pimples and mild acne have been approved in Australia (Priest 1993).

Commercial variety tea-tree seeds were brought in from Australia and planted in Serdang, Selangor and Beseri, Perlis in early 1993. Adaptability studies of tea-tree to our

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tropical climate such as environmental factors, planting density, harvesting index, insect and weed control, crop management and distillation techniques were carried out. Results showed that tea-tree is adaptable to the Malaysian climate and oil yields are higher than the corresponding yields of plants grown along the north coast of New South Wales, the major production centre in Australia (Hunter 1997). The quality of the locally produced tea-tree oil was reported to meet the Australian Standard AS 2782-1985 (Ahmad et al. 1997).

There are three basic types of commercial distillation of essential oils, i.e. water distillation, water-steam distillation and steam distillation (Guenther 1972). The principles of hydro-distillation of oils from plant materials are known, but they have to be applied in many different ways to get the best results from widely different herbs. It was reported by Denny (1990a) that no satisfactory rigorous tests have been carried out to determine the best way of distilling tea-tree oils. Variables such as distiller type (water, water-steam and steam), fuel type, steam speed, lag factor, virtual height and extraction time must be tested to find the distillation method which returns the best yields and acceptable quality of oil at the minimum operating cost.

This study was to establish the basic parameters and standard conditions for the test distiller. The information is not available locally and we have to develop our own set of data which are more practical to local conditions. The test distiller was then used to evaluate two prototype distillers which were designed, fabricated and tested for distillation of tea-tree grown in Serdang and Beseri. This is also a study to scale-up the distillation of charge material from 20 kg to 400 kg.

Materials and methods

Design and fabrication of test distiller

A test distiller was designed and fabricated as shown in *Figure 1*. The test distiller was a water-steam distillation type made of stainless steel. The distillation pot was double walled with 385 mm diameter, 640 mm maximum charge height and 120 mm maximum water level in the water bath. The maximum weight of tea-tree leaves and twigs (charge capacity) for this test distiller was 20 kg. Two 1-kW electrical heaters were immersed in the water bath. A stainless steel condenser was constructed using nine condenser tubes (18 mm diameter), one condenser tube surrounded by eight condenser tubes, in a cylindrical shell measuring 125 mm in diameter and 600 mm long. Running tap water was used to cool the condenser. The distillate was channelled into an oil separator made of glass with the condensed water directed back into the water bath of the distillation pot. This process is known as cohobation (Guenther 1972).

Determination of basic time and increment parameter per unit charge height

The test distiller described above was used to determine these parameters. The bucket experiment (Denny 1990b) with some modifications was used in these determinations. Exact 1-min fractions of the distillate were taken at precise intervals of 5 min throughout the distillation using a 1-L measuring cylinder. The volumes of water and oil in each fraction were measured. The volumes of water and oil were also estimated for the time the measuring cylinder was removed from the condenser outlet by using the mean volumes of water and oil between measurements. Finally, the total oil delivered by distillation was also recorded to obtain the correction factor between the calculated volume of oil and the actual volume of oil collected.

In these determinations, two charges of different weight, 10 kg and 15 kg, were distilled under similar rate of steam flow with the two 1-kW heaters turned on and distilled for 150 min. The bucket experiment was used, and plots of oil produced against distillation run time and oil produced against total water passed were made to determine

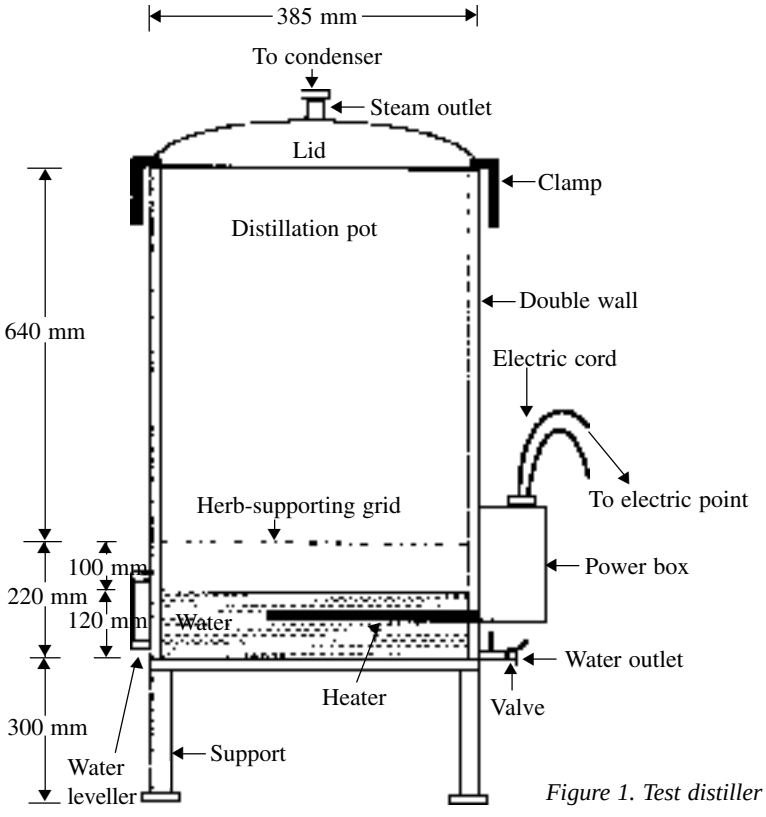

Figure 1. Test distiller

the estimated virtual exhaustion (EVE) oil content. The value at 95% EVE from these plots was used to derive the extraction time and actual water flow rate. The EVE at 95% was regarded as the commercial end-point (Denny 1990b).

Determination of lag factor

The lag factor for tea-tree oil distillation was obtained using the test distiller. This determination was carried out using two charges of the same weight (10 kg) with similar oil yield, but distilled at substantially different rate of distillate flow as described by Denny (1990b). The determination was done with one set of charges distilled using one heater and the second set of charges distilled using two heaters. The two sets of charges were distilled for 150 min and the bucket experiment described above was followed. The 95% EVE values were used from the plots of oil produced against time and oil produced against water passed to derive the extraction time and actual water flow rate.

Design and fabrication of prototype distiller 1

Prototype distiller 1 was a water-steam distillation type made of stainless steel with a water bath located at the bottom of the distillation pot (*Figure 2)*. The distillation pot was insulated with brick wool and surrounded with mild steel sheet. The diameter of the distillation pot was 1 000 mm and the maximum charge height was 1 250 mm. The charge material was suspended in the distillation pot by means of a herb-supporting grid. The length between the herb-supporting grid and the bottom of the water bath was 250 mm. The water bath was filled with water to a depth of about 150 mm which could be observed through the external glass water level indicator. The water bath was then heated by two high pressure stoves using liquefied petroleum gas (LPG) as fuel. The condenser is a

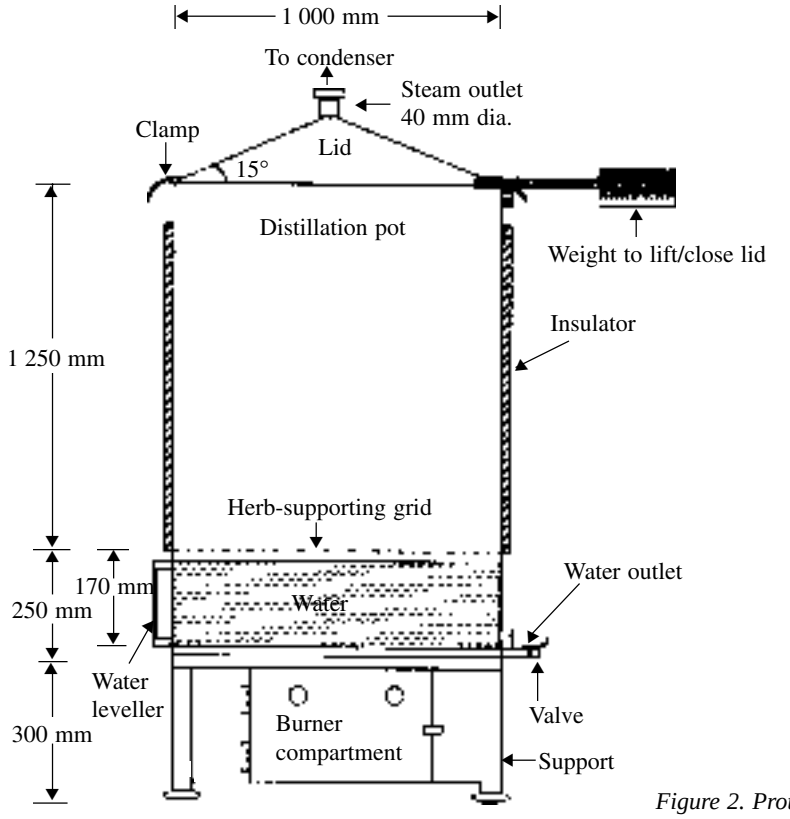

Figure 2. Prototype distiller 1

vapour-in-tube type constructed with 12 condenser tubes (18 mm diameter) arranged in a hollow square pattern in a cylindrical shell of 400 mm diameter and 825 mm long as shown in *Figure 3.* Water from a nearby pond was pumped into the condenser to cool it. The distillate was channelled into a stainless steel oil separator *(Figure 4)* designed for oils lighter than water. The condensed water was directed back into the water bath located at the bottom of the distillation pot as described in the test distiller.

Design and fabrication of prototype distiller 2

Prototype distiller 2 was a steam distillation type made of stainless steel (*Figure 5)*. A separate evaporator (steam generator), as shown in *Figure 6,* operated at atmospheric pressure was connected by a steam pipe to a steam inlet manifold located at the bottom of the distillation pot. The steam inlet manifold was to ensure an even distribution of steam at the bottom of the distillation pot. The charge material was supported by a herb-supporting grid located 200 mm from the bottom of the distillation pot. The maximum charge height for this distiller was 1 500 mm. The condenser was a vapour-in-tube type constructed from nine condenser tubes (19 mm diameter) arranged in a solid square pattern in a cylindrical shell of 146 mm diameter and 1500 mm long *(Figure 7)*. Five coolant baffles were evenly spaced in the cylindrical shell to create turbulent flow on the coolant side. Water from a nearby pond was pumped to cool the condenser. The stainless steel evaporator (*Figure 6)* was constructed with nine parallel pipes (38 mm diameter) at the bottom and placed on a support attached to a flue. The condensed water from the oil separator *(Figure 4)* was directed back into the evaporator through the

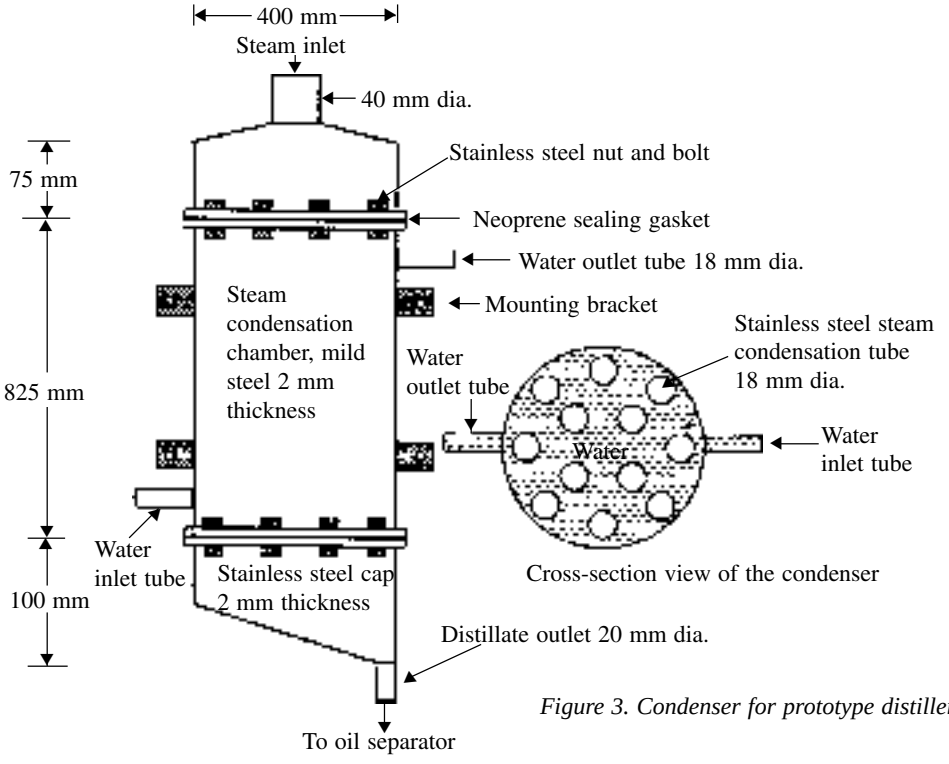

Figure 3. Condenser for prototype distiller 1

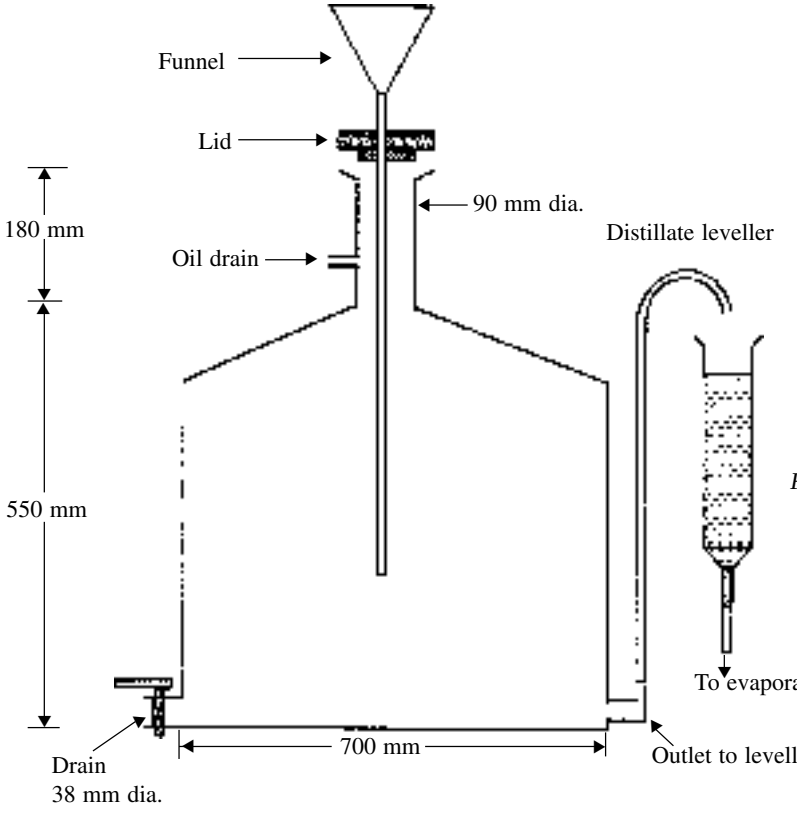

Figure 4. Oil separator

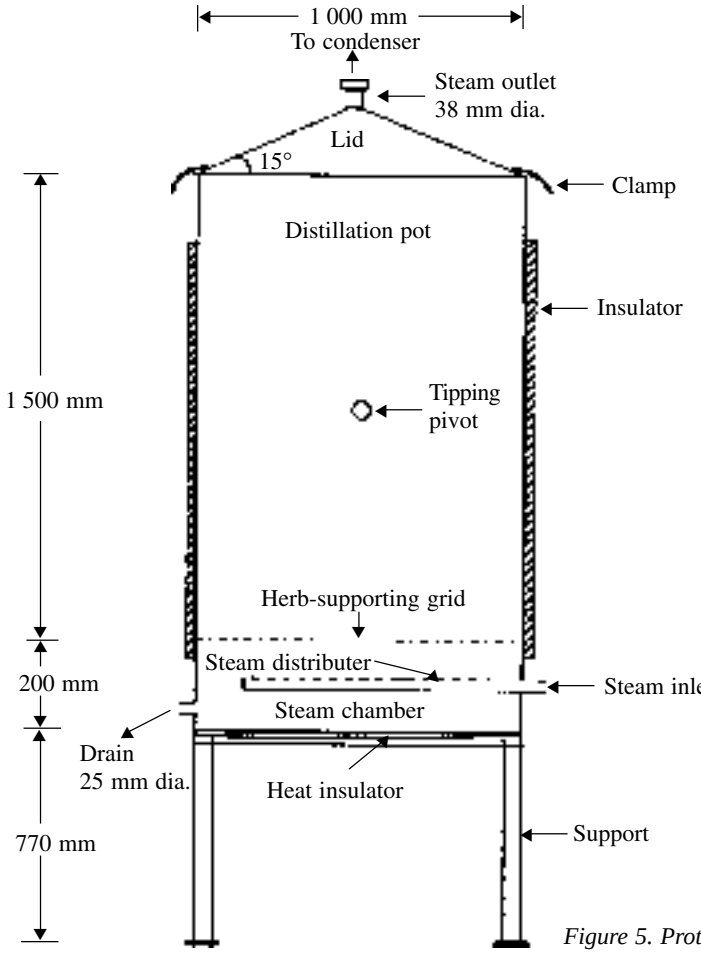

Figure 5. Prototype distiller 2

evaporator feed water tank. The parallel pipes of the evaporator were heated by two high pressure stoves using LPG as fuel. The complete distillation system for distiller 2 is shown in *Figure 8.*

Preparation of charge material for distillation

Tea-tree from the experimental plot was harvested using a chain saw, weighed and transported to the distillery. The tea-tree was then passed into a chipping machine to reduce the leaves, twigs and branches into smaller pieces and transferred into the distillation pot.

Measurement of parameters during distillation

The heating time for prototype distiller 1 and prototype distiller 2 was recorded. This was the period from the first admission of the steam into the distillation pot until the point when the steam passed through the top of the charge and the distillate could be taken as flowing steadily at the intended flow rate. The bucket experiment described earlier for the test distiller was followed and distillation was carried out for 120 min. The EVE was obtained from the plots of oil produced against distillation run time and oil produced against water passed. The 95% EVE was used to derive the extraction time and actual water flow rate. Four distillation runs were made for each prototype.

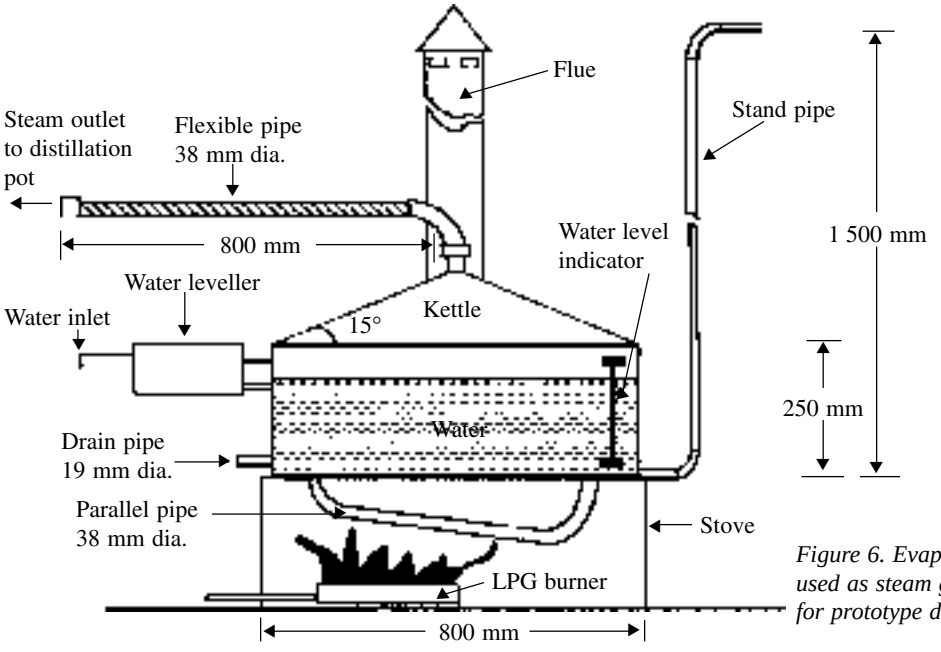

Figure 6. Evaporator used as steam generator for prototype distiller 2

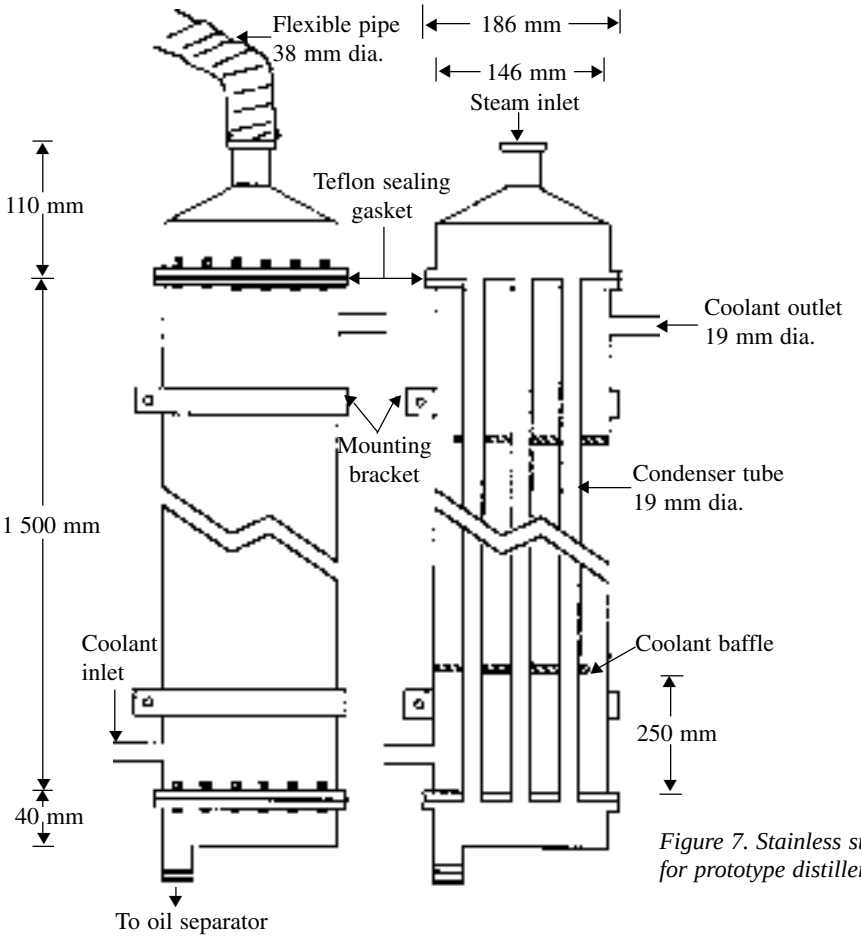

Figure 7. Stainless steel condenser for prototype distiller 2

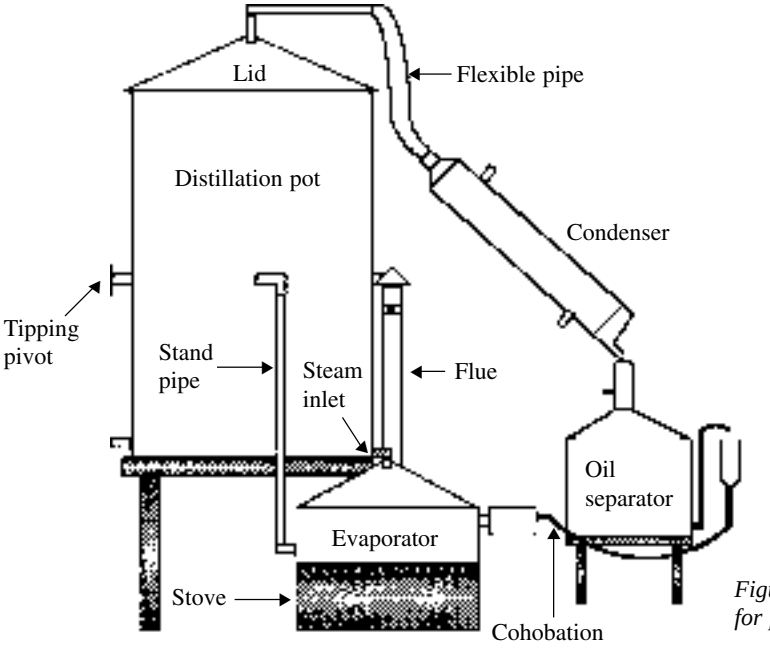

Figure 8. Distillation system for prototype distiller 2

Statistical analysis

The data collected were subjected to statistical analysis using analysis of variance as outlined in the SAS system (SAS Institute Inc. 1985).

Results and discussion

Determination of basic time and increment parameter per unit charge height

Tea-tree oil is a subcutaneous oil and is found between the palisade layers of very tough leaves. The hydro-diffusion hypothesis is applied in its distillation (Denny 1990b). In this hypothesis, the oil from a single gland diffuses to the leaf surface and the time for the complete exhaustion from that single gland is called the basic time. This is an empirical value and the calculated or observed extraction time (T) is related to the basic time (t) and overall charge height or virtual height (H) by the equation:

$$T = t + H.dt \qquad \text{Eqn. 1}$$

where

dt = increment parameter per unit charge height of which the basic time is proportional to the tea-tree oil content per kilogram of charge material

H = overall charge height or virtual height based on the number of tea-tree layers in the distillation pot. Each unit thickness of standard charge material (tea-tree) layer contain the same amount of oil

Graphs of the volume of oil produced against distillation run time and volume of oil produced against volume of water passed are shown in *Figure 9* and *Figure 10* respectively. The EVE was determined using the asymptotic line on each curve extrapolated to the x-axis and the 95% EVEs were used to derive the extraction time and the actual volume of water passed. The parameters derived from these graphs are shown in *Table 1*. The adjusted extraction time was taken as T, after correction for the ratio between the actual water flow rate and the mean water flow rate.

The basic time (t) and the increment parameter per unit charge height were calculated using equation 1 by substituting the adjusted extraction time (T) for both charges, the actual charge height for the

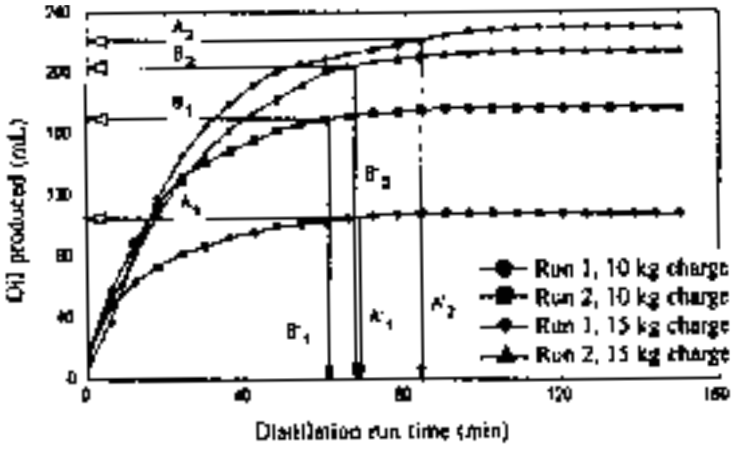

Figure 9. The oil produced against distillation run time showing the 95% EVE (A_1, A_2, B_1 and B_2) and the extraction time (A'_1, A'_2, B'_1 and B'_2) for the respective runs

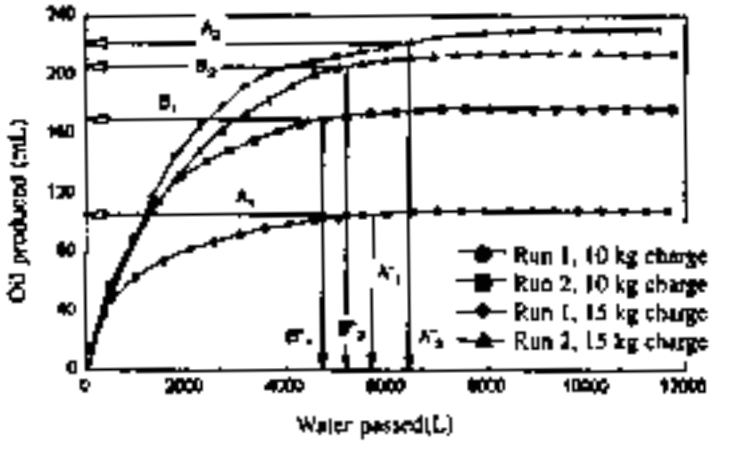

Figure 10. The oil produced against the water passed during distillation as per the 95% EVE (A_1, A_2, B_1 and B_2) and the actual volume of water passed (A''_1, A''_2, B''_1 and B''_2) for the respective runs

Table 1. Parameters obtained from test distiller with a cross-section area of 0.1164 m^2 at atmospheric pressure

Parameter	Values at 95% EVE	
	*10 kg charge	15 kg charge
Mean oil produced (mL)	137.0	213.8
Mean water passed (mL)	5 200.0	5 796.5
Actual water flow rate (mL/min)	80.0	76.0
Mean water flow rate (mL/min)	78.1	78.1
Standard water flow rate (mL/min/m^2)	671.0	671.0
Mean observed extraction time (min)	65.0	76.0
Adjusted extraction time (min)	66.6	74.2
Actual charge height (cm)	32.0	47.0
Virtual charge height (cm)	**	50.0
Oil content (mL/cm layer)	4.28	4.28
Standard oil content (mL/cm/m^2)	36.78	36.78

*The 10 kg charge was taken as the base charge for the calculation of standard water flow rate and standard oil content (Denny 1990b)
**Being the base charge value was not calculated

10 kg charge and the virtual charge height for the 15 kg charge obtained from *Table 1*. The two equations obtained are as follows:

66.6 = t + 32.0 (dt) Eqn. 2

74.2 = t + 50.0 (dt) Eqn. 3

Solving for t and dt,

t = 53.0 min

dt = 0.4247

It is now possible to calculate the extraction time for any other distiller for distilling tea-tree using the values of basic time (t) and the increment parameter per unit charge height (dt) obtained above. Immediate applications of these parameters are in the designing of new commercial distillers and testing of existing distillers for tea-tree or other closely related herbs.

Determination of lag factor

Under the hydro-diffusion hypothesis, any factor that may bring about an increase in oil diffusion to the surface unit layer will require a specific factor of increase in the rate of steam flow. In practice, fluids do not increase their rate of flow along restricted channels in the full proportion of the accelerating force. A lag factor for the forces resisting diffusion must be included in any factor of increase in oil diffusion to the surface unit layer. The equation relating rates of steam flow to the speed with which a subcutaneous oil can be distilled is given by Denny (1990b) as

$Z = R.Y^{2/3}$ Eqn. 4

where Z = factor of change in speed of oil recovery per unit time

Y = factor of change in steam speed over the herb surfaces

R = lag factor for the forces resisting diffusion

An empirical relation between lag factor (R) and factor of change in steam speed (Y) for tea-tree oil distillations was obtained as shown in *Figure 11*. The curve was smoothened using negative exponential smoothening. The curve applies for the base flow (Y = 1) at 671 mL/min/m^2. This curve will be used to estimate the lag factor (R) in any other distillers for distillation of tea-tree. A typical application is in the determination of the calculated extraction time (T_C) of new distillers to be designed or existing commercial distillers using equation 1. The values obtained will be corrected for $Y^{2/3}$ and R derived from equation 4.

Evaluation of prototype distiller 1 and prototype distiller 2

The mean volume of oil produced against the distillation run time for distiller 1 and distiller 2 is shown in *Figure 12*. The standard error difference of the two curves overlapped for the first 36 min and both plots started with virtually a straight line. This indicated that the distillation for both prototypes were similar for the first 36 min with constant distillate ratio (oil to water). Both plots curved away from linear as the oil content of the distillate declined in the

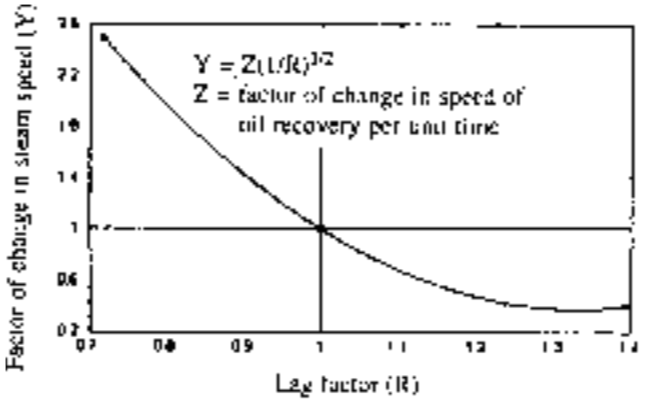

Figure 11. Empirical relation between lag factor of change in steam speed (Y) for tea-tree oil

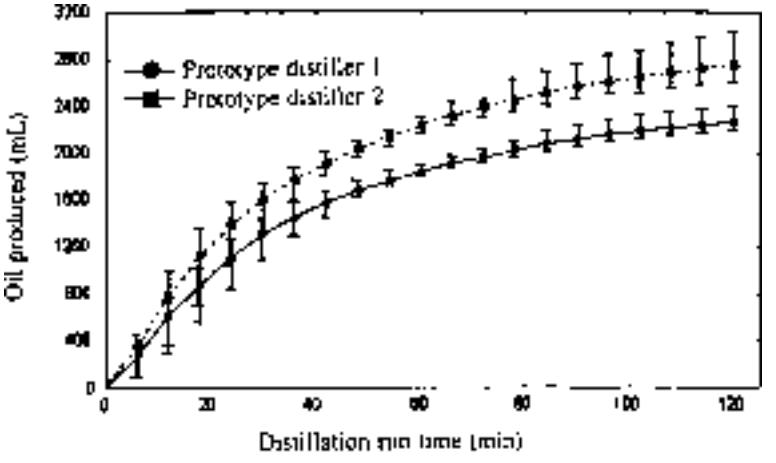

Figure 12. The mean oil produced against the distillation run time for prototype distiller 1 and prototype distiller 2. Values are means of four replicates ± standard error difference

Table 2. Oil produced, virtual height at 95% EVE, extraction time, percentage error and heating time of tea-tree oil distilled using prototype distiller 1 (P1) and prototype distiller 2 (P2) with a mean charge weight of 368 kg

Distiller type	Oil produced at 95% EVE (mL)	Virtual height (cm)	Extraction time (min)		Error (%)	Heating time (min)	Total extraction time (min)
			Observed	Calculated			
P1	2 165.5b	75.0b	97.9a	90.2a	–8.7a	69.3a	166.6a
P2	2 671.5a	92.4a	99.9a	89.0a	–9.2a	52.7b	148.3b

Mean values with same letter(s) within a column are not significantly different according to LSD ($\alpha = 0.05$)

Table 3. Water flow rate, lag factor and $Y^{2/3}$ of tea-tree oil distilled using prototype distiller 1 (P1) and prototype distiller 2 (P2) with a mean charge weight of 368 kg

Distiller type	Water flow rate (mL/min/m^2)	Lag factor	$Y^{2/3}$
P1	579.6b	1.0386a	0.9067b
P2	739.7a	0.9737b	1.0672a

Mean values with same letter(s) within a column are not significantly different according to LSD ($\alpha = 0.05$)

latter part of the run and the standard error differences of both plots were no longer overlapping. This indicated that the overall distillation run for both prototypes were different. The mean volume of oil produced at 95% EVE for both prototypes differed at similar charge material weight *(Table 2)*.

Mean comparison of the efficiency of the distiller types are shown in *Table 2* and *Table 3*. The observed extraction time (T_o) was derived from the plots at 95% EVE and the calculated extraction time (T_c) was obtained using equation 1 and corrected for $Y^{2/3}$ and R. The mean observed extraction time (T_o) for prototype distiller 1 and prototype distiller 2 was 97.9 min and 99.9 min respectively. This was comparable to T_o of 90–120 min by Australian distillers of tea-tree oil (Colton and Murtagh 1990). The percentage error between T_c and T_o was computed to indicate the validity of the distillation (Denny 1990b). It was found that the T_c, T_o and percentage error, $[(T_c - T_o)/T_c]100$, were not significantly different. The mean percentage error between T_c and T_o for both prototypes was considered small (<10% absolute) and the distillations could be regarded as acceptable within the experimental limits. Ideally, the error between T_c and T_o should be approaching 0%. Too large a percentage error is an indication that a distillation is not running correctly (Denny 1990b).

The mean heating time and total time for prototype distiller 2 was significantly shorter compared with prototype distiller 1. This was desirable as a shorter extraction time means less fuel cost. This indicated that prototype distiller 2 was more efficient than prototype distiller 1. The shorter heating time was perhaps due to the design of the evaporator of prototype distiller 2 which used nine tilted parallel pipes (38 mm diameter) located at the bottom of the evaporator. The parallel tubes were heated at the higher end by two high pressure stoves fuelled by LPG. This resulted in faster boiling of water in the parallel tubes which then rose up into the reservoir, forcing the cold water to enter the lower end of the parallel tubes. This brought about faster circulation of the water from the reservoir into the parallel tubes resulting in faster heating of the evaporator. The other advantage of this evaporator design was the possibility of using an oil burner employing other sources of fuel such as kerosene or diesel. The evaporator provided fairly dry steam at atmospheric pressure into the distillation pot through an inlet manifold or simply steam distillation for prototype distiller 2. Prototype distiller 1 had a different design in which the steam was generated at the bottom of the distillation pot. It was observed that the water in prototype 1 took a longer time to boil compared with the evaporator of prototype 2 at the same rate of LPG consumption. Wet steam was generated or simply water-steam distillation for prototype distiller 1. The oil produced at 95% EVE for prototype distiller 1 was less than that of prototype distiller 2 for similar charge material weight. This was perhaps due to the steam generated in prototype distiller 1 containing more moisture than prototype distiller 2. The lower oil yield in prototype distiller 1 might be due to the flooding of the herb surface with water to the point of run-off.

The excess water may be originated from the aqueous plant cells or from cloud particles deposited by excessively wet steam (Denny 1990a). Discoloured water was observed in the water bath of prototype distiller 1 as an evidence of run-off happening in the distiller. There was no evidence of run-off in prototype distiller 2 by observing the drainage outlet of the distiller. The lower oil yield in prototype distiller 1 could also be contributed by the hydrophilic effect, a phenomenon where oil-coated water droplets escaped through the top of the charge, passed the oil separator and flowed into the water bath of prototype distiller 1. In principle, hydrophilic loss is proportional to the wetness fraction of the steam and to the cross-section area of the top charge (Denny 1990a). Prototype distiller 1 was operated with significantly wet steam compared with prototype distiller 2 and the hydrophilic effect was expected to be more pronounced in prototype distiller 1.

The prototype distillers were evaluated based on the standard conditions (standard water flow rate and standard oil content) at atmospheric pressure established by the test distiller as shown in *Table 1*. It was observed that the water flow rate, lag factor and $Y^{2/3}$ for prototype distiller 1 and prototype distiller 2 were significantly different. The mean water flow rate of prototype distiller 1 was less than the standard condition and prototype distiller 2. The mean water flow rate of prototype distiller 2 was higher than the standard condition and prototype distiller 1. The faster water flow rate generated by the evaporator of prototype distiller 2 resulted in an increase in $Y^{2/3}$ and a reduction in the lag factor (R) relative to the test distiller. It was expected that the observed extraction time (T_o) for prototype distiller 2 was to be faster than that of prototype distiller 1. This did not occur probably because the water flow rate for prototype distiller 2 was not fast enough to maintain the temperature gradient between the bottom of the distillation pot and the point of vaporisation on the herb surface. In principle, the oil's rate of evaporation from the herb surface is proportional to the magnitude of the

temperature gradient and the rate that the oil receives heat and boils away declines proportionately with increasing charge height (Denny 1990b). The virtual height (H) for prototype distiller 2 was higher than that of prototype distiller 1. This was probably the reason why the observed extraction time (T_o) and the calculated extraction time (T_c) for prototype distiller1 and prototype distiller 2 were not significantly different. A water flow rate above 1 000 mL/min/m^2 would probably give sufficient temperature gradient and hence improve the extraction time of prototype distiller 2. This means that the evaporator of prototype distiller 2 must be heated using a fuel with a much higher thermal energy level.

Conclusion

The establishment of a test distiller to obtain the basic parameters, i.e. basic time, increment parameter per unit charge height and lag factor, and the standard conditions at atmospheric pressure, i.e. the standard water flow rate and the standard oil content, was important as a prerequisite to evaluate existing commercial distillers or to design new distillers for distillation of tea-tree or other related herbs.

Prototype distiller 2 was found to be superior to prototype distiller 1 in terms of oil recovery and total extraction time. The use of fairly dry steam for prototype distiller 2 resulted in an increase in oil yield compared with prototype distiller 1. This is probably due to the loss of oil through run-off and hydrophilic effect was minimised in prototype distiller 2. Provision in the design of prototype distiller 2 which allows the use of other fuels such as kerosene and diesel for heating the evaporator is an advantage over prototype distiller 1. This means that higher flow rate of water can be achieved for prototype distiller 2 by using a higher thermal energy fuel. Furthermore, the use of steam from high pressure boilers can be incorporated for further study with the objective of getting the best oil yield at a minimum operating cost.

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