Selection of the raw material for efficient distillation of cinnamon leaf oil

C P Rupasinghe, K D N Weerasinghe and K M S Kodikara
Department of Agricultural Engineering, Faculty of Agriculture, University of Ruhuna, Mapalana, Kamburupitiya

Abstract
A laboratory experiment test was conducted in the Cinnamon Research Station, Thihagoda to assess the best resting period for cinnamon leaves prior to distillation, to maximize the leaf oil output and its quality. There were two treatments (leaves with twigs and without twigs) with three replicates. Samples were subjected to distillation at daily intervals for 5 days, to detect the optimum resting time prior to distillation; quantity and quality of the leaf oil obtained in each treatment were assessed. Clevenger’s arm method was used to determine the oil yield, moisture content was determined by the Dean and Stark method and GLC was used to analyze oil quality. While the average oil yield of leaves with twigs was 1.80%, leaves alone had an oil yield of 2.32%, which was significantly (P=0.05) higher by 28%. The maximum oil yield was observed after a resting period of 5 days in both treatments. The average oil yield of leaves on the 5th day was 2.63% and that of leaves with twigs was 2.32%. At this stage, the moisture content of leaves and leaves with twigs were 18% and 28% respectively. The eugenol content in leaves without twigs was 10.8% higher than that of leaves with twigs. Cinnamaldehyde content of leaves without twigs was lower by 160% than that of leaves with twigs.

Keywords: cinnamon, oil distillation, leaf oil, eugenol, cinnamaldehyde

Introduction
Sri Lanka has been known for spices from times immemorial. The growing and processing of spices provide cash income to a wide range of rural Sri Lankans, particularly small holders. In 2001, the value of Sri Lankan spice exports was Rs.6,098 million, equivalent to 1.4% of total exports, 7.3% of agricultural exports, and more than half of the tea, coconut and rubber (Central bank report 2001).

Cinnamon (Cinnamon verum) is an indigenous spice crop in Sri Lanka. It belongs to the family Lauraceae. Cinnamon and Sri Lanka are synonyms in the world spice trade. It is one of the most important export agricultural commodities, suitable for investment by the government as well as the private sector due to its unique position in catering to over 70% of the total requirements of the world demand. Sri Lanka is the largest producer of cinnamon in the world, accounting for about 60-70% of the global production. It is the third largest export crop in the Agriculture export sector in the country.

The total area under cinnamon cultivation in Sri Lanka is about 25000 ha. Most of the cinnamon lands are concentrated in southern Sri Lanka. Quills account for the highest income among cinnamon products. Quillings, featherings, chips and cinnamon oil contribute the rest. Two types of cinnamon oils, namely bark oil and leaf oil are exported from Sri Lanka. Income generated from bark oil production is much higher with respect to the quantity exported due to the high price per unit of the product. In the year 2000, the average price of cinnamon leaf oil was Rs 662/Kg. This was very low compared to bark oil. However since the cost of production of leaf oil is comparatively low, the industry can be run at a profit (Central Bank Report, 2000).

Leaf oil is a product of the leaves and twigs left in the field after sticks were collected for the quills production. Therefore the cost of raw material is negligible and the only costs encountered are the cost of labour and the cost of distillation. It is reported that many people engaged in this industry gave up their attempts at production in recent years due to insufficient profits. Therefore the continuity of the industry could be helped by...
improving the technology to obtain higher oil yields with good quality. This target could be achieved by adopting an efficient distillation procedure and selecting suitable raw materials for distillation. Steam or water-distillation may be employed for the production of cinnamon leaf oil. The yield and quality of the product are dependent upon a number of factors. These include the botanical variety, geographical origin of the leaf material and the agronomic practices of the leaves and the distillation practices used (Senanayaka, 1998).

In Sri Lanka, cinnamon leaf oil is generally produced by steam distillation of dried leaves on rather simple, but nevertheless effective, equipment. At one time, two types of oil were produced which possessed different properties. In the Negombo area, north of Colombo, where the finest cinnamon quills are produced, the leaf oil was lighter in color and had a low Eugenol content (60-65%), while the oil produced in the Ambalangoda and Matara districts of the extreme south was darker and contained 75 to 80% of eugenol (Senanayaka, 1998). This difference may have been due to environmental influences on the plant material, but it is more likely to result from the fact that in general, the distillation time in Ambalangoda and Matara was much longer than that practiced in Negombo.

The oil is distilled from leaves trimmed off from shoots cut for quill production, and dried for three days before distillation in order to economize on fuel. The traditional stills used by many distillers are large wooden vessels capable of holding a charge of about 200Kg of leaves. The copper still head is rendered steam-tight by sealing with soft clay. Steam is generated in a separate wood-fired boiler and the leaves are distilled usually for 8 to 9 hours. The distillate is condensed in a copper coil immersed in a tank of water. Metal still bodies are also used in certain areas, and here heating water in the still body itself generates the steam. The leaves are placed on a grid above the surface of the water. In these cases the same type of condensing system is used. McConnell and Upawansa considered that the distillers usually underestimated their oil yield and distillation efficiency, and they concluded that the average distillation yield of oil was closer to 0.9%.

The objective of the present experiment was to assess the optimum resting time of cinnamon leaves and their raw materials for the maximum production of the good quality composition cinnamon leaf oil.

Materials and methods

In order to achieve the objective, the experiment was designed to assess the quantity and quality of the leaf oil where leaves are subjected to distillation with twigs and without twigs, while giving different time periods of resting. A laboratory experiment was conducted at the Cinnamon Research Institute, Thihagoda, during March 2002.

The experiment comprised two main treatments.
Treatment 1: The leaves separated from the harvested branches.
Treatment 2: Harvested branches (leaves with stem parts)

The second factor of the experiment was testing the quality and quantity of the cinnamon leaf oil with the resting period. Therefore the experiment was conducted over 14 days. First five readings were taken daily on the first five days. Next three readings were taken on every other day and the last reading was taken three days after the date of the previous reading.

Following parameters were considered in the experiment;

i. Quantity of leaf oil
ii. Quality of leaf oil
iii. Moisture content of raw material used for distillation

Sample selection for this experiment was very important as the oil content and its quality show high variation among different varieties of cinnamon (Senanayaka and
Wijesekara, 1989). Therefore leaves of Cinnamon line T\textsubscript{7} selected by the Cinnamon Research Center were used for the research. The samples were obtained from sticks with adequate maturity for harvesting.

Just after harvesting, the sample was prepared for treatment 1 by separating the leaves. The sample for treatment 2 was prepared by taking the harvested leaves with twigs as they were. Each treatment was replicated three times and the average was considered. Samples of the two treatments were used separately for determination of oil content and determination of moisture content by the distillation method. Modified Clevenger’s method was used for extraction of volatile oil.

The samples were subjected to distillation for 3 \( \frac{1}{2} \) hours. The oil part was separated from the water and collected into a clean, oven dried, weighed bottle. The minute water particles suspended in oil were removed using a glass syringe. The extracted moisture free oil was weighed and kept in an airtight container until it was analyzed for its quality. As oil yield is expressed in dry matter basis, the moisture content of each and every day of distillation should be known. A part of the sample prepared for the oil determination was used for the moisture determination.

Dean and Stark distillation method was applied for moisture determination. Each treatment was replicated twice and the average was taken as the moisture content.

\[
\text{Moisture \%} = \frac{\text{Volume of water (ml) \times 100}}{\text{Weight of sample}}
\]

\[
\text{Volatile oil \%} = \frac{\text{Weight of the oil (g) \times 100}}{\text{Dry matter content of the sample (g)}}
\]

G.L.C. method (SHIMADZU GC-8A, with carrier gas Argon) was used for the quality assessment of the experiment. Eugenol and cinnamaldehyde percentages were taken as main quality parameters of cinnamon leaf oil. Oil samples obtained from three replicates were mixed together to produce a single oil sample for G.L.C. analysis. Time duration was categorized into three groups and oil were mixed together in 1, 2 and 3 days, 4, 5 and 7 days and 9, 11 and 14 days.

Results and discussion

Variation of the oil yield according to the time period

The results of the oil yield production with and without twigs, exposing them to drying for different time intervals are presented in Table 1.

<table>
<thead>
<tr>
<th>Days after harvesting</th>
<th>Oil yield (% on dry matter)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Leaves</td>
</tr>
<tr>
<td>1</td>
<td>2.04</td>
</tr>
<tr>
<td>2</td>
<td>2.22</td>
</tr>
<tr>
<td>3</td>
<td>2.30</td>
</tr>
<tr>
<td>4</td>
<td>2.33</td>
</tr>
<tr>
<td>5</td>
<td>2.63</td>
</tr>
<tr>
<td>7</td>
<td>2.33</td>
</tr>
<tr>
<td>9</td>
<td>2.44</td>
</tr>
<tr>
<td>11</td>
<td>2.23</td>
</tr>
<tr>
<td>14</td>
<td>2.19</td>
</tr>
</tbody>
</table>
According to the SPSS analysis for independent samples, it was revealed that there was significantly higher oil content at 5% significance level in leaves when they were subjected to distillation without twigs.

![Graph showing oil yield vs. days after harvesting.](image)

**Fig. 1 Variation of oil yield with time period**

The variation of oil content with time in different treatments is shown in Fig. 1. According to the results, the maximum oil yield was observed on the 5th day in both treatments. The reason for the low oil contents during the first four days may be due to the cell wall barrier for the oil to come out and mix with the liberating steam. As the moisture content reduces with time, the cell wall barrier also reduces, facilitating oils to come out. Further reduction of moisture content causes degradation of some volatile compounds which results in lower oil yields.

The maximum oil yield in pure leaves was 2.62% with an average of 2.30%. The maximum oil yield in leaves with twigs was 2.32% with an average of 1.80%. Maximum oil productions in both cases were achieved when leaves were distilled on the 5th day after harvesting. The rapid drop down of the oil yield was clearly noticed on the 6th day after harvesting when leaves were distilled with twigs. Significant drop down was not exhibited when pure leaves were distilled (Figure 1). It was evident that oil yield was always higher when pure leaves were used for the distillation (Figure 2).

![Graph showing oil content vs. type of raw materials.](image)

**Fig. 2 Variation of oil content according to the type of raw materials**
Variation of moisture content of the raw materials during the time period

The moisture content determined on reference days are shown in Table 2. Initial moisture content of both treatments was about 60% and the drying pattern of both was almost similar. The moisture content of the leaves was stabilized at a constant of about 9% on the 8th day after the harvesting. The moisture content of the leaves with twigs was about 16% on the 14th day and showed a further decreasing trend (Figure 3).

<table>
<thead>
<tr>
<th>Number of days after harvesting</th>
<th>Moisture content (%)</th>
<th>Leaves</th>
<th>Leaves + Twigs</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>60.0</td>
<td>58.0</td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>51.0</td>
<td>50.0</td>
<td></td>
</tr>
<tr>
<td>3</td>
<td>44.0</td>
<td>47.0</td>
<td></td>
</tr>
<tr>
<td>4</td>
<td>32.0</td>
<td>42.5</td>
<td></td>
</tr>
<tr>
<td>5</td>
<td>18.0</td>
<td>28.0</td>
<td></td>
</tr>
<tr>
<td>7</td>
<td>10.0</td>
<td>21.0</td>
<td></td>
</tr>
<tr>
<td>9</td>
<td>9.5</td>
<td>19.0</td>
<td></td>
</tr>
<tr>
<td>11</td>
<td>9.0</td>
<td>18.0</td>
<td></td>
</tr>
<tr>
<td>14</td>
<td>9.0</td>
<td>16.0</td>
<td></td>
</tr>
</tbody>
</table>

Figure 3 shows that the moisture content of the leaves with twigs was always higher than the moisture content of the pure leaves during the time period after the harvesting.

![Figure 3: Variation of moisture content with days](image-url)
Variation of oil yield with moisture content

The moisture content varied with the time period. Oil yield also varied with the content of moisture in sample. Figure 4 illustrates the relationship between moisture content and the oil yield. It is evident that maximum oil yield in both treatments could be achieved on the 5th day after harvesting when the moisture content of the pure leaves was 18%, while that of the leaves with twigs was 28%. The oil yield of both treatments were reduced with decreasing moisture levels as the volatile compounds of the cinnamon leaf oil vaporized during the drying process.

Oil quality

Eugenol and cinnamaldehyde contents were considered as quality parameters. Table 3 shows the variation of eugenol and cinnamaldehyde contents of leaves alone and leaves with twigs during the three days time intervals.

Table 3. Variation of the chemical compositions among the treatments during time intervals

<table>
<thead>
<tr>
<th>Time duration</th>
<th>Eugenol</th>
<th>Cinnamaldehyde</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Leaves</td>
<td>Leaves + Stem</td>
</tr>
<tr>
<td>1 (Average values of 1,2,3 days)</td>
<td>81.26</td>
<td>76.78</td>
</tr>
<tr>
<td>2 (Average values of 4,5,7 days)</td>
<td>88.64</td>
<td>78.38</td>
</tr>
<tr>
<td>3 (Average values of 9,11,14 days)</td>
<td>89.8</td>
<td>83.21</td>
</tr>
</tbody>
</table>
Variation of the eugenol content

It was observed that the eugenol percentage of pure leaves showed a rapid increase from 81.26% to 88.64% at the second time duration and subsequently a slight increase was observed at the end of the third time duration period (Fig. 5). The eugenol percentage of the leaves with twigs increased from 76.78% to 83.21% at the end of the third time duration. Average eugenol content of the oil extracted from pure leaves and the leaves with twigs were 86.57% and 79.46% respectively.

Eugenol content was increased with time duration due to some other volatile compounds of the oil being vaporized with time, thus increasing the concentration of the eugenol compound.

Fig. 5 Variation of the eugenol content between the two treatments within three days time duration

Eugenol is the main valuable constituent of the leaf oil. The eugenol percentages of pure leaves were always higher than the treatment of leaves with stem parts. This may be due to the eugenol being mainly assimilated in the leaves, while in the stem eugenol is converted into nonvolatile lignin like substances (Senanayaka and Wijesekara, 1989).

Variation of the cinnamaldehyde content

The variation of the cinnamaldehyde content with the different time durations is shown in Figure 6. Leaves with twigs had higher cinnamaldehyde content during the experiment time duration whereas cinnamaldehyde content in pure leaves was comparatively low. This is expressed because cinnamaldehyde is the main constituent in the bark oil and the twigs contain the bark component of cinnamon.

It is evident that cinnamaldehyde content of the pure leaves and the leaves with twigs showed a slight increase up to the second time duration and then they showed a decreasing trend. Average cinnamaldehyde content of the oil extracted from pure leaves and the leaves with twigs were 1.75% and 4.58% respectively.
Fig. 6 Variation of the cinnamaldehyde content of the pure leaves and the leaves with twigs during the different time durations

Conclusions

Optimization of cinnamon leaf oil yield in respect of both quantity and quality could be achieved through the selection of the proper raw materials and maintaining of resting time to reset the leaves prior to distillation. Better leaf oil quality and quantity can be achieved by the selection of raw materials without twigs.

When pure leaves are taken for the distillation process, average oil yield could be 2.32%, which is higher by 27% compared to distillation with unselected material including twigs and branches.

Optimum resting period for maximum production of leaf oil in Matara can be observed on the 5th day after harvesting the leaves. On the 5th day the eugenol and cinnamaldehyde content would be 88.64% and 1.85% respectively which were the parameters of better quality leaf oil.

Acknowledgements

The authors greatly appreciate the valuable assistance given by Mr. Gamini Wijesinghe, Research Officer in Charge and the staff of the Cinnamon Research Institute, Thihagoda in providing the laboratory facilities. The first author is extremely thankful to the National Science Foundation (NSF) for providing grants to pursue the studies.

References: