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Studies on the Pinus Species growing in Sri Lankan Plantations
II. Turpentine Content of *Pinus caribaea* Oleoresin and Studies Directed towards the Commercial Extraction of Turpentine

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Abstract : The average turpentine content of *Pinus caribaea* oleoresin (Erabedde Plantation) is about 18% to 20%. However, individual trees showed marked variations not only from each other but also from season to season and day to day. Turpentine content may be as high as 30% or as low as 10%. The rate and efficiency of recovery of turpentine depends on (1) the quantity of water present and (2) cohobation. Detailed laboratory studies have led to a refinement in operating conditions, so that a simple fire still (50 kg capacity) can be made to yield turpentine quantitatively in a very short distillation time.

1. Introduction

Pinus caribaea is a tropical pine now grown to the extent of about 32,000 acres in Sri Lanka.[1] Investigations on the tapping and oleoresin output of the *P. caribaea* plantation of Erabedda had been reported previously by this group.[3] In this study we report the turpentine content of the oleoresin when the plant is grown under local conditions as well as investigations aimed at the commercial extraction of the turpentine.

There are several methods for isolating the terpenes of Pine. (1) Tapping of the oleoresin.[4] (2) Solvent extraction.[5] (3) Steam distillation of wood and chips, and (4) Dry distillation.[7] However, only the first two are widely used for producing rosin and turpentine with the solvent extraction process being favoured by most developed countries. In other parts of the world, the tapping method is still popular, and in this situation the next step is necessarily the separation of rosin and turpentine by steam or water distillation.[6] Most rosin producing countries use large centralised and continuous stills using steam under pressure,[2,4,8] a situation clearly not suitable for Sri Lanka at this early stage. An alternative method is the use of a simple fire still,[4,7,8] which was popular in the early part of the century. Unfortunately, the critical factors involved in the operation of a fire still are not clearly documented.

*Some of this work has been carried out as a part of the M. Phil. Thesis of L. A. Goonetillake.

2. Experimental

2.1. Determination of Turpentine Content of Oleoresin

Oleoresin (25 to 100 g) was water distilled using 150 ml water for 4 hours, and the turpentine collected in a light oil (clavenger) arm. During 4 hours nearly all the turpentine is recovered (Figure 1), but the method has an error of about 5%.

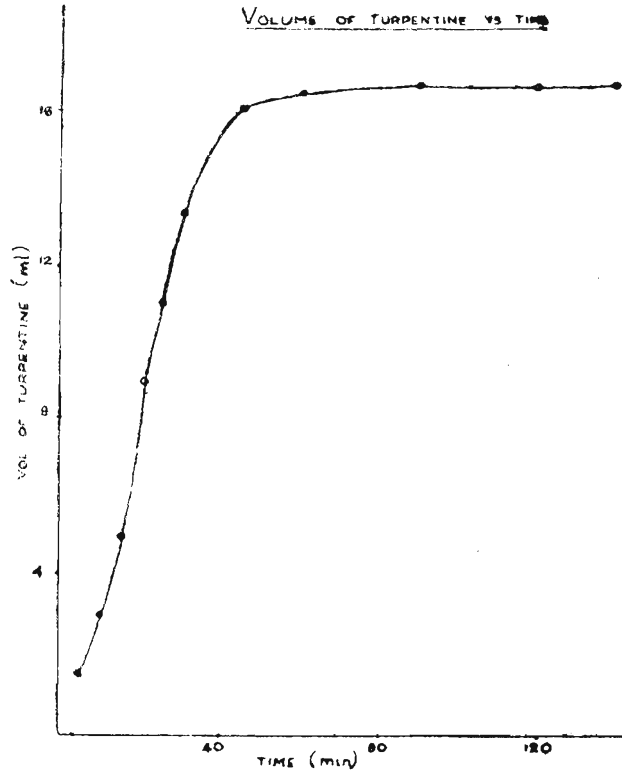

Figure 1. *Time Courses of Turpentine Recovery.* Weight of Rosin, 60 g ; Volume of water, 150 ml.

2.2. Factors Affecting Efficient Recovery of Turpentine

Experimental details are given in legends of figures.

2.3. Pilot Plant stills

(*a*) *Model* 1.

Designed for steam distillation and the production of high quality rosin, this is a single container where the oleoresin chamber is separated from the boiler by a partition that allows only the passage of steam. As a result, the temperature of the oleoresin does not rise above 100°C. The boiler is constructed of iron and the rosin chamber of aluminium.

(*b*) *Model* 2.

This is a copper fire still, where the rosin is in the boiling compartment (Figure 2). By controlling the amount of water, the rate of turpentine recovery can be optimised.

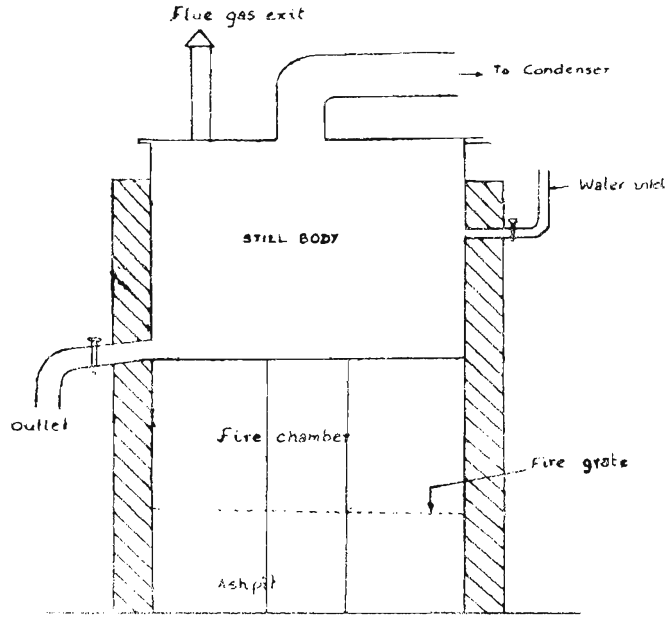

Figure 2. *Turpentine, Fire Still* Maximum capacity = 50kg.

The still comprised a still body 1M by 0.7M containing an inlet (2 cm diameter) for the entry of cohobate and exit (25 cm diameter) for hot rosin. The lid contained the exit to the multitubular condensor. The still body is heated at the base by direct firing. The furnace contained a fire chamber and ash-pit separated by a fire grate and a flue gas exit.

3. Results

3.1. Turpentine Content of the Oleoresin.

The mean turpentine content of the oleoresin of *Pinus caribaea* (of Erabedda) is about 18% to 20%. Turpentine content of the oleoresin varies markedly : (*a*) from tree to tree, (*b*) tapping cycle to tapping cycle and (*c*) day to day.

The variation from tree to tree is illustrated in Table 2. There appears to be a distinct direct relationship between yield of oleoresin and turpentine content of oleoresin.

Studies also showed the turpentine content of the oleoresin also markedly varied with tapping cycle (Table 1).

TABLE 1. Variation of Turpentine Content of Oleoresin

Code No. of Tree	Tapping Cycle			
	Sept. 1978	Dec. 1978	March 1979	June 1979
101	18.6	19.9	16.3	20.5
102	16.4	21.8	25.0	20.0
104	—	19.5	19.3	19.3
106	—	—	10.6	18.2
107	—	—	—	18.9
108	18.3	10.6	9.4	—
109	19.0	13.4	18.9	21.2
110	9.1	17.9	13.1	17.9
111	9.7	13.5	12.4	16.7
112	19.2	18.0	16.9	19.1
113	17.6	—	12.5	21.7
114	17.6	18.5	5.7	19.3
115	17.5	16.7	14.7	19.2
117	10.8	—	—	13.6
118	11.7	—	18.1	24.9
119	—	—	—	22.6
120	—	—	15.6	13.3

—, signifies not determined due to low oleoresin output.

The situation is further complicated by the fact that there is nearly always even a daily variation of turpentine content of oleoresin even in the same tree ; turpentine content generally decreasing with time. In one tree (studied in detail), turpentine content which was 27.3% in the first day had declined to 16.5% by the fifth day.

3.2. Laboratory Studies on Recovery of Turpentine.

These studies were concentrated on two main lines (*a*) the effect of the oleoresin to water ratio and (*b*) the effect of cohobation on the rate of turpentine recovery.

3.2.1. *Effect on Oleoresin to water ratio.*

Table 2 shows that large quantities of water result in a relatively inefficient rate of turpentine recovery. In this experiment, relative rate of turpentine recovery is measured by determining the quantity of turpentine recovered for every 100 ml water distilled. At limiting water levels, the temperature of the contents of the flask is higher than 100°C.

TABLE 2. Effect of Water to Rosin Ratio.

Volume of Water added (ml)	Vol. Turpentine per 100 ml distillate (ml)
150	18.0
200	12.7
300	11.4
400	9.8
500	7.7

Rosin (300g) was distilled with the above quality of water at constant rate (heating mantle). Rate of distillation 100 ml/15 min. Total turpentine in 300 g Rosin = 30 ml. Values for turpentine given above refer to first 100 ml of distillate produced in each case.

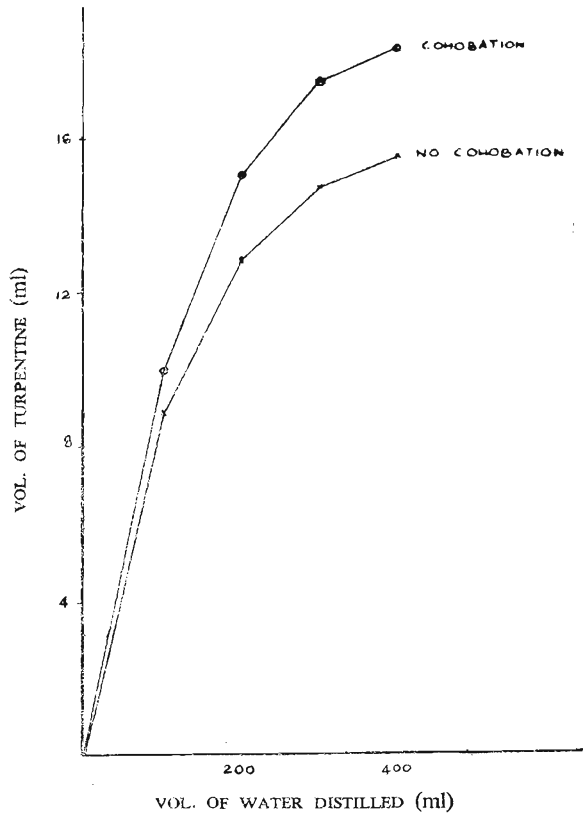

Figure 3. *Effect of Cohobation on Turpentine Recovery.* Rosin (150 g) was distilled with water (100 ml)
X — X, without Cohobation
O — O, with Cohobation.

3.2.2. *Effect of Cohobation*

At a temperature of 35°C to 40°C the turpentine forms an emulsion with water (containing 0.5 ml turpentine/100 ml water). Therefore by recycling the separated water it was possible to increase yields. Figure 4 shows the effect of cohobation on the distillation of turpentine. A similar experiment on a larger scale gave the same result (Figure 4.).

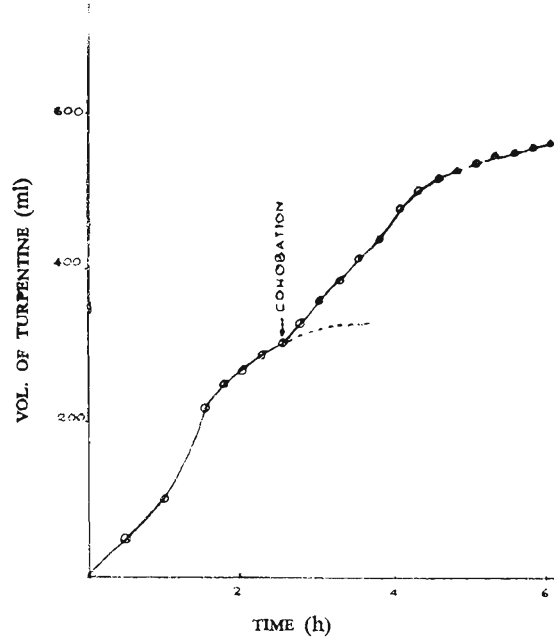

Figure 4. *Effect of Cohobation on Turpentine Recovery.* Rosin (6.5 kg) was distilled with 41 water at point indicated by cohobation was initiated. The experiment was terminated when only about half the turpentine was recovered.

3.3. Pilot Plant Studies

Steam distillation using the still-model 1 resulted in both a very low efficiency of recovery and low rate of recovery of turpentine (Table 3). After 8 hours distillation, only 84% of the turpentine was extracted. However using model 2, (the fire still) the situation was reversed ; in only 3.0 h over 99.5% of the turpentine was recovered (Table 4). The efficiency of the second method was proved beyond doubt when the residual oleoresin from model 1 (containing 3.8% turpentine) was exhausted of turpentine in only 1h using model 2 (Table 5).

TABLE 3. Recovery of Turpentine Using Steam Distillation Still (Model 1).

Time (hours)	Water distilled (l)	Turpentine recovered (l)
1	12	0.75
1.5	21	1.025
2	27	1.225
3	42	1.550
4	58	1.775
5	75	1.935
6	92	2.095
7	109	2.245
8	127	2.355

Approx 13.89 kg of Oleoresin was used. Yield of Turpentine was 17.1 v/w. (2355 ml). Residue contained 3.8% turpentine. Efficiency of recovery = 84%. Furnace temperature = 900°C. Water was cohobated.

TABLE 4. Recovery of Turpentine Using Fire Still (Model 2).

Time (hour)	Rate of distillation (ml/min)	Turpentine (ml/min)
0.2	225	160
0.5	300	140
1.0	240	60
1.25	370	45
1.5	345	25
2.2	512	10
3.0	350	2.5

Laboratory estimation of turpentine. 20.1%; 36.45 kg Oleoresin was distilled. Yield of turpentine 20.8% v/w (7.6 Litres). Residual Turpentine (0.1%). Efficiency of recovery 99.5%. Initial water = 10 l. Cohobation, 4 l at a time.

TABLE 5. Recovery of Residual Turpentine Using Fire Still.

1.	Weight of Oleoresin	= 8.5 kg
2.	Water added	= 3 L
3.	% Turpentine (Laboratory Determination)	= 3.8 % v/w
4.	Time of distillation	= 1.25 h
5.	Rate of distillation	= 400 ml/min
6.	Observed yield	= 335 ml
7.	*Expected yield	= 323 ml
8.	Residual turpentine	= Not detected
9.	% α– Pinene—Laboratory Distilled	= 66%
10.	% α– Pinene—Fire Still	= 67%

*Based on sample drawn for laboratory estimation.

4. Discussion

Results show that the average turpentine content of oleoresin is about 20%. However, there is wide variation (1) between trees, (2) from tapping cycle to tapping cycle and (3) day to day. There appeared to be some relation between turpentine content of oleoresin and oleoresin yield. However, other unidentified factors probably also affect yield. The relationship is interesting as the turpentine content of the oleoresin will affect fluidity.

Results of pilot plant studies were highly encouraging. By controlling water content of the distillation and using cohobation, it is possible to obtain turpentine (of the same quality as laboratory distilled material) not only in a very short distillation time but also at nearly quantitiatve yields by using a simple fire still.

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