Part II.—The Distillation of some South Indian Woods.

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In part I a brief description has been given of the general methods of distilling wood and of working up the products.

Before drawing definite conclusions as to the financial aspects of the wood distillation industry in India it is necessary to know something about the yields of by-products which would be obtained from Indian woods. Data, derived from both small scale and commercial experiments, are available for many European and American woods, but practically no experiments have been made with woods available in large quantities in India.

The experiments made with American woods prove that the yields are not the same when commercial retorts are used as when small scale experiments are made in the laboratory, as it is practically impossible to reproduce on a small scale the exact conditions of a commercial plant, so that any series of experiments in the laboratory give only relative values. These are of use for determining the most suitable woods for distillation, and moreover if one or two of the samples can be treated on a larger scale, the values may then be made absolute.

The wood samples used in our experiments were, in all cases, kept in a well ventilated room for some months before distillation. They were then sawn into two foot lengths, and split, if necessary, into pieces 3 to 4 square inches in cross sectoinal area. The bark was not removed and no distinction was made between heartwood and sapwood. At the time of cutting, thin sections were removed for moisture determinations. The wood was then immediately packed into the still until the latter was quite full. The charge varied from 110—140 lbs.

The still itself was electrically heated and consisted of a horizontal cylinder of 1/32 inch sheet iron, 4 feet long and 15 inches in diameter, and was closed at the ends by circular iron plates bolted to flanges. It was covered with a sheet of asbestos card 1/8 inch thick over which was wound a length of 300 feet of No. 14 S. W. G. nickel wire the resistance of which when cold was 3 ohms. The wire was protected and held in place by being covered to a depth of about a quarter of an inch with a paste of finely powdered magnesium oxide and water. The whole was enclosed in a wooden

case, 4 feet square and 6 feet long, which was packed with asbestos powder for heat insulation. One end of both the still and wooden case was removable for charging and emptying the still.

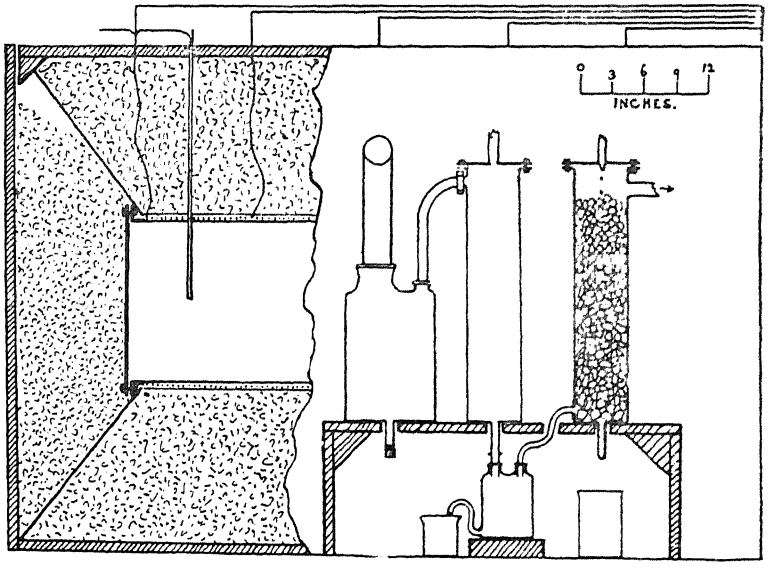
Three extra electrical connections were made to the heating wire at equidistant points, dividing it into 4 sections which could be heated independently if necessary. The current was controlled by suitable carbon or liquid resistances, but in practice the increase of the resistance of the heating wire with rise in temperature of the still afforded a very satisfactory automatic regulator. It was also found necessary to continue the heating on the end coils rather longer than on those in the middle owing to the extra loss of heat at the extremities of the still.

The temperature was measured by means of three copper constantan thermocouples inserted into iron tubes reaching to the axis of the still. Two of the tubes were inserted about six inches from the two ends and the third one midway between the other two.

The gases were carried off by a vertical two-inch pipe from the centre of the top of the still. This was bent downwards and connected to a tar-pot from which a glass tube led to a copper condenser. At the bottom of this was a separator for collecting the condensed liquid, and the gases finally passed through a scrubber before being allowed to escape. The gas was occasionally used for redistilling pyroligneous acid.

The whole arrangement is shown in diagram No. 2.

Diagram No. 2.



It was found that the outlet pipe got completely choked with a hard carbonaceous mass after about sixteen distillations and it was necessary to take it down and clean it at frequent intervals.

When conducting a distillation, a considerable amount of heat was supplied at first, and as soon as water began to distil the current was reduced. Subsequently the current was adjusted so that the rate of distillation was, as far as possible, constant, (about 1 litre in fifteen minutes), and the temperatures at the ends and middle of the retort approximately the same. The maximum temperature reached was in all cases not less than 350°C, and rarely exceeded 400°C.

Table I gives a complete set of readings for one distillation and shows details as to current, temperature, and the manner of regulation. It also shows that there were occasionally very violent local actions in the still. The temperatures given are in all cases measured on the central axis; those at the surface, while the current was running, could be measured by partially withdrawing the thermocouples, and were usually 20°—30° higher than those on the axis.

After completion of a distillation, the retort was left closed overnight and opened the next day. Owing to the very thick lagging the charcoal was still quite hot, and it was necessary to rake it out very quickly into an airtight vessel to avoid rapid combustion. It was weighed hot.

The distillate was mixed, allowed to stand for two days, and the tar and aqueous portions separated and weighed. They were then analysed by the methods given by Klar (Technologie der Holzverkohlung p. 337). As for purposes of comparison it is essential that the methods of analysis should be the same and as Klar's book may not be available for many, we give a brief outline of the analytical methods.

The acetic acid and dissolved tar were estimated by distilling 100 c. c. of the pyroligneous acid from a distilling flask heated by an oil bath, the temperature of which was gradually raised to 140°; when no more liquid distilled over steam* was

*The method sometimes used of distilling off the water and acid, heating to 140°, adding 50 c. c. of water and repeating the distillation, gives low results for the acetic acid, as about 7% of the total acid is retained by the tar. The method of blowing steam through or over the tar until the distillate is neutral is more accurate.

Accurate determinations of acetic acid cannot be made by directly titrating the crude liquid, this is largely due to the colour of the liquid and the presence of phenolic compounds making it difficult to detect the end point.

TABLE I.

Distillation No. 10.

Sample of Wood No. 16. Kendala, *Schleicheria trijuga.*

Charge= 54·3 Kilos. Moisture 16·5 per cent. = 8·95 Kilos.

	Current in Amps.		Temperatures in degrees cent.			Distillate in litres.	
Time.	Left.	Right.	Left.	Middle.	Right.	Total.	Per 15 mins.
8· 0	25	25	44	44	43		
8·15	25	25	49	49	45		
8·30	23	23	64	59	64		
8·45	21	21	76	74	85		
9· 0	20	20	97	92	107		
9·15	20	20	102	99	113		
9·30	19	19	108	108	120	0·48	0·48
9·45	18	18	110	110	125	1·13	0·65
10· 0	18	18	110	109	129	1·85	0·72
10·15	18	18	115	110	137	2·55	0·70
10·30	18	18	119	114	139	3·30	0·75
10·45	18	17	120	119	147	4·10	0·80†
11· 0	18	17	123	120	150	4·98	0·88
11·15	18	16	129	126	158	5·88	0·90
11·30	18	15	131	131	167	6·86	0·98
11·45	18	15	134	141	172	7·81	0·95
12· 0	18	15	142	151	176	8·90	1·09
12·15	18	15	153	169	185	10·00	1·10‡
12·30	18	0	153	190	195	11·18	1·18
12·45	16	0	155	195	196	12·27	1·09
1· 0	16	10	172	205	199	13·26	0·99
1·15	16	10	188	211	206	13·98	0·72
1·30	16	16	225	236	215	14·82	0·84
1·45	15	15	259	272	227	15·64	0·82
2· 0	17	17	257	310	245	16·41	0·77
2·15	19	17	271	319	299	17·26	0·85
2·30	20 *	18 *	274	319	361	18·84	1·58
2·45	14	0	280	331	399	20·31	1·46
3· 0	12	0	304	344	379	21·33	1·02
3·15	10	0	349	351	368	22·29	0·96
3·30	10 *	0	359	359	360	23·16	0·87
3·45	0	0	368	360	359	23·90	0·74
4· 0	0	0	381	362	358	24·50	0·60
4·15	0	0	388	360	354	24·68	0·18
4·30	0	0	388	359	351	24·73	0·05

* Current in end coil only. † Gas burns unsteadily. ‡ Gas burns well.

blown through the residual tar until the distillate was no longer acid. The residual tar was weighed and an aliquot part of the distillate titrated with standard sodium hydroxide solution using one per cent. solution of phenolphthalein as indicator.*

The wood spirit was determined by distilling 500 c. c. of pyroligneous acid untill 60 per cent had been collected in the receiver, then neutralising this with sodium hydroxide and redistilling again until 60 per cent. had been collected, the operation was repeated a third time using more sodium hydroxide. The density of the final distillate was determined with a specific gravity bottle and the percentage of spirit calculated, assuming it to be pure methyl alcohol and using Dittmar and Fawsitt's table. During the distillations, special precautions were taken to avoid loss of alcohol, as the temperature of the condenser water was usually 25° and sometimes higher.

The acid in the tar was determined by heating 100 grams of the tar to 130° in an oil bath, blowing steam through it until the distillate was no longer acid, and titrating the distillate with standard alkali.

The acetic acid and alcohol in the scrubber liquid were also analysed in a similar manner. The quantity of acetic acid passing over with the gas was negligible, (about 1%), but as much as 10% of the total methyl alcohol was usually collected owing to the high temperature of the condenser water.

The percentages of acid and alcohol given in the different tables include the amounts found in the tar and scrubber liquid.

In all cases at least two distillations were performed, and it was found that the variations obtained in the cases of acetic acid, methyl alcohol and tar were usually within 7% and in the case of charcoal 3%.

The mean results for the Mysore woods are given in table II. as percentage yields by weight on the air-dried wood. The moisture content is also given, so that the yields from dry wood may be calculated approximately, but it must be borne in mind that the wood, if dried previous to distillation, would give a different yield.

*If one or two drops of phenolphthalein are used the end point is not sharp; the addition of 10 or 12 drops of the indicator is therefore recommended.

TABLE II.

Table showing the yields of products obtained by the distillation of various kinds of wood under the same conditions. All results are given as the number of parts by weight from 100 parts of undried wood.

Vernacular name.	Botanical Name.	Moisture.	Total Distillate.	Charcoal.	Gas.	Acetic Acid.	Methyl Alcohol.	Tar.
Casurina (old)	Casurina equisetifolia (Forst.)	24·0	50·5	28·0	21·5	3·68	1·15	6·2
Do. (young)	Do.	32·4	55·2	26·2	18·6	2·66	1·13	6·1
Dindiga	Anogeissus latifolia (Wall)	16·8	47·7	33·8	18·5	3·54	1·26	8·1
Godda	Garuga pinnata (Roxb.)	32·0	55·2	28·8	16·0	3·36	1·55	5·7
	Eucalyptus globulus	18·1	49·2	26·2	21·2	3·24	1·34	5·2
Kanagal	Dillenia pentagyna (Roxb.)	16·8	47·3	34·8	17·9	3·12	1·24	6·9
Padri	Stereospermum suaveolens (D. C.)	15·0	49·8	30·9	19·3	3·06	1·84	10·0
Jalari	Shorea Talura (Roxb.)	18·1	51·9	36·6	11·5	2·94	1·14	6·0
Nelli	Phyllanthus emblica (Linn.)	17·0	49·2	33·5	17·3	2·93	1·26	5·7
Teak	Tectona grandis (Linn.)	19·5	54·1	31·3	14·6	2·83	1·35	8·9
Tari	Terminalia belerica (Roxb.)	12·4	46·0	29·0	25·0	2·69	1·55	6·8
Thadasal	Grewia tiliaefolia (Vahl.)	17·5	49·2	32·3	18·5	2·64	1·56	7·0
Zettiga	Adina cordifolia (Hk. f.)	19·1	53·2	29·0	17·8	2·54	1·39	8·0
Kalvala	Anthocephalus cadamba (Miq.)	16·2	44·0	32·7	23·3	2·54	1·48	6·9
Beete	Dalbergia latifolia (Roxb.)	16·1	48·0	34·1	17·9	2·53	1·86	11·0
Kendala	Schleichera trijuga (Willd.)	16·5	49·7	33·4	16·9	2·51	1·59	8·0
Nerlu	Eugenia Jambolana (Lam.)	21·4	45·5	32·1	22·4	2·50	0·93	5·4
Nandi	Lagerstroemia lanceolata (Bedd.)	22·0	47·1	36·0	16·9	2·47	1·08	5·8
Honal	Terminalia paniculata (Roth)	16·1	45·5	37·8	17·7	2·47	0·90	5·8
Jambe	Xylia dolabriformis (Benth.)	11·2	40·0	42·0	18·0	2·46	1·17	4·9
Honne	Pterocarpus marsupium (Roxb.)	26·1	47·3	30·7	22·0	2·40	1·25	5·5
Matti	Terminalia tomentosa (W. and A.)	20·8	42·3	36·1	20·6	2·08	1·05	4·6
Huli Nelli	Bassia malabarica (Bedd.)	36·0	60·3	25·8	14·9	1·95	1·33	5·3

In table III are given some results for teak from Nilambur. These experiments were conducted with the object of finding out the value, for distillation purposes, of the various portions of the trees not required for timber.

TABLE III.

South Indian Teak (Tectona grandis).

No.	Diameter of wood inches.	Moisture.	Total Distillate.	Yields calculated on moist wood as distilled.				Yields calculated on wood with 15% moisture.			
				Charcoal.	Acetic acid.	Alcohol.	Tar.	Charcoal.	Acetic acid.	Alcohol.	Tar.
1	1½ 2½	11·2	38·6	33·2	3·05	1·06	9·6	31·8	2·92	1·01	9·2
2	2½—3½	10·1	19·7	31·5	3·32	1·47	9·4	29·8	3·14	1·39	8·9
3	3½—5½	13·6	43·0	31·0	2·89	1·20	9·2	30·5	2·84	1·18	9·1
4	5—6	17·9	50·4	27·3	2·58	1·49	6·4	28·3	2·63	1·54	6·6
5	8	21·3	57·7	26·4	2·54	1·12	7·5	28·2	2·71	1·19	8·0

All the above were cut into pieces about 16 inches long. No. 1 was distilled whole with bark, Nos. 2 and 3 were split into two, and Nos. 4 and 5 split into four.

The larger pieces contained the most water and consequently, weight for weight give considerably less by-products than the smaller pieces. If the second part of the table is examined, where the yields are calculated for a uniform moisture content of 15 per cent., it will be seen that the yields given by the smaller pieces still preponderate. This difference may be a real one depending on the actual structure of the wood, but it is more likely due to the fact, mentioned in part I p. 85, that wood which contains excessive moisture gives a lower yield of acid than drier wood.

In any case from the commercial point of view it would seem more advantageous to distil the smaller pieces, both because they are more readily dried, and because the labour required for breaking them up is less; or perhaps, in view of the fact that the distiller is not usually able to control the sizes of wood available, it would be advisable to say that there is no disadvantage in distilling wood down to 1½ inches in diameter. For smaller sizes than this the charcoal would be of little value

for ordinary purposes and this would be the deciding factor rather than the yields of by-products, unless the method of briquetting the charcoal was adopted.

A certain number of experiments were made with the object of ascertaining relationships between variations in the conditions of distillation and the yields of by-products. The results are not sufficiently numerous to justify any definite conclusions being drawn, but apparently small variations, which must inevitably occur during a distillation in the normal manner, have no appreciable effect on the results.

As already mentioned a most important question which arises in connection with our experiments is, how do the yields obtained in the laboratory on a small scale compare with those which would be obtained from the same woods on a commercial scale? It is, impossible to answer this definitely, but a rough idea can be formed by comparing the results for Casurina and Eucalyptus with some which were obtained in England with samples of these woods on a commercial scale. These results are given in Table IV.

TABLE IV.

Comparison of commercial and small scale distillations.

Type of wood.	Moisture.	Charcoal.	Acetic acid.	Methyl alcohol.	Tar.
Casurina 26 years old, c.	23·0	29·7	3·76	2·0	4·5
„ 14 „ „ , c.	22·0	29·0	3·98	1·9	4·5
„ from table I., l.	24·0	28·0	3·68	1·15	6·2
Eucalyptus, c.	15·0	30·0	3·5	1·0	4·7
„ from table I., l.	18·1	26·2	3·24	1·34	5·2

From this comparison it would appear that the yields of acetic acid obtained in the experimental retort are lower than those in the large scale operations. The discrepancies in the case of methyl alcohol are too large for any conclusions to be drawn.

In this connection it is interesting to compare the present experiments, with a series carried out by L. F. Hawley and R. C. Palmer* on the Destructive Distillation of Hardwoods. The still used by these authors was similar to the one described in this paper, but rather smaller; it was heated in an oil bath and

* U. S. Dept. of Agriculture Forest Bulletin No. 129, 1914.

was provided with an outlet at one end. The yields of acetic acid obtained were about 50 per cent. higher than the usual commercial figures. This is possibly due to the fact that the large mass of the retort and oil compared with that of the wood, very effectively controlled the temperature and prevented sudden changes. In the present experiments, the retort was larger and its heat capacity very small, so that a nearer approximation to the conditions in a large factory retort is attained. The difference is shown in a striking manner by examining the rates of distillation. From the one example given in detail by Hawley and Palmer it appears that in the oil-heated still a smaller charge gave a larger amount of distillate in less time than a larger charge in the electrically heated still, and yet the temperature rise in the former was considerably less, indicating a great loss of heat in the walls of the still.

Diagram III.

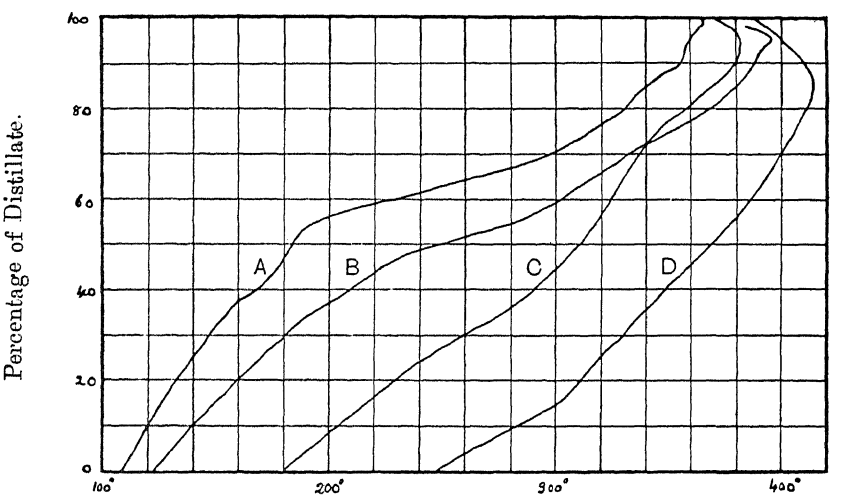


Diagram 3 shows the percentage of total distillate at various temperatures. Curves A and B are selected at random from the present experiments, the woods chosen being Kendala (*Schleicheria trijuga*) (A) and Honne (*Pterocarpus Marsupium*) (B) with 16·5 and 26 per cent. of moisture respectively. Curve C is given by Palmer* as the mean curve for maple wood in his small retort, and curve D by the same author for distillation on a commercial scale. It is obvious, that curves A and B are of a type quite different from C and D. In the two former the

* Journ. Indus. Eng. Chem., 1915, 7, 663.

distillate comes off rapidly at first for equal changes in temperature, and then much more slowly, while in the latter the reverse is the case. At the same time it is not clear how it is possible for no distillate to be evolved until the centre of the retort reaches a temperature of 180° and even 250° as is apparently the case in curves C and D.

Apart from this, however, it is evident that the exothermic reaction was much more rapid in the curves A and B, and this is probably the reason for the lower yields of acetic acid.

Since these experiments were carried out, a number of tests have been made on the woods of the Phillipine Islands by A. H. Wells* using a very similar, but much smaller electrically heated still. Unfortunately this author gives no details as to the course of individual experiments, so that his results cannot be compared with the others.

In addition to knowing the yields of by-products given by Indian woods, it is important to compare them with those given by woods in other countries, as in the event of the establishment of the industry in India, there would most certainly be a considerable amount of foreign competition. It is however very difficult to obtain any reliable data for other woods, particularly on a commercial scale, owing to the practice of measuring the quantities of wood distilled by the cord, a very indefinite quantity, and also owing to the fact that the moisture content of the wood is never determined in ordinary works. Table No. V. comprises a number of results from various sources for a few selected woods. In all cases where the moisture content of the wood is known, the figures are recalculated for a wood containing 15% moisture.

Column 6 gives the source of information, the abbreviations being as follows:—

H. P. L. F. Hawley and R. C. Palmer, U. S. Department of Agriculture. Bull. No. 129.

P. R. C. Palmer, U. S. Department of Agriculture—Bull. No. 508 and Journ. Indus. Eng Chem. 1915, *7*, 663.

D. Imp. Inst. Bull., 1916, *14*, 572.

S. Senfft, Ber., 1885, *18*. 60.

L. J. C. Lawrence, Journ. Soc. Chem. Ind., 1918, *37*, 11T,

* Phillipine Journ. of Sci. A. 1917, *12*, 111.

W. A. H. Wells, Phillipine Journ. of Sci., A, 1917, *12*, 111.

I. Results taken from the present paper.

l. denotes a laboratory, and c a commercical value.

The last column gives the actual amount of moisture in the wood used, although the yields are all calculated for a 15% moisture content.*

On studying these results it will at once be seen what little reliance can be placed on the absolute values. The experiments of Senfft were performed over 30 years ago, and he gives no details as to his methods of analysis. Hawley and Palmer's laboratory yields of charcoal appear to be very high, while those obtained at the Imperial Institute are low; no details as to the maximum temperature of distillation are given, however, in the latter case. Hawley and Palmer conclude that in actual factory practice the values for acetic acid would be about 66 per cent. of those obtained in their own small scale experiments.

With regard to acetic acid, it was hoped that a connecting link might be found between Palmer's results and the present series in Eucalyptus. This wood gave almost the lowest yield in the former set of experiments, and among the highest in the latter. Palmer's actual figure reduced to 15% moisture basis is 4·2% which is much higher than our figure while his reduced value 2·8% is much lower. The charcoal yields moreover are very different, and it is obvious that no comparison of any value can be made; indeed the actual species may be different.

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* Since the above was written a paper by R. C. Palmer and H. Cloukey has appeared in the Journ. Ind. and Eng. chemistry (1918, *10*, 262). In this paper experiments are described in which woods with different percentage of moisture were distilled under similar conditions. The authors draw the conclusion that both beech and birch actually give more acid and alcohol when the percentage of moisture is high. The results cannot be considered as altogether conclusive as the moisture content varied between 20 and 30 per cent only and the differences in the yields were small and in some cases were less than the differences between the results given in a previous paper (U. S. Forest Bull 1914 No. 129) and the present results, *e g.* in the case of birch the figure 6·68 per cent of acetic acid calculated on the dry wood is given in the earlier paper, and in the later paper the figure 5·02 is given for air dried wood presumably under similar conditions. The highest value given for birch in the later paper is only 5·62 namely for a wood containing 30·2 per cent of moisture. A larger number of experiments are required before the matter can be regarded as settled and in any case the other disadvantages of using wet wood, to which attention has been drawn, remain the same.

TABLE V.

Showing percentage yields of pure acetic acid, pure methyl alcohol, charcoal and tar for woods containing 15% of moisture.

Name of wood.	Source of wood.	Char-coal.	Acetic acid.	Methyl alcohol	Tar.	Autho rity.	Per cent of moisture actually present in the wood distilled.
Oak	America	30·3	3·79	1·21	4·7	L. c.	30
,,	England	30·7	4 60	1·34	12·5	L. l.	
,,	America	29·8	4·35	1·13	5·4	L. l.	
,,	Germany	34·7	4·08	—	3·7	S. l.	
,,	England	25·6	4·93	1·09	6·6	D. l.	17·1
,,	England	26·0	3·8	1·1	4·5	D. c.	
Beech	America	32·6	4·43	1·66	8·4	L. l	
,,	Indiana	35·8	4·98	1·59	8·8	H&P. l.	
,,	Germany	26·7	5·21	—	5·8	S. l.	
Maple	America	30·9	3·63	1·62	4·4	L. c.	15
,,	America	31·3	4·17	1·72	10·7	L. l.	
,,	Wisconsin	39·6	4·48	1·64	10·1	H&P. l.	
,,	America	30·8	3 19	1·45	—	P. c.	
Pine		33 0	2·19	0·86	17·2	L. l.	
,,	Germany	30·3	2·7	—	4·4	S. l.	
,,		28·9	2·4	0·60	17·2	D. l.	11
Eucalyptus	California	40·0	4·20	1·27	5·5	P. l.	
,,	S. India	27·2	3·37	1·39	5·4	I. l.	18·1
,,	S. India	30·0	3·5	1·0	4·7	I. c.	
Casurina	S. India	31·3	4·12	1·19	6·9	I l.	24·0
,,	S. India	32·2	4·24	2·14	4·9	I. c.	22·5
Teak	S. India	29·6	2·86	1·27	8·4	I. l.	
Vera (*Hemicyclia Sepiaria*). W. A.	Ceylon	25·0	1·69	—	1·9	D. c.	unstated.
Black Wattle.	East Africa	26·4	4·74	1·12	10·9	D. l.	8·8
Cocoanut Shells.	Phillipines	27·6	5·36	0·85	8.6	W. l.	
Apitong (*Dipterocarpus*)	Phillipines	27·7	3·23	0·77	16·8	W. l.	10·0
Bacauan (*Rhizophora*)	Phillipines	29·9	4·38	1·80	3·5	W. l.	8·1

On the whole, however, it would seem that Casurina gives yields as high as those from the woods commonly distilled in other countries, and Eucalyptus rather lower ones. If this is so, the majority of woods examined, for example teak, give considerably lower yields, a fact which must be taken into careful consideration when considering the establishment of wood distillation factories in India.

SUMMARY.

1. 22 species of South Indian woods have been distilled in the laboratory, and the yields of charcoal, acetic acid, methyl alcohol and tar ascertained.

2. The results appear to agree approximately with those obtained under commmercial conditions.

3. The yields of acetic acid are low when compared with those from the woods which are usually distilled in other countries,

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