THE CHEMISTRY OF THE EXUDATION FROM THE WOOD OF PENTASPODON MOTLEYI.

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Mr. C. E. Lane-Poole, Inspector-General of Forests, Australian Forestry School, Canberra, in a communication dated 28th November, 1927, enquired if the Sydney Technological Museum would undertake the examination, with special reference to its economic utilisation, of a certain oil which exudes from a tree occurring in New Guinea, and identified as close to Pentaspodon Motleyi. In compliance with this request, the examination was readily undertaken, and a quantity of the oil, which previously was obtainable in very limited quantity and difficult to procure, was made available. As a matter of fact, Mr. Lane-Poole reports that the quantity supplied, about 2 pints, took him four years to obtain.

The botanical description and general characters of the tree yielding this remarkable exudation are given in Mr. Lane-Poole's Report, "The Forest Resources of the Territories of Papua and New Guinea," 1925, page 109. (This publication is printed and published for sale by the Commonwealth Government of Australia).

The following interesting and necessary particulars which furnish an account of the physical characters and source of the exudation are extracted therefrom, viz.:—
The wood of this large tree, 8 feet girth, bole 80 feet, 120 feet over all, contains an oil in such abundance that it may be collected in conveniently placed receptacles, much as resin is collected from the Maritime Pine, only the cut must reach the heart. In many cases the flow is very heavy, and in one instance a gallon of oil was collected in three hours. In such cases it is probable that reservoirs of oil have been formed in hollows caused by rot, and the axe has tapped a crack that has piped off the supply. While a microscope may yield some explanation of the formation of the oil in the wood, a lens shows no special canals or vessels as one would expect to see. The oil is heavy and misty brown in colour; it resembles motor lubrication oil as used for cylinders. It has a smell which is hard to describe, though somewhat familiar—somewhat fishy linseed oil is the nearest I can get to it.”

We can confirm the physical characters so aptly described by Mr. Lane-Poole. To all intents and purposes, the oil, on account of its dark brown colour, viscosity, odour, etc., might easily be mistaken for a commercial boiled linseed oil.

Referring again to the origin of the exudation, Mr. Lane-Poole, in a private communication, states:—

“Microscopic examination shows that the medullary rays have canals, and these, even from dried specimens of the wood, still store their quantity of oil. This is clearly visible with the liberkuhn method of illumination. I am sending you a sample of the wood, so that you may examine it, also you will see that the oil exudations are visible to the naked eye.”

Mr. M. B. Welch, B.Sc., A.I.C., Economic Botanist at the Sydney Technological Museum, who examined the small sample of wood referred to above, furnished the following report thereon, viz.:

“The wood has been examined microscopically, and Sudan III. and alkannin both show the presence of oily bodies in the cavities of the sparsely distributed medullary secretory passages, in the cells of the thick walled protective sheath surrounding the canal, and to a slight extent in some of the wood parenchyma adjoining the canal, but nowhere else. The canals observed varied from 18-55 μ in diameter. The contents were practically all soluble in 95% alcohol.”

The very small original sample of exudation secured by Mr. Lane-Poole was submitted to Mr. T. G. H. Jones, University of Queensland, Brisbane, and his report is pub-
Experimental.

The two samples of oily exudation received gave the following chemical and physical constants on examination:

<table>
<thead>
<tr>
<th>Sample No. 1</th>
<th>Sample No. 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specific Gravity, 20°C</td>
<td>1.011</td>
</tr>
<tr>
<td>Refractive Index, 20°C</td>
<td>1.5280</td>
</tr>
<tr>
<td>Acid Number</td>
<td>139.08</td>
</tr>
<tr>
<td>Saponification Number</td>
<td>142.09</td>
</tr>
<tr>
<td>Solubility in 70% Alcohol (by weight)</td>
<td>2.6 vols.</td>
</tr>
<tr>
<td>Acid Number after acetylation</td>
<td>102.07</td>
</tr>
<tr>
<td>Iodine Number (Wijs)</td>
<td></td>
</tr>
</tbody>
</table>

It was found early in the investigation that the crude oil was soluble in 8% aqueous sodium hydroxide solution, and, therefore, in order to determine if the constituents were of a variable nature it was treated with 1% aqueous solution ammonium carbonate, 5% aqueous sodium carbonate, and 8% aqueous sodium hydroxide solutions respectively. The best procedure was to dissolve the crude oil in approximately four times its volume of ether, and to treat repeatedly with the reagents mentioned.

Ammonium Carbonate Extract. — On acidification with dilute sulphuric acid, extraction with ether and removal of solvent, only 0.8% of a dark brown viscous residue was obtained. It gave a faint violet colouration with ferric chloride in alcoholic solution much resembling the crude oil and main component, and was found to possess an acid number of 114.2.

Sodium Carbonate Extract. — Shaking at room temperature with this reagent failed to remove more than a trace of acid bodies.

The present investigation of the larger samples submitted has shown the exudation to consist approximately of about 90-95% of acid bodies possessing unusual characters. The crude oil is non-volatile in steam, and we were unable to effect its distillation under reduced pressure (1 mm.) without decomposition. Consequently, it is very difficult to produce evidence as to whether the principal component is a chemical entity or a mixture. For the purpose of this announcement it is regarded as a single acid. Evidence is adduced under "Experimental" which shows the principal acid to be mono-carboxylic with two hydroxyl groups, and to possess the molecular formula C_{24}H_{36}O_{4}. It gives a beautiful violet colouration with ferric chloride in alcoholic solution. Unfortunately, no crystalline or solid derivatives (except the silver salt) could be prepared.

The original objective of the investigation was to ascertain if the oil possessed any economic value. Consisting essentially of an acid or acids of high molecular weight, it yielded soaps with alkalies, which technically possessed special merits on account of their valuable emulsifying properties. At present, no other commercial use can be suggested. Its economic utilisation depends entirely on the possibility of supplies being obtained in commercial quantities. At present the prospects of its availability in large quantities at a cost which would enable it to compete with rosin or similar products is not promising, judging from Mr. Lane-Poole's report. However, very little is known of the extent of the natural products of New Guinea in its present undeveloped condition.
Experimental.

The two samples of oily exudation received gave the following chemical and physical constants on examination:

Sample No. 1. Sample No. 2.

Specific Gravity, 1.011 1.01
Refractive Index, 20° C 1.5280 1.5295
Acid Number 139.08 138.24
Saponification Number 142.09 146.64
Solubility in 70% Alcohol (by weight) 2.6 vols. 2.5 vols.

Acid Number after acetylation 102.07 102.53
Iodine Number (Wijs) 192.1

Reaction with Ferric Chloride in ethyl alcohol (Deep violet colour with precipitate forming on standing).

It was found early in the investigation that the crude oil was soluble in 8% aqueous sodium hydroxide solution, and, therefore, in order to determine if the constituents were of a variable nature it was treated with 1% aqueous solution ammonium carbonate, 5% aqueous sodium carbonate, and 8% aqueous sodium hydroxide solutions respectively. The best procedure was to dissolve the crude oil in approximately four times its volume of ether, and to treat repeatedly with the reagents mentioned.

Ammonium Carbonate Extract.—On acidification with dilute sulphuric acid, extraction with ether and removal of solvent, only 0.8% of a dark brown viscous residue was obtained. It gave a faint violet colouration with ferric chloride in alcoholic solution much resembling the crude oil and main component, and was found to possess an acid number of 114.2.

Sodium Carbonate Extract.—Shaking at room temperature with this reagent failed to remove more than a trace of acid bodies.
Sodium Hydroxide Extract.—On treatment with this reagent the greater part of the oil went into solution. On acidification with dilute sulphuric acid, extraction with ether, and removal of solvent, 93% of a dark reddish brown viscous oil, much resembling the crude exudation, was recovered.

Neutral Residue.—The main ethereal solution on removal of solvent yielded 5% of a yellow viscous oil with an acid number of 11 and refractive index of 1.5330. It did not give a colour reaction with ferric chloride in alcoholic solution.

Examination of Principal Acid Constituent.

Soluble in 8% sodium hydroxide solution.

This component, which constituted over 90% of the crude exudation, was found to possess the following chemical and physical characters, viz.:—

Specific Gravity, ........................................ 1.0132
Refractive Index, 20° C .................................. 1.5270
Solubility in 70% alcohol (by weight) ............... 3.3 vols.
Acid Number ............................................. 145.52
Do. after acetylation .................................... 106.5
Saponification No. ..................................... 152.90
Do. after acetylation .................................... 203.24
Iodine Number (Wijs) ................................ 188.3
Molecular weight for monobasic acid calculated from Acid Number .................. 385.

Colour Reaction.—A very striking violet colour reaction was obtained when a drop of ferric chloride solution was added to a dilute alcoholic solution of this acid. A similar coloured precipitate separated on standing.

Molecular Formula.—The following results were obtained on combustion, viz.:—

(1) 0.1068 gram gave 0.2902 gram CO₂ & 0.0919 gram H₂O

C = 74.1%.  H = 9.56%.
(2) 0.1040 gram gave 0.2824 gram CO₂ & 0.0884 gram H₂O
C = 74.0%  H = 9.44%.

(3) 0.1154 gram gave 0.5136 gram CO₂ & 0.0970 gram H₂O
C = 74.6%  H = 9.34%.

C₂₄H₃₆O₄ requires C = 74.23%  H = 9.23%.

Molecular Weight Determination.—A molecular weight determination by the Landsberger boiling point method, using acetone as solvent, gave the following result, viz.:—
1.5416 grams in 21 c.c. acetone elevated the boiling point 0.42°
(average of 8 readings).  M.Wt. = 384
C₂₄H₃₆O₄ required M.Wt. = 388

Silver Salt.—The silver salt was prepared by neutralisation of the acid body with dilute ammonia solution and precipitation with silver nitrate solution. 0.7658 gram silver salt gave on ignition 0.1650 gram silver = 21.55% silver. The silver salt of C₂₄H₃₆O₄ requires 21.82% silver.

Copper Salt.—On trituration of the acid with excess of copper carbonate no action appeared to take place at room temperature, but upon heating at water bath temperature a vigorous reaction resulted. The green copper salt was extracted by means of acetone, and was found upon removal of the solvent to be a very viscous and sticky green paste which would not solidify.

0.5722 gram of copper salt gave 0.0544 gram CuO on ignition = 9.51% CuO.

The copper salt of a monobasic acid of molecular formula,
C₂₄H₃₆O₄ would yield by calculation 9.44% CuO.

Presence of "CO" and "OH" Groups.

The presence of "carbonyl" groups could not be detected by the use of hydroxylamine or semi-carbazone salts. The solubility, colour reaction, and general chemical deportment, so far observed, point to the presence of one
"carboxyl" group and two "hydroxyl" groups in the molecule of this acid. The presence of the latter was demonstrated by the reactions with phenylisocyanate and particularly with napthylisocyanate, but no definite crystalline derivatives could be isolated from the reaction mixtures.

Action of Bromine.—Treatment with bromine at \(-20^\circ\) in both dry ether and carbon disulphide solutions respectively yielded sticky masses which could not be induced to crystallise.

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