UTILISATION OF MYROBALANS.

PART I. PREPARATION AND PURIFICATION OF MYROBALAN EXTRACT.

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Several families of the genus "Combretaceæ" contain trees rich in tannin but the most important commercial species is *Terminalia chebula* Retz, the chebulic or black myrobalans, which is the best known of the Indian tanning materials. *Terminalia chebula* occurs throughout India and Burma as a deciduous tree which varies in size according to the locality and which attains a height of 40 to 50 feet. The nuts contain from 30 to 40% of tannin. The commercial supplies of myrobalans are obtained from forests owned by Government of India or by Native States. There are five main commercial varieties which are known after the districts from which they are marketed: (1) "Bhimlies" or B's from Bhimilipatam in Madras, (2) "Rajpore" or R's from Kolhapur State, (3) "Jubbulpore" or J's from Jubbulpore in Central Provinces, (4) "Vingorlas" or V's from Bombay forests, and (5) "Madras Coast". Each of these different varieties is usually separated into two grades known as No. 1 and No. 2.

Parker and Blockey (*Jour. Soc. Chem. Ind.,* 1903, **22**, 1182) have analysed these different commercial grades of myrobalans. Besides, these have been analysed in the Imperial Institute, London. These investigators have compared these samples for weight-giving properties, for bloom-yielding capacities and for the development of acidity in the tanning liquors. They concluded that J's were the most valuable in weight giving properties. With regard to bloom yielding capacity, J's and V's were found to be distinctly superior to all the others. In the acid tests B's were found to develop the most acidity and J's the least.

The myrobalans are richer in tannin when fully ripe. Reporting on the examination of nine species collected in South Thana, Bombay, Purnasingh (*Indian Forester*, 1911, **37**, 509) states "the fruits that have remained longest on the tree, *i.e.,* those quite ripe, should be classed as the richest in tannin, irrespective of their colour".

Myrobalans are one of the most important tanning materials found in India. When used in leather tanning, the tannin penetrates the hide very slowly and when used alone, yields a soft, mellow and rather spongy leather which does not possess good wearing properties. The myrobalans are, therefore, used in conjunction with the

most astringent and quickly penetrating tannins such as quebracho, wattle bark and in India with "babul", "avaram" and "mangrove barks", and as a bleaching agent at the end of the tannage. One of the principal properties of myrobalans is their acid-forming power. They contain from 3 to 5% of sugar matter, fermentation takes place readily and satisfactory plumping is secured during the early stages of tanning. The ellagitannic acid, on hydrolysis, produces ellagic acid which is the chief bloom-yielding tanning material, and is useful in the production of the sole leather.

In addition to their use for tanning purposes, myrobalans are also used to a less extent in the dyeing industry, as a mordant for the basic aniline dyes. The yellow colouring matter, however, renders them unsuitable for use as a mordant for bright shades on cotton or silk, as the original yellow colour of the mordant makes the shades obtained, dull. Dyers require tannins of the pyrogallol class and the only suitable materials available are sumach and myrobalans. The latter material is generally used because of the higher price of the sumach. Myrobalans are also employed for weighting of black silk.

Besides the above main uses, myrobalans have been used for centuries in the manufacture of writing inks. Even at present their aqueous extract, which is allowed to ferment for days together, is used for making inks in several villages. There is a good demand for the purified tannic acid prepared from myrobalans in the manufacture of ink.

Myrobalans are largely exported either as whole nuts, or, in order to reduce transportation cost, as crushed myrobalans freed from the stones (which contain only about 3 to 5% tannin). Myrobalan extract, in both solid and liquid forms, is also exported. During war and even afterwards there has been a great demand for myrobalan extract in Europe, which was chiefly used for tanning and dyeing. From 1928 to 1932, the export of myrobalan was steadily diminishing, but after 1932, it is gradually increasing. In 1935–36. about 1.5 million cwt. of myrobalans costing about 4.5 millions of rupees and 42,000 cwt. of myrobalan extract worth 4.2 lacs of rupees were exported.

In our investigation, we have employed throughout the variety "picked Rajpore". The nuts were oval shaped, solid in structure and from greenish yellow to brownish colour in section. Light, hollow and black nuts were rejected.

The use of myrobalan in reducing the viscosity of the Rotary Drilling Mud has been investigated in this laboratory, the results of which are under publication. Of all the tannin-bearing materials,

myrobalan powder brings about the greatest reduction in viscosity, the lowest value being only 6% of the original, which remained constant with larger additions of myrobalan. Mehta and Jatkar (*This Journal,* 1935, **18A**, 101) showed that the addition of myrobalan powder changed the pH of the mud in the manner analogous to its effect on viscosity, the range of constant viscosity corresponding to 7.5 pH. And they also showed that the initial effect of adding small quantities of myrobalan was equivalent to the tannic and ellagitannic acid content of the myrobalan which were estimated by electrometric titration. The subject of the electrometric titrations of the various tannic acids and their physical properties has been investigated by us in detail, the results of which will be published in Part IV of this series.

The object of the present investigation was to study the optimum conditions for the preparation and purification of the myrobalan extract with a view to improve the tannic acid content and to utilise the tannic acids for preparation of inks and for use as mordant. The oil from the myrobalan seed has also been examined (*cf.* Part II).

EXPERIMENTAL.

Analysis of Myrobalans.—Myrobalans were crushed in roller grinders to separate the seeds. The percentage of seeds separated was 33%. The soft rind or pulp was ground to pass through a 20-mesh sieve. Extraction was carried out in Procter's extractor. Tannin and non-tannin were determined both by the hide powder method and by the Lowenthal's oxidation method. Results of analysis of myrobalans (without seeds) by the hide powder method are: Moisture 10.0%, Tannin 46%, Non-tannin 17.3%, Insolubles (by difference) 26.7%.

The tannin percentage was 39.7 as calculated from the permanganate required for the oxidation of the tannin matter. Kahlbaum's A.R. tannic acid was used to standardise potassium permanganate. The analysis of this tannic acid was carried out according to the hide powder method adopted by the International Society of Leather Trades Chemists, and gave Tannin 95%, Non-tannin 3.8%, Moisture 1.2%.

A variation was found for the values of tannins by the above-mentioned methods for the same material. However, the volumetric method was preferred because of the quickness and ease of operation. (*cf.* H. R. Procter, *Leather Industries Laboratory Book,* p. 223.)

The question regarding the temperature at which myrobalans should be extracted has been carefully investigated by Parker and

Procter (*Jour. Soc. Chem. Ind.*, 1895, 635). They extracted myrobalans at different temperatures and determined the tannins, non-tannins and colouring matters so extracted. The figures given in their experiments with myrobalans, clearly indicate that the maximum temperature of extraction for getting the maximum yield of tannin is 90°–100°. However, it would be worthwhile emphasising here, that such a high temperature as 90° to 100° C. for the extraction throughout all the leaching vats, has a deteriorating effect on the extract in presence of air, both from the point of view of tannins and colour.

It was found that by keeping the myrobalan extract at 90° to 100° C. exposed to the air for about 5 hours, some yellowish insoluble substance is thrown out, which is soluble in alkali and which proved to be ellagic acid. This substance, being practically insoluble in water, can be hardly used as such. As regards the colour, the pale coloured extract becomes slightly darker in colour, if it is exposed to air at 90° to 100° C. for some time. Moreover, according to the official methods of analysis, the major part of extraction of tannin material is carried out at or below 50° C. in order that no decomposition of the tannin should take place. J. A. Wilson (*The Chemistry of Leather Manufacture*, Vol. 1, p. 410) emphasises this point and states that "the rate at which tannin can be extracted from the raw materials increases with the temperature of the water used, but so also does the rate at which the dissolved matter decomposes. It is customary to extract the fresh material at low temperature and to increase the temperature of extraction until the material is practically exhausted".

In the following experiments, the temperature was maintained at 50° to 60° C. for the first two leachings on the same material and then was increased to 90° C. for the remaining leachings. When initial temperature was maintained higher we found that the extract obtained was turbid and was of colloidal nature, probably due to pectisation. Experiments were carried out in order to determine the time necessary for extraction by taking a known weight of the material and treating with a known weight of water for ½, 1 and 1½ hours at two temperatures, namely, at 50°–60° and at 90° C.

These experiments showed that the leachings at 50°–60° C. should be carried out for an hour-and-a-half and as the temperature is raised to 90° C., the time of leaching can be shortened to an hour. Myrobalan tannins are easily soluble in hot water and the time mentioned above is sufficient to extract tannin by diffusing through the porous cell walls.

Quantity of water required for leaching was determined by extracting for six times, the known weight of myrobalans with 2½, 5 and 7 times the quantity of water and analysing the liquors by the

Lowenthal's oxidation method by permanganate solution and by determining the total soluble solids in each liquor obtained after leaching.

Relation between Specific Gravity and the percentage of soluble matter in the Myrobalan Extract.—In order to estimate quickly and fairly accurately the total soluble matter contained in the myrobalan extract obtained in successive leachings and hence to follow closely the process of extraction, it was found convenient to determine the relation between (1) Sp. Gr. and temperature and (2) Sp. Gr. and percentage of total soluble matter. The following table gives the variation of Sp. Gr. with temperature.

TABLE A.

1		2		3		4	
Temp. °C.	Sp. Gr.	Temp. °C.	Sp. Gr.	Temp. °C.	Sp. Gr.	Temp. °C.	Sp. Gr.
26	1·0485	26	1·0295	26	1·082	26	1·058
32	1·0460	28	1·0290	30	1·080	32	1·056
36	1·0445	41	1·0275	43	1·0745	38	1·053
46	1·0415	45	1·0245	53	1·070	45	1·050
55	1·037	50	1·0205	73	1·059	53	1·046
64	1·032	57	1·0185			66	1·040
73	1·027	61	1·0165			77	1·033
		69	1·012				

The following table shows the variation of the percentage of the total solubles with Sp. Gr. at 26° C.

TABLE B.

Sp. Gr.	% Soluble matter	Sp. Gr.	% Soluble matter
1·128	31·5	1·046	11·6
1·103	24·3	1·037	9·4
1·084	21·0	1·031	8·4
1·072	17·7	1·032	5·9
1·062	15·3	1·010	3·4

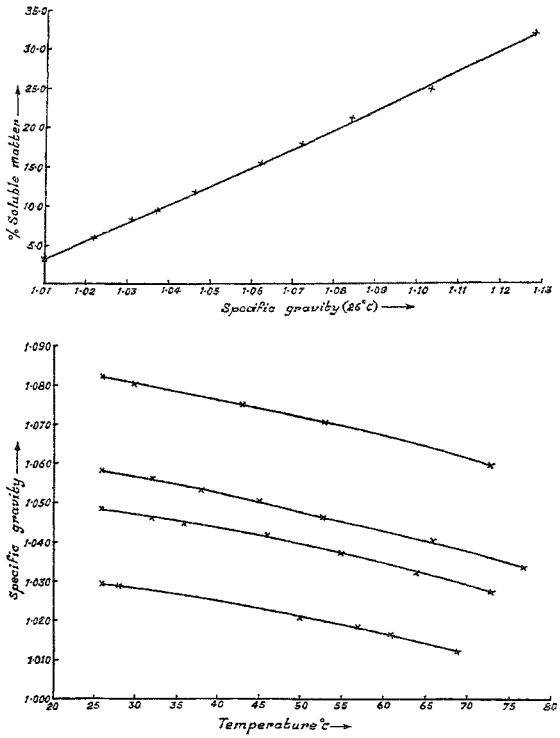

FIG. 1.

The results, when plotted (Fig. 1), showed a linear variation of Sp. Gr. with percentage composition. The Sp. Gr. also varied linearly with temperature.

EXTRACTION OF MYROBALANS.

300 grams of the coarsely ground myrobalan powder were treated with 750 c.c. of water in a beaker. For the first three leachings, the temperature was kept at 50° to 60° for one hour and a half each, and for the remaining leachings, the temperature was raised to

90° C., and kept for an hour. After the first leaching, the whole was filtered through Buchner funnel. The residue was treated again in the same way with an equal quantity of fresh water. Small particles that passed through the holes of the funnel were collected as sediment and analysed separately for tannins and soluble matter.

The following Tables I, II and III show the results of the extraction of myrobalans with 2½, 5 and 7½ times water respectively.

TABLE I.

Weight of myrobalan powder taken = 300 grams.

No. of leachings	1	2	3	4	5	6	Total
Liquors obtained in successive leachings c.c. ..	500	500	500	500	500	500	3 litres
Total solubles in the liquors obtained	77·5	35·0	20·5	9·5	6·0	4·0	152·5 gms.
Total solubles in 100 c.c. (gms.)	15·5	7·0	4·1	1·9	1·2	0·8	
Amount of tannin in 100 c.c. (gms.)	10·1	5·1	3·3	0·97	0·6	0·4	
Amount of tannin in liquors obtained	50·5	25·5	16·5	4·8	3·0	2·0	102·3 gms.

Soluble matter in the spent material = 6·5%
Soluble matter obtained .. = 50·8%
Tannin obtained .. .. = 34·1%

TABLE II.

Weight of myrobalan powder taken = 150 grams.

No. of leachings	1	2	3	4	5	6	Total
Liquors obtained c.c. ..	550	550	550	550	550	550	3·3 litres
Solubles in 100 c.c. (gms.) .	7·2	3·8	1·8	0·95	0·43	0·25	
Total solubles in liquors obtained (gms.) ..	39·6	20·9	9·9	5·2	2·1	1·1	78·8 gms.
Tannin in 100 c.c. (gms.) ..	5·6	2·2	1·0	0·52	0·19	0·16	
Tannin in liquors obtained ..	30·8	12·1	5·5	2·8	1·1	0·88	53·18 gms.

Soluble matter in the spent material = 5·8%
Soluble matter obtained .. = 52·5%
Tannin obtained .. .. = 35·4%

TABLE III.

Weight of myrobalan powder taken = 100 grams.

No. of leachings	1	2	3	4	5	6	Total
Liquors obtained c.c. ..	650	650	650	650	650	650	3·9 litres
Solubles in 100 c.c. (gms.) ..	5·8	1·7	0·56	0·22	0·1	0·08	
Total solubles in liquors obtained (gms.) ..	37·7	11·05	3·64	1·43	0·65	0·52	55 gms.
Tannin in 100 c.c. (gms.) ..	3·8	1·1	0·48	0·14	0·08	0·05	
Tannin in liquors obtained (gms.)	24·7	7·15	3·12	0·91	0·52	0·32	36·72 gms.

Soluble matter in the spent material = 4·5%
Soluble matter obtained .. = 55·0%
Tannin obtained .. .. = 36·72%

It can be observed in the experiments of extractions that there is a considerable loss in the total soluble matters obtained, due to the decomposition and the precipitation of the difficultly soluble tannins in the vats. At the dilutions used in the analysis, such difficultly soluble matter remains in solution, but at the higher concentration of the vat-liquors, it comes down in the form of precipitate and settles as a sludge. Moreover, in the extraction of materials in a series of leaches, the cooling effect on the liquor containing 12 to 15% solids will cause further precipitation of tannin.

The following results show the loss of soluble matter in the extraction of myrobalans.

TABLE IV.

	Experiment Numbers		
	1	2	3
	%	%	%
Total extractable matter in myrobalans	63·3	63·3	63·3
Total extractable matter in the liquors obtained	50·8	52·5	55·0
Total extractable matter in spent material	6·5	5·8	4·5
Loss			
% loss of soluble matter calculated on total solubles ..	9·5	8·0	6·0

The analytical results of the last three experiments are summarised in Table V.

TABLE V.

	Experiment Numbers		
	1	2	3
Number of leachings .. .	6	6	6
Total liquor obtained calculated on 100 grams of powder (in litres)	1·0	2·2	3·9
Solubles in 100 c.c. of the last leaching (gm.)	0·8	0·25	0·08
Tannin in 100 c.c. of the last leaching (gm.)	0·4	0·16	0·05
% Soluble matter obtained ..	50·8	52·5	55·0
% Soluble matter in the spent material	6·5	5·2	4·5
% Soluble matter in the material ..	63·3	63·3	63·3
% Tannin obtained in the extract ..	34·1	35·4	36·72
% Tannin in the material ..	39·7	39·7	39·7

The following table shows the exhaustion of myrobalan pulp powdered coarsely and indicates the tannin percentage in different liquors passing successively through myrobalan powder.

TABLE VI.

No. of washings	Experiment Numbers		
	1	2	3
1	10·1	5·6	3·8
2	5·1	2·2	1·1
3	3·3	1·0	0·48
4	0·97	0·52	0·14
5	0·6	0·19	0·08
6	0·4	0·16	0·05

The previous experiments show that six washings suffice. Supplementary washing continued on the sixth liquor in Experiment 3, *i.e.*, 7th washing, showed tannin percentage 0.03, which shows that working with seven waters and "a fortiori" eight is useless in commercial extraction.

Next two experiments deal with the extraction in a battery system of six vats.

Experiment No. 4: 100 grams of myrobalan powder were taken separately in 6 beakers with 5 times the quantity of water. To the first beaker 500 c.c. of water was added and to the others only 125 c.c. of water was added. No. 1 beaker was kept at 50° to 60° C. for an hour and a half, stirring every now and then. This liquor was then transferred to No. 2 beaker which was also kept at 50° to 60° C. for an hour and a half and the whole thing was filtered as above. This liquor was again transferred to No. 3 beaker and so on, continued up to the No. 6 beaker. Here it was leached for the first time for an hour and a half, at 50° to 60° C. and after filtering was withdrawn to the storage vessel. 375 c.c. of liquor of Sp. Gr. 1.100 were obtained.

After transferring the first liquor to No. 2 beaker, fresh water equal in quantity that was withdrawn was added to No. 1 beaker, every time after each withdrawal. When the liquor from the sixth beaker was withdrawn, material in No. 1 beaker was washed six times. This exhausted material from first beaker was removed and it was charged with 100 grams of fresh material.

After this the liquor from No. 6 was transferred to No. 1 containing fresh material and from No. 5 to 6 and so on. At this stage, No. 2 was leached with fresh water for the seventh time. After the seventh leaching of No. 2 beaker, the exhausted material was withdrawn and it was filled again with fresh material. Liquor from No. 1 in which fresh material was filled and which was leached by liquor from No. 6 for the first time was withdrawn to the storage vessel and No. 2 emptied for the first time. This was continued till 6 liquors were withdrawn from all the beakers after passing through fresh material for the first time. Every time fresh water for leaching was used only on the exhausted material and the concentrated liquor before withdrawal passed through the fresh material for the first time. Average Sp. Gr. of the liquors collected was 1.09. Total quantity of liquors obtained was 2.25 litres, which contained 490 grams of solids.

Experiment No. 5: This experiment was carried out in the same way as above, but with 7½ times the quantity of water instead of 5 times. Average Sp. Gr. of the liquor was 1.065 (mean of 6

liquors). Quantity of liquors obtained was 3.6 litres and contained 576 grams of solids. The results of the experiments are given below:—

TABLE VII.

	Experiment Numbers	
	4	5
Number of leachings	6	6
Liquors obtained (litres)	2·25	3·6
Average Sp. Gr.	1·090	1·065
Total extract obtained (gms.) ..	490·0	576·0
Solubles in 100 c.c. of the last leaching (gms.)	0·2	0·08

COOLING THE EXTRACT.

In the manufacture of tannin extracts, it has been found advisable to cool the liquors to 5° C. before concentrating. It is not, however, known how far this cooling affects the tannin content and the total yield of the solid extract. We found that the liquor was more and more clarified as the temperature was lowered, depending upon the concentration of the liquor. The liquor having a Sp. Gr. about 1.040 at 25° deposited a small quantity of yellowish precipitate at 15° C. The liquor at this stage, though slightly colloidal, was clearer than the original. At 10° C. there was a distinct change in colour, and a greater amount of yellowish precipitate was thrown out, which gathered at the bottom as a reddish sticky mass when the temperature was lowered to 5° C.

Myrobalan extract of Sp. Gr. 1.070 was prepared and divided into four portions of 100 c.c. each. (1) The original extract cooled to room temperature 25° C. (2) The same extract cooled to 15° C. and filtered. (3) The same extract as in (2) but cooled to 10° C. (4) The same extract as in (2) cooled to 5° C.

Tannin and non-tannin were determined by Lowenthal's oxidation method. The colour was determined by Lovinbond Tintometer in ½% solution in 1 cm. cell and expressed in units of Red and Yellow. The following table shows the results:—

TABLE VIII.

	Experiment Numbers			
	1	2	3	4
Temperature .. .	25°	15°	10°	5°
Tannin in 100 c.c. (gms.) ..	9·95	9·6	8·66	7·32
Ratio tannin/non-tannin ..	3·87	4·4	3·97	3·47
% loss of tannin	..	3·51	13·0	26·4
Total solubles (grams) ..	17·6	17·0	16·2	14·1
% loss of yield	..	3·4	8·0	20·0
Tintometer reading—				
Yellow	4·0	3·0	1·9	1·9
Red	1·0	0·9	0·2	0·2

The ratio tannin/non-tannins is calculated from the quantities of permanganate required to oxidise tannin and non-tannin matters.

DECOLORISATION AND CLARIFICATION OF THE EXTRACT.

The decolorisation of the tannin extracts is a matter which has received considerable attention and has been the subject of numerous patent specifications. Generally, liquors which have been obtained by extracting at higher temperatures almost invariably require decolorisation by the addition of some substance which precipitates a small portion of tannin, but which carries down with it much of the colouring matter. This loss of tannin can be avoided by careful extraction at moderate temperatures and this is specially to be aimed at in the case of strong tanning materials, which can easily yield liquors of much greater strength than 1.030 Sp. Gravity.

For tanning, British and German tanners prefer the crude extracts simply clarified by mechanical means, which ensures uniformity in the products and especially a higher percentage of tannin and consequently a higher yield in leather. This is specially true of "Bloom" yielding tanning materials such as myrobalans. They contain ellagitannic acid which, on hydrolysis, produces ellagic acid which is technically known as "Bloom". Tannic acid used in the dyeing process as a mordant for fixing the basic dyestuffs must be pure and colourless. With this object in view, the following experiments were carried out.

A number of substances were tried for decolorising, such as, charcoal, fuller's earth, sulphur dioxide, etc., but no appreciable

change was noted. Sulphur dioxide has little action on the colour of the extract which becomes slightly paler, but on exposing to air again the colour reappears. In one case it was observed that when the extract was kept saturated with sulphur dioxide, a yellow substance was thrown out which was later identified as ellagic acid.

The following experiment was carried out to find the action of the alumina cream in decolorising and clarifying the myrobalan extract. Alumina cream was prepared from potash alum by precipitating with sodium carbonate and thoroughly washing. The alumina cream contained 30% of dry Al_2O_3. Myrobalan extract of 1.025 Sp. Gr. was prepared and divided into four portions, and alumina cream was added to all but the first portion, in the proportion 3.5, 7 and 14 c.c. per 100 c.c. of the extract respectively. On analysing the solutions by Lowenthal's method, the following results were obtained:—

TABLE IX.

	Experiment Numbers			
	1	2	3	4
Alumina cream added c.c. ..	..	3·5	7	14
Ratio tannin/non-tannin ..	3·3	2·7	2·4	1·7
Tannin (gms.)	3·1	2·45	1·85	1·5

It is clearly seen from the above that the ratio of tannin/non-tannin goes on decreasing progressively as the quantity of alumina cream added goes on increasing. Alumina removes more tannin than non-tannins. Tannins removed in (4) are practically 50% of the values in (1). Considering the loss of tannin matter, this reagent is not worth trying on commercial scale.

PURIFICATION OF TANNIC ACID.

The following experiments were carried out with the object of purifying myrobalani tannic acid by fractional precipitation with lead acetate.

Myrobalan extract containing 5% solubles was treated with varying quantities of 5% lead acetate solution. The precipitate was filtered and washed. The first fraction was rejected and the middle fraction obtained by adding varying quantities of 10% lead acetate solution, was decomposed with dilute sulphuric acid, filtered and washed. The analysis was carried out by the volumetric method. Tannins and non-tannins are expressed in terms of oxalic acid equivalent of permanganate required. The results are given in Table X.

TABLE X.

	Experiment Numbers										
	1	2	3	4	5	6	7	8	9	10	11
Lead acetate solution used in first fraction c.c.	..	5·0	7·5	10·0	15·0	5·0	7·5	10·0	5·0	5 0	5·0
Lead acetate solution used in middle fraction c.c.	..	40	40	40	40	45	45	45	40	50	60
Tannins (gms.)	1·04	0·76	0·74	0·73	0·737	0·85	0·81	0·765	0·76	0·95	0·95
Non-tannins (gms.)	0·27	0·143	0·143	0·143	0·145	0·16	0·16	0·16	0·143	0·19	0·227
Ratio tannin/non-tannin ..	3·8	5·3	5·2	5·2	5·1	5·3	5·1	4·8	5·3	4·7	4·2
Yield	5·0	4·0	..	3·9	3·7	..	4·0	3·8	4·0	4·4	4·7

It is seen from the table that there is a minimum quantity of lead acetate solution that can be added in the first fraction, which will precipitate more non-tannins than tannins. In the middle fraction proportionately more non-tannins are precipitated with increasing quantities of lead acetate solution than tannins (Expts. 9–11).

Preparation of Myrobalan Extract on Large Scale.

As in the case of other tannin materials, the extraction of tannin from myrobalans is best effected in open leaches instead of in autoclaves. The leaching vats were made of copper, cylindrical in shape, 2 ft. × 2 ft., and could hold 40 lbs. myrobalans, and were provided with false bottom. The liquor after extraction was withdrawn through taps fitted near the bottom on the sides. The heating of the liquor was carried out by steam at atmospheric pressure (97° C.) passing through a lead coil 30 ft. in length and ½ inch in diameter. About 15 minutes were required to raise the temperature of the liquor from room temperature 25° to 90°C.

Using 30 lbs. of myrobalans in each of the two vats, the following results were obtained. Temperature of extraction for the first two leachings was maintained between 50° to 60° C. and for the next five extractions was raised to 80° to 90° C. Time of extraction was 1½ hours for the first two leachings and one hour for the next five. Quantity of water used was four times the quantity of myrobalans.

Table XI.

Leachings	Vat 1 % soluble matter	Vat 2 % soluble matter	Total extract obtained from 60 lbs.
			lbs.
1	6·5	9·2	9·2
2	2·5	6·0	5·7
3	1·4	3·8	3·0
4	0·8	3·0	1·8
5	0·5	1·3	1·0
6	0·35	0·5	0·5
7	0·18	0·25	0·2

Total yield of extract .. .. = 35.6%
Soluble matter in myrobalans .. = 46%
„ „ spent material .. = 6%

The turbid liquor from the vats was filtered through a bronze filter press of six plates using thick canvas and kieselguhr as a filtering aid. When the extract was allowed to settle overnight, it was found that there was a good quantity of yellow solid deposit at the bottom of the vessel, consisting of finely divided particles of the material and of ellagic acid which is technically known as "bloom" in leather industry.

The clear filtered liquor was concentrated in the laboratory vacuum climbing and falling film copper evaporator shown in Fig 2. The liquor is drawn in by vacuum and passes through the evaporating tube and is continuously discharged from the separator, concentrated to the required density.

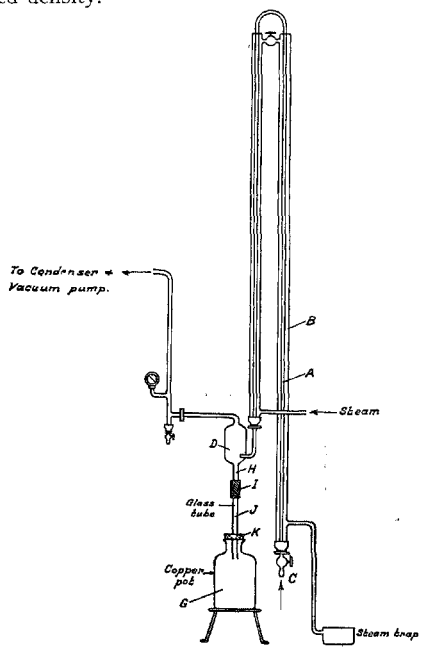

FIG. 2.

The evaporator tube A, 1" in diameter, is surrounded by a jacket B containing steam as a heating medium; one side of the

evaporating tube is 10 ft. long, while the other is 8 ft. The liquor is sucked in by vacuum at C, then passes through the tube which is heated by steam, rises to the top, then ascends through the bend and comes down through the other side of the evaporator tube to the separator D, from where the concentrated extract flows down into the receiver G through the tube H which is connected to the glass tube J by means of rubber tubing I. The glass tube serves the purpose of sight glass and it passes through the rubber stopper K fitting tightly to the neck of the receiver. The object of the separator is to remove the vapour from the concentrated extract. To accomplish this, the velocity of the liquor itself is used, by means of centrifugal action, to make the separation of the vapour from the liquor. The vapour escaping is condensed in the condensor. It was found convenient to carry out the concentration in two stages, first to a strength of 1.12 Sp. Gr. and second to 1.270 Sp. Gr.

The evaporating capacity of this tube is about 5 gallons of water per hour at a steam pressure of 15 lbs. gauge under a vacuum of 55 cms. of mercury.

The thick extract was further dried in a vacuum shelf drier under a vacuum of 65 cms. of mercury. The concentrated extract was spread in trays and heated to 60° C. The product contained 6% moisture. From 100 lbs. of myrobalans 620 lbs. of thin extract Sp. Gr. 1.030 were obtained, which on concentrating yielded 76 lbs. of thick extract Sp. Gr. 1.270 and yielded 38 lbs. of dry powder showing a recovery of 83%.

SUMMARY.

The optimum conditions for the extraction, clarification and decolorisation of tannic acid from myrobalans have been studied and applied to large-scale experiments. By treating myrobalan extract with alumina, more of tannins were removed than non-tannins, although the colour of the product was quite satisfactory. Fractional precipitation with increasing quantities of 10 per cent. lead acetate solution and decomposition with sulphuric acid showed that the ratio of tannins to non-tannins is approximately the same in all but the first precipitate. Extraction with battery of six vats shows that most of the tannins are extracted by water at 70°, with about seven times the quantity of myrobalan. Cooling the extract to 15° caused the separation of a good deal of colloidal matter without appreciable loss of tannins.

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