UTILISATION OF TAMARIND.

By F. Marsden, Ph.D., M.Sc.

An inquiry into the commercial prospects of manufacturing tartaric acid from tamarind, conducted by Sudborough and P. N. Vridhachalam [1] showed clearly that with the price of the acid at about 4s. per lb. the prospects were good. In 1921, the suitability of fruits grown in the Kistna District of Madras arose and a quantity of tamarind was received from that area for the determination of the acid content.

The material differed from that examined by Sudborough and Vridhachalam in that it was not cleaned pulp, but a mass of the dry compressed fruit containing husk and 'nerve', and was therefore in the condition suitable for market in bulk.

Extracted with boiling water, filtered through cloth, worked up, and analysed by the method given by Sudborough, the material showed an acid content of 9·55 per cent. tartaric acid. During the course of analysis a quantity of tamarind had been set aside with cold water and in a day or two showed signs of a vigorous fermentation. This was allowed to proceed in the hope that the glutinous matter, which renders the washing of the pulp and complete extraction of the acid so tedious, would be broken down and thus obviate the use of an autoclave for commercial treatment. The fermentation was apparently due to yeast organisms carried by the tamarind. These were able to function in the presence of tartaric acid when the extract was diluted (1 part tamarind to 3 parts water) and thus convert the sugar contained in the extract into alcohol. The fermentation seemed to proceed best at a temperature of about 40° C and was complete in 3–4 days. Distillation tests showed that alcohol yields of 12-14 per cent. on the weight of the tamarind taken could be obtained when working on a small scale.

The expectation that the fermentation would disintegrate the glutinous matter was not, however, realised, as the nature of the pulp remained unchanged and it was difficult to extract the last traces of acid by washing with hot water.

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[1] This Journal, 1920, vol. 3.

The effect of previous disintegration of the mass, including seeds, was tried by passing the tamarind through a mincing machine, but though it appeared that by an extended washing a little more acid was extracted, the process was too tedious and filtration slow, even when diatomaceous earth (combined with pressure filtration) was employed as an assistant, but the experiments showed that after fermentation there was, in all cases, a slightly higher yield of tartaric acid. Thus :—

Acid obtained by boiling the broken material	9·55 per cent.
,, ,, ,, ,, ,, minced material	10·1 ,, ,,
,, ,, ,, ,, ,, broken material and filtering at pump	12·1 ,, ,,
,, ,, ,, fermenting and washing the broken material	10·0 ,, ,,
,, ,, ,, fermenting and washing the minced material	10·4 ,, ,,
,, ,, ,, fermenting and washing the broken material (pressure filtration)	12·5 ,, ,,

It appeared from these experiments that with this material the total acid contents were not easily and completely removed by direct washing with an economic quantity of water, but that the main proportion (about 4/5ths) could be removed by using a volume of cold water equal to some five times the weight of the tamarind taken. A battery of three percolators was therefore set up ; these were filled in rotation and water passed through intermittently. The extract after passing through the most recently filled vessel was removed when it amounted to 3½ times the weight of the tamarind charged into each percolator. After some days when the extraction was working regularly, the extract was analysed ; 200 grams tamarind was the weight of the refilling charge and 700 cc. of extract was taken after passing through vessel No. 3. After fermentation, the alcohol was distilled off and was found to amount to 11·5 to 12·5 per cent. on the weight of the tamarind, whilst the tartaric acid was equivalent to 11·7 per cent.

A large scale experiment was then made with a fresh sample of tamarind analysing 14 per cent. tartaric acid, with the object of separating a quantity of acid in the pure state. 20 lbs. (=9,000 grams) were extracted in the cold until 32 litres of extract had been collected. This was allowed to ferment, distilled to recover the alcohol, and then precipitated with lime, finally completing the precipitation with chalk. The extract was dull brown and the calcium salt obtained appeared a little darker than that obtained by Sudborough and Vridhachalam.

It was decomposed with sulphuric acid in the manner described by them, except that instead of adding the salt to the measured volume of diluted sulphuric acid the calcium tartrate was made into a paste with water and the diluted acid stirred in until the decomposition was completed. Further, in place of the method for detecting the end-point given by them, I preferred to use a spotting test with methyl violet paper, methyl violet being unaffected by organic acids but changing colour (to green or yellow) with free mineral acid according to the excess and dilution of the mineral acid. A little experience suffices to decide from the colour change when the correct excess of sulphuric acid has been added, and the final test can then be made in the manner described by Sudborough, using sodium hydrate solution equivalent to 0·0797 grs. tartaric acid per cc. and barium chloride solution equivalent to 0·005 grs. sulphuric acid per cc.

Working in this way, the calcium tartrate was effectively decomposed, but instead of the coloured matters, which had been precipitated with the calcium tartrate, remaining behind on filtration in association with the calcium sulphate, they passed into solution with the tartaric acid and the crystals of the latter obtained upon evaporation were too deeply coloured to permit of a pure acid being recovered except at great expense. The mother liquors also were deeply coloured and difficult to manipulate.

The tartaric acid liquors obtained by Sudborough were very light in colour and the question arose whether the deeply coloured liquors obtained by me were not due to the husk present in the raw material. I therefore autoclaved a quantity under 50 lbs. pressure and obtained a clear, bright brown extract from which a calcium salt and finally tartaric acid of good colour could be made.

I therefore extracted a fresh quantity of tamarind with cold water, fermented the solution and distilled off the alcohol produced. The residual liquor was then autoclaved under low pressure (35 lbs.). Upon cooling, the extract was still dull coloured and deposited a little dark coloured sludge, and a sample precipitated with lime gave a dark coloured calcium salt. The bulk of the extract was filtered through paper and again autoclaved for one hour at 50 lbs. pressure. This resulted in a heavy brown deposit being formed which filtered easily, and the filtrate was then bright brown and gave a calcium salt and tartaric acid of good colour.

It is evident therefore that autoclaving under a fairly high pressure is essential for efficient isolation of tartaric acid from tamarind, and my experiments show that the material can be effectively extracted in the cold and the alcohol recovered. The extracted tamarind pulp, together with the residual liquors, yield a quantity of potassium hydrogen tartrate when autoclaved, and, after removal of this, yield calcium tartrate of good quality when precipitated with lime.

500 grs. tamarind treated in this way (autoclaving at 160° C = 75 lbs. pressure) gave :—

Alcohol ...	... 55 grs.
Potassium hydrogen tartrate ...	7 grs.
Calcium tartrate ...	... 95 grs.

My experiments have thus far demonstrated that :—

(1) Autoclaving at 60-70 lbs. pressure is essential for the satisfactory treatment of tamarind for tartaric acid recovery.

(2) Alcohol in appreciable quantity can be produced from the material by permitting the cold aqueous extract to undergo fermentation.

(3) Filtration is improved if, after distilling off the alcohol from the fermented liquor, the residual acid liquor is autoclaved together with the extracted tamarind residue.

(4) By this process of fermentation and distillation, the production of potassium hydrogen tartrate and calcium tartrate of good quality is not impeded provided the extracted tamarind and the extract are autoclaved at about 160° C. In fact it appears that a slightly improved yield of calcium tartrate is achieved in consequence of the preliminary fermentation.

(5) Tamarind as usually marketed contains 12-14 per cent. of tartaric acid, reckoned as free tartaric acid, and that for every 1,000 lbs. of tamarind 15-19 gallons of 96 per cent. alcohol should be recoverable besides the salts of tartaric acid (=14 lbs. of crude potassium hydrogen tartrate and 190 lbs. of calcium tartrate).

THE PROSPECTS OF COMMERCIAL UTILISATION

In 1919 the selling price of tartaric acid was about 4*s*. per pound, Sudborough and Vridhachalam calculating that with tamarind costing Rs. 58-12-0 per 1,000 lbs., a profit of Rs. 30 per 1,000 lbs. of tamarind might be expected. Since then the rates for tartaric acid have declined considerably, being about 1*s*. 4*d*. per lb. in July, 1922, so that the maximum price paid for tamarind must be less than Rs. 50 per ton in order to cover the working costs. In the northern districts of the Madras Presidency, where tamarind is marketed in bulk, the selling rates are Rs. 60-80 per ton, so that there is little prospect of any manufacture of tartaric acid alone being profitable. The outlook is brighter if account is taken of the recoverable alcohol, and if the acid contents of the fruit are converted into calcium tartrate and this salt marketed. The yield per ton of tamarind would be :—

Calcium tartrate	.... 420 lbs.
Potasssium hydrogen tartrate	.... 30 lbs.
Alcohol (96 per cent)	.... 30-40 gallons.

With 1,000 lbs. of tamarind as the unit the cost of materials might be taken as :—

	Rs	A	P
Tamarind, 1,000 lbs.	50	0	0
Lime (free from magnesia) 40 lbs.	1	0	0
Fuel	2	8	0
Total	53	8	0

With tartaric acid selling at 1*s.* 4*d.* per pound the value of calcium tartrate would not exceed 6*d.* and we might have to deal on the basis of a works' selling price of As. 4 or As. 5 per lb.

Pure potassium hydrogen tartrate would approximate to As. 12, and As. 7 to 8 might be expected for the crude product. Alcohol prices run from Rs. 2.5 to 3 per gallon, but for power purposes it would be required at a lower rate and I prefer to adopt a value of Re. 1 per gallon. Upon this basis, the return from 1,000 lbs. of tamarind would be :—

	Rs	A	P
Calcium tartrate, 190 lbs. @ As. 4	47	8	0
Acid potassium tartrate, 14 lbs. @ As. 7	6	2	0
Alcohol, 15-20 gallons (about)	15	0	0
Total	68	10	0

The cost of materials, therefore, works out at Rs. 53-8-0 and the value of the products obtained Rs. 68-10-0, equal to a balance of Rs. 30 per ton of tamarind treated. This return is only about one-half that anticipated by Sudborough and Vridhachalam owing to the lower values which must now be assumed for the tartaric acid products ; and even this is only possible by taking into account the value of the alcohol recovered, the fact that the whole fruit is taken and not the cleaned pulp only, and by marketing the product at the calcium tartrate stage instead of proceeding to the manufacture and marketing of the pure acid.

A factory working upon this material alone would need to be on a large scale for efficient working and alcohol recovery; it would require to operate some 400-500 tons of tamarind per month. Difficulties would most probably arise in the supply of the materials at sufficiently low rates as a largely increased demand for the raw material would immediately raise its cost; but the manufacture should be remunerative on a smaller scale if worked in conjunction with a distillery which could undertake the extraction of the tamarind along with fermentation and distillation of the extract, delivering the alcohol-free liquors and the extracted tamarind to a properly equipped factory adjacent to the distillery, there to be autoclaved and worked up for the tartaric acid.

As a source of sugar for alcohol production, tamarind at Rs. 60 per ton would appear comparable with molasses at Rs. 20 per candy of 500 lbs., the latter being an ordinary market rate.

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