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THE PRODUCTION OF LINE OIL AND CALCIUM CITRATE IN THE PROVINCE OF BOMBAY.

INTRODUCTION.

Lime (*Citrus medica*, acida), which is erroneously known as lemon throughout this country, is indigenous to India and is grown in almost all parts of the country. In the Bombay Province the area under cultivation for this fruit at present (1938), is about 1,200 acres. The fruit is generally used in the preparation of beverages and also finds a place in the Indian household as a source of food acid.

2. Limes are available throughout the year. They are, however, harvested in abundance twice a year. The season that yields the largest crop in the Bombay Province commences about the beginning of July and ends by the first week of September. Another important season is in the month of December. During the pick seasons the price of limes sometimes declines to such an extent that the growers often do not realise even the bare cost of picking, packing and transporting the fruit to the markets. If there are heavy rains, as is usually the case in the pick season between the beginning of July and the end of August, it is not possible to transport limes from plantations situated at some distance from railway stations to places where there is a good demand for them, i.e., to places like Bombay, Poona, Surat, Ahmedabad, Baroda, etc. The growers have, therefore, to sell their harvest at very cheap or nominal rates. Sometimes tons of limes are thrown away in big ditches as waste.

3. A preliminary investigation^{*} by us showed that the Bombay limes yield on an average about 53 per cent. juice and the remainder consists of rind, seed, pulp, etc., on a laboratory scale. The total acidity of the juice in terms of citric acid varies between $6\cdot 6$ and 7 per cent. while the citric acid content is found to vary from $6\cdot 3$ to $6\cdot 6$ per cent. The vitamin C (ascorbic acid) content of the fresh juice is about $0\cdot 3$ mg. per c.c.

4. On large scale extractions one ton of limes yield about 1,050 lbs. of juice. Distillations of the juice and the pulp prepared out of the rinds carried out in the laboratory showed that it would be possible to obtain not less than 0.18 per cent. of lime oil on the weight of limes, i.e., 4 lbs. per ton of limes treated.

5. No serious attempt it appears has so far been made in the Bombay Province and probably in other parts of India, for the utilisation of cheap limes by converting them into products like lime oil and calcium citrate. This investigation was, therefore, undertaken in order to find out ways and means of utilising limes for the production of marketable commodities and to work out sufficient data that would eventually enable the owners of lime plantations to put up commercial plants for the production of the same.

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^{*}Messrs. B. B. Sardeshpande and V. C. Amin, former scholars in technical chemistry of the Department of Industries, Bombay, also did a considerable amount of the preliminary work under Dr. Patel which justified further detailed work on the problem.

CITRUS PRODUCTS.

6. There are three important products made out of citrus fruits which are on the international market. These products are :--

- (1) Citrus juices like Lime juice, Lemon juice, Grape fruit juice, Orange juice etc.
- (2) Oils of citrus fruits like Lime oil, Lemon oil, Orange oil, Mandarin oil and Bergamot oil.
- (3) Calcium citrate from the juice of limes and lemons.

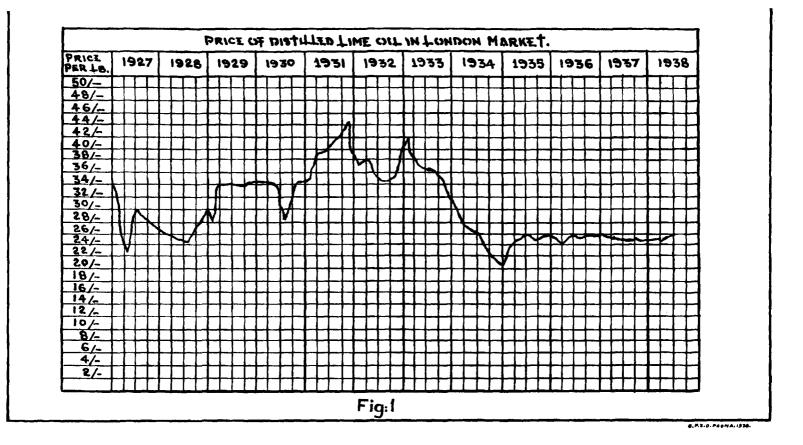
7. Citrus juices.—These are sold in the market in two forms. One of them is the raw juice which is prepared from limes, lemons and oranges and is used for making beverages. Another form of juice is the concentrated juice prepared out of limes and lemons. This is mostly produced at places where it is impracticable to make calcium citrate owing to the absence of suitable lime or chalk or through other reasons. Concentrated juice is used not only as a source of the citrate of lime and ultimately of citric acid, but is also employed sometimes directly for certain industrial purposes.

The Department of Agriculture of this Province seems to have done considerable amount of work on the production and preservation of citrus juices. The methods of production of citrus juices have, therefore, not been gone into by us.

8. Citrus oils.—These are important commodities in the international trade. They are used in the manufacture of flavouring materials for foods and beverages. The processes for the production of citrus oils can be divided into two groups : i.e., the cold and the hot process. In the cold process the oil is separated from the rind of the fruit by mechanical means while in the hot process it is separated by distillation.

9. There is a great demand for Lime oil both in the United Kingdom and on the continent of Europe as well as in the United States of America. Most of the present demand for this oil is supplied by the West Indies. The price of distilled lime oil in London market varies considerably from year to year. During the last ten years it has varied from 44 shillings to 21 shillings per pound. The graph in Fig. 1 shows the retail price of distilled lime oil purchased in bottles in London market during the last ten years. The price realised for oil packed in tins, C.I.F. European ports, is considerably less than this figure.

10. Calcium Citrate.—Lime and lemon juices were a few years ago the most important sources of citric acid. The first operation for the production of citric acid from citrus juices consists of treating the hot juices with good quality of slaked lime. Calcium hydroxide in the slaked lime combines chemically with citric acid forming calcium citrate which is very sparingly soluble in hot water and therefore settles down at the bottom. Citric acid is subsequently prepared from calcium citrate by treatment with sulphuric acid and is purified by re-crystallisation. Processes have recently been developed for the manufacture of calcium citrate produced by the fermentation of sugar with certain moulds. Still fairly large quantities of citric acid are manufactured from calcium



citrate prepared from the juices of citrus fruits. Table I shows the imports of calcium citrate into the United Kingdom from 1931 to 1936 :---

TABLE I.

*											
	1931.	1932.	1933.	1934.	1935.	1936.					
			Quantity	in tons.							
British countries		14 444									
		· Va	ue in tho	usand pour	nds.						
British countries	120.3	0.8 102.4	0·4 77·0	0.8 64.4	1·0 47·0	0·3 14·8					
	Average value in pounds per ton C.I.F. U.K. Port.										
	55.4	35.3	21.2	23.5	25.9	32.9					

Imports of Caloium Citrate into the United Kingdom.

From Table I it will be seen that most of the imports of calcium citrate into the United Kingdom are from countries outside the British Empire.

11. There is a fairly good demand of citric acid in this country. Table II shows the imports of citric acid in bulk into India during the last five years :---

TABLE	Π.
-------	----

	Year.			Value in Rupees C.I.F. Indian Ports.	Quantity in Tons.	Equivalent in Calcium Citrate Tons.
1933-34		••	••	89,000	91	152
193435	••	••		123,000	132	220
1935-36	•••	••		141,000	121	2 02
193637	••	••		84,000	77-	128
1937– 38	••	••		138,000	129	215
	Average	for five years		115,000	110	183

Imports of Citric Acid into India.

Besides the figures shown in Table II, appreciable amount of citric acid is imported in small packages, which are included in the Custom Returns as other sorts of chemicals. The values in the above table are C.I.F. Indian Ports. There is at present (May, 1938) an import duty of 25 per cent. on citric acid. To arrive at the value of citric acid landed in India the figures of value in the above table have to be increased by say about 30 per cent.

12. It will be seen from Table II, that the demand of citric acid in India is increasing from year to year and is sufficient for an economic unit plant for its manufacture; preferably in connection with a plant manufacturing sulphuric acid. Before, however, one can think of manufacturing citric acid in India, there should be a constant and dependable supply of calcium citrate. This can only be done at present by utilising the cheap limes available during the pick seasons. From Table I, it will be seen that the United Kingdom is a great importer of calcium citrate. There will, therefore, be no difficulty in finding a market for calcium citrate produced in this Province until a plant for the manufacture of citric acid is put up in India.

PRODUCTION OF CITRUS OILS.

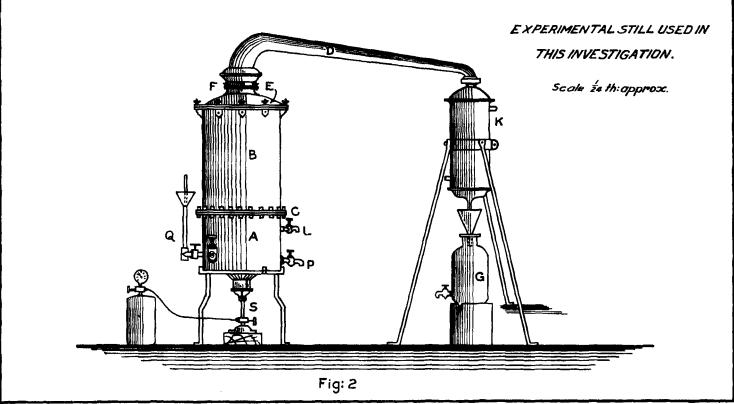
- 13. Citrus oils are produced by the following four methods :--
 - (i) Sponge method.
 - (ii) Ecuelle method.
 - (iii) Pressing and centrifugal separation method.
 - (iv) Distillation method.

The first three are known as cold extraction processes while the fourth is known as the hot extraction process.

(i) The sponge method.—The fruit is cut into two halves and the pulp from the halves is removed with a spoon. The hollow rind of the fruit is turned inside out. By this operation most of the oil cells are ruptured. The oil is then caught upon sponges held in the hand. The sponges are periodically squeezed out for removing the oil. The oil obtained by this method is of a very high quality. A large part of the oil, however, remains in the rind unextracted.

(ii) The écuelle method.—This method depends on the laceration of the oil cells in the rind of citrus fruits. The fruit are rubbed against sharp points in hollow cups known as écuelle. In the bottom of the écuelle there is a hole leading to a hollow tube. The escaping oil flows into the hollow tube from which it is periodically removed.

(iii) Crushing and centrifuging method.—The fruit is passed through crushing roles. The juice is squeezed out of the fruit and the oil cells in the rind are ruptured. A portion of the oil thus liberated goes into the juice while the rest remains in the crushed rind and pulp. The oil from the juice is separated by passing the juice through a centrifugal separator. The oil remaining in the pulp of the rinds is recovered by steam distillation.



4. P. Z.O. POONA /338.

(iv) The distillation method.—The peel is pulped and then subjected to steam distillation in a still. The escaping essential oil and water vapour are condensed and the oil is separated from the condensate.

Taking into consideration the present condition of Indian citrus plantations, their size and the type of labour available, the steam distillation method would be the most suitable of all the four methods described above for the production of lime oil for years to come. This method was, therefore, thoroughly investigated.

EXPERIMENTAL PRODUCTION OF LIME OIL.

14. A copper still made by the Ripon Copper Works, Bombay, according to our designs and capable of holding a charge of about 300 lbs. was used for carrying out the experimental work. Fig. 2 shows the diagram of the experimental still. A and B form the main body of the still with a perforated removeable false bottom at C. E is a dome-like cover which is placed on the main body after the still is filled with the material to be distilled. The cover fits in the rim which is fitted with asbestos packing all along its circumference. The cover is fixed steamtight on the rim by means of butterfly nuts. F is an opening at the top, in which fits the base of the delivery tube D. Here also asbestos packing and butterfly nuts make the connection steam-tight. K is a tubular condenser fitted to the end of the delivery tube D by a screw joint. Cold water circulates through K, during the process. It is held in position by means of an iron stand with three legs. The part A of the still is fitted with three cocks, P, Q and L. P is used to remove water from the vessel. L is used to have a rough idea about the level of water in part A (water is not allowed to rise above L). Q is another cock to which is connected a vertical copper tube with another tube in it. By this arrangement it is possible to admit water into the still while the distillation is in progress. It can also be used to find approximately the level of water in the still while it is running. The still is heated by means of a kerosene pressure burner S. The burner is connected to the oil tank by means of a hollow copper wire and the oil from the tank is forced into the burner, by pumping air into the tank. The pressure used for running the burner is about 40 lbs. per square inch.

15. The distillation is carried out as follows.—The part A of the still or the portion below the perforated bottom is filled with water. The part B of the still or the portion above the perforated false bottom is charged with the material to be distilled. The cover E and the delivery tube D are kept in position and the butterfly nuts are tightened to make the connection steam-tight. The condenser K is connected at the end of the delivery tube D. The burner is lighted and after some time the water in part A begins to boil. The steam passes through the perforated bottom and then through the material and goes along with the oil to the delivery tube and then to the condenser. At this stage cold water circulation in the condenser is started. The steam and the oil in vapour form are condensed and fall into the aspirator where they are allowed to settle. The oil being lighter than water forms a separate layer on the top of water. After settling, the water is removed by means of a syphon or through the cock near the bottom of the aspirator. This brings about a rough separation of the oil. Last traces of visible water in the oil, however, are removed by means of a separating funnel.

16. Different ways of cutting the limes and separating the juice were tried. The glass cone, which is a common device used in the ordinary household for extracting juice from oranges, limes, etc. as well as a wooden screw press specially designed for the purpose were used for pressing out juice from cut limes. Ordinary domestic hand operated mincing machines were employed for pulping the peels.

17. Several experiments regarding different ways of cutting limes, separating the juice, mincing the rinds and distilling the pulp of rinds were carried out. From these experiments it was found that the yield of oil was nearly 50 per cent. more when limes were minced without the separation of juice and the pulp thus obtained distilled directly, than when the juice was first separated from limes and the juice and the pulp distilled separately. In the subsequent experiments, therefore, limes were minced without the separation of juice. Table III shows the yield of oil by this method :--

TABLE III.

Weight of Limes pulped.	Weight of Oil recovered.	Yield of oil on the weight of Limes.
Lbs.	Gms.	Per cent.
194	201	0.229
248	283	0.252
230	227	0.218
205	223	0.240
210	200	0.210
240	233	0.215
234	227	0.214
254	24 8	0.215
227	240	0.533
250	253	0.553
260	296	0.2251
250	310	0.273
240	255	• 0•234
250	300	0.562
201	253	0.278
245	264	0.237
300	370	0.272
280	282	0.222
295	340	0.2254
	Avera	vge 0·239

Yield of Oil from the Pulp of Limes minced without separating juice.

From Table III it will be seen that the average yield of oil by this method was 0.239 per cent. on the weight of limes or 5.3 lbs. of oil per ton of limes treated.

18. The cost of treating limes by this method worked out as follows :---

Mincing 300 lbs. of limes ; 3 men for 2 hours	6 Man-hours.
Distillation takes about 4 hours. For looking after distillation and sundry work such as cleaning and recharging the still, adjusting kerosene burner, pumping air in the kerosene pressure tank, cleaning containers etc. 2 men for	, ,
4 hours	8 Man-hours.
Total man-hours for treating 300 lbs. of limes	14 Man-hours.
Man-hours per ton of 2,240 lbs	104.5 Man-hours.
Kerosene consumed for one distillation	1·31 Gals.
Kerosene consumed per ton of limes treated	10 Ġa ls.
Water used for condenser	1,075 Gals.
Labour charges at 1 anna per man-hour (at planta-	D_{-} 9.000
······································	R_{s} . 3.266
Cost of fuel, kerosene at 11 annas per gallon for 10 gallons	Rs. 6.875
Cost of water used for condenser at 2 annas per 1,000 gallons for 1,075 gallons	Rs. 0.134
	Rs. 10.275

The above figures of cost are exclusive of the cost of limes, supervision charges, depreciation on equipment, repairs and replacements.

19. Samples of this oil were sent to several firms in the United Kingdom and other countries for tests and opinion. Encouraging replies were received from several firms and some of these were even prepared to buy the oil if it was forthcoming in large quantities. The encouraging response from importers of distilled lime oil abroad showed that if the oil from cheap limes could be produced at a fairly reasonable cost, there was every possibility of its finding a good market.

20. As described before, all the previous experiments were carried out with kerosene as fuel. If the oil was to be produced on a commercial scale, it would be necessary to replace kerosene by a cheaper form of fuel, as the cost of kerosene would be prohibitive in commercial manufacture. Coal from the Central Province was, therefore, tried first. A furnace with fire bars was constructed using ordinary bricks. The still was placed on the furnace and heated on direct coal fire. Table IV shows the yield of oil, the amount of coal used, etc. during the distillations carried out with coal as fuel.

7

TABLE IV.

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240 10 2 00 2 30 4 30 54 0·99 216 290 0 275 10 2 30 2 00 4 30 56 1·54 192 353 0 801 10 2 30 3 00 5 80 52 1·54 192 353 0 277 10 2 00 2 00 4 00 60 1·54 180 388 0 281 10 2 30 2 30 5 00 60 1·54 180 360 0 280 10 2 00 2 30 5 00 60 1·52 144 330 0 280 10 2 00 2 30 4 30 56 1·43 144 378 0 296 12 2 00 2 30 4 30 55 1·43 144 374 0	260	10	3	00	2	00	5	00	55	1.43	216	327	0.27
275 10 2 30 2 00 4 30 56 1·54 192 353 0 301 10 2 30 3 00 5 80 52 1·54 180 388 0 277 10 2 00 2 00 4 00 60 1·54 180 388 0 281 10 2 30 2 30 5 90 60 1·54 180 360 0 280 10 2 00 2 30 4 30 56 1·43 144 378 0 295 12 2 00 3 00 5 60 1·10 144 400 0 296 12 2 00 2 30 4 30 55 1·43 144 374 0 276 12 2 00 2 30 4 30 53 1·54 144 366 0	230	10	2	00	2	00	4	00	51	1.32	216	281	0.56
301 10 2 30 3 00 5 30 52 1.54 180 388 0 277 10 2 00 2 00 4 00 60 1.54 180 388 0 281 10 2 30 2 30 5 00 60 1.54 180 360 0 280 10 2 30 2 30 5 00 60 1.32 144 330 0 280 10 2 00 2 30 5 00 60 1.43 144 378 0 295 12 2 00 3 00 5 00 60 1.10 144 400 0 296 12 2 00 2 30 4 30 55 1.43 144 374 0 276 12 2 00 2 30 4 30 58 1.54 144 366 0	240	10	2	00	2	30	4	3 0	54	0.88	216	290	0-26
277 10 2 00 2 00 4 00 60 1.54 180 360 0 281 10 2 30 2 30 5 00 60 1.54 180 360 0 280 10 2 00 2 30 5 00 60 1.32 144 330 0 280 10 2 00 2 30 4 30 56 1.43 144 378 0 295 12 2 00 3 00 5 00 60 1.10 144 400 0 296 12 2 00 2 30 4 30 55 1.43 144 374 0 276 12 2 00 2 30 4 30 53 1.54 144 366 0	275	10	2	30	2	00	4	30	56	1.54	192	353	0.28
281 10 2 30 2 30 5 00 60 1.32 144 330 0 281 10 2 30 2 30 5 00 60 1.32 144 330 0 280 10 2 00 2 30 4 30 56 1.43 144 378 0 295 12 2 00 3 00 5 00 60 1.10 144 400 0 296 12 2 00 2 30 4 30 55 1.43 144 374 0 276 12 2 00 2 30 4 30 53 1.54 144 366 0	301	10	2.	30	3	00	5	80	52	1•54	180	388	0.58
280 10 2 00 2 30 4 30 56 1*43 144 378 0 295 12 2 00 3 00 5 00 60 1*10 144 400 0 296 12 2 00 2 30 4 30 55 1*43 144 374 0 296 12 2 00 2 30 4 30 55 1*43 144 374 0 276 12 2 00 2 30 4 30 58 1*54 144 366 0*	277	10	2	00	2	00	4	00	60	1.54	180	360	0-28
295 12 2 00 3 00 5 00 60 1°10 144 400 0 296 12 2 00 2 30 4 30 55 1°43 144 374 0 276 12 2 00 2 30 4 30 53 1°54 144 366 0°	281	10	2	30	2	30	5	00	60	1.32	144	\$30	0-25
296 12 2 00 2 80 4 30 55 1·43 144 374 0 276 12 2 00 2 30 4 30 55 1·43 144 374 0 276 12 2 00 2 30 4 30 58 1·54 144 366 0	280	10	2	00	2	30	4	80	56	1•43	144	378	0.29
200 12 2 00 2 30 4 30 53 1·54 144 366 0 276 12 2 00 2 30 4 30 53 1·54 144 366 0	295	12	2	00	8	00	5	00	60	1.10	144	400	0.29
	296	12	2	00	2	80	4	80	55	1.43	144	374	0. 27
243 12 2 00 3 00 5 00 50 1.54 144 330 0	276	12	2	00	2	30	4	30	53	1.24	144	366	0-29
	243	12	2	00	8	00	5	00	50	1.54	144	830	0.29

Yield of oil in the experimental still with direct coal-fire as the Heating Medium.

21. It will be seen from Table IV that in the first few distillations the consumption of coal was very high. By carefully adjusting the various factors it was eventually possible to cut down the quantity of both coal as well as water for the condenser. It was found that 300 lbs. of pulp of limes could be distilled with about 60 lbs. of coal and about 150 gallons of water. The use of coal brought down the cost of fuel considerably but the time required for completing one distillation was 4 to 5 hours. Adding to this the time required for cleaning and recharging the still, each distillation took more than 5 hours. Besides it was not possible to control the temperature and consequently the rate of distillation. There was always the danger of frothing over and so a man had to be on constant attendance throughout the distillation.

22. The cost of treating limes by this method worked out as follows :---

Mincing 300 lbs. of limes; 3 men 2 hours	for 6 man hours.
For looking after distillation and sundry such as cleaning and recharging the adjusting the coal fire, cleaning contai etc., 2 men for 5 hours	still,
Total man hours for treating 300 lbs limes	
Man hours per ton of 2,240 lbs	= 119.5
Coal consumed for heating the still for	
ç	= 63.22 lbs.
Coal consumed per ton of limes treated	= 472 lbs.
Water used for the condenser	$\ldots = 1,075$ gallons.
Labour charges at half anna per man hou plantations in rural areas) for 119.5 r	
hours	=Rs. 3.736
Cost of fuel; coal at Rs. 12 per ton 472 lbs	for =Rs. 2·528
Cost of water for condenser at 2 annas 1,000 gallons for 1,075 gallons	
Total cost of treating one ton of limes	=Rs. 6·398

The above figures of cost are exclusive of the cost of limes, supervision charges, depreciation on equipment, repairs and replacements.

23. In order to reduce the cost of fuel further and to cut down the time of distillation as well as to ensure the convenient handling of the process it was thought desirable to use steam under pressure for heating. A coil made of copper and tinned from outside was fitted in the still and steam generated in a steam boiler was used as the heating medium.

24. Table V shows the amount of steam used, the time required for distillation, the pressure of steam used, the yield of oil, etc., during some of these distillations :---

TABLE V.

	Yield of oil in th	e experimenta	l still with steam	as the h	heating medium.
--	--------------------	---------------	--------------------	----------	-----------------

1	2		8		4		5	6	7	8	9	10	11
Weight of limes tren- ted.	Amount of water added to the pulp.	t st dis	me o art til- ion.	acta	or ual stil-	ti	otal me or stil- jon.	Pressure of steam in lbs. per square inch.	Amount of steam used for heating.	Amount of distil- late.	Water required to con- dense distil- late.	Amount of il recove- red.	Yield of oil on the weight of limes.
lbs.	Gall.	H.	m.	B	(. m.	H	. m.		lbs.	Gail.	'Gall.	Gms.	Per cent
800	8	0	30	0	20	0	50	201020	SO	0 -99	150	872	0 • 273
290	8	0	30	0	30	1	00	20-10-20	85	0.84	120	860	0 • 274
800	8	0	85	0	30	1	5	20 -10-20	80	0.88	144	890	0 -286
290	8	0	80	0	3 0	1	00	201010	80	0 •99	144	360	0 .274
290	8	0	30	0	30	1	00	201020	82	0·88	144	845	0 -262
300	8	0	30	0	30	1	00	20-10-20	80	0.88	144	360	0 - 264
800	12	0	35	0	30	1	5	201020	80	1 • 10	144	869	0 ·271
800	10	0	80	0	30	1	00	20- 10-20	80	1 • 10	144	375	0 • 276
3 00	8	0	3 0	0	30	1	00	20-10-20	75	0.77	144	380	0 - 279
800	10	0	25	0	3 0	0	55	20-10-20	70	1 - 21	144	369	0 • 272
800	10	0	30	0	30	1	00	201010	80	1 - 23	144	380	0 •279
800	10	0	80	0	30	1	00	20- 10-20	80	1 • 10	144	, 378	0 -278
800	10	0	30	0	30	1	00	20–10– 10	80	1.14	144	879	0 • 278
300	10	0	45	0	30	1	15	10-10-10	90	0 •99	144	\$55	0 -261
300	10	0	45	0	30	1	15	10-10-10	90	1.06	144	390	0 -286
800	10	0	80	0	30	1	00	201010	75	1 • 32	144	387	0 • 284
234	10	0	80	0	30	1	00	20-10- 10	75	0 •77	144	285	0 .272
800	10	0	30	0	30	1	00	80-10- 20	80	1.10	144	387	0 •284
800	10	0	80	0	3 0	1	00	20-10-20	70	1 • 21	144	389	0 • 286
800	10	0	85	0	30	1	5	201020	75	1 •10	144	882	0 -281
800	10	0	30	0	30	1	00	20-10-20	80	1 • 21	144	890	0 -286
300	10	0	3 0	0	30	1	00	20-10-20	70	0 -99	144	382	0 •281
800	10	0	85	0	30	1	5	20-10-20	85	1 • 27	144	405	0 • 298
300	10	0	35	0	30	1	5	20-10-20	80	1.36	144	386	0 -284

.

Average

0-278

From Table V it will be seen that the time for distillation by this method is usually one hour. Apart from the short time required for the distillation, this method ensures convenient handling of the process.

25. The cost of treating limes by this method worked out as follows :--

Mincing 300 lbs. of limes ; 3 men for 2 hours	= 6 man hours.
Distillation takes from 1 to 1.5 hours. For looking after the distillation and sundry work such as cleaning and recharging the still, regulating steam, cleaning containers,	
	= 4 man hours.
Total man hours for treating 300 lbs. of limes	= 10
Man hours per ton of limes treated	= 74.67
Coal consumed through steam (1.5 lbs. of steam=1 lb. of coal) for one distillation	= 52.5 lbs.
Coal consumed per ton of limes treated	= 392 lbs.
Water used for the condenser	= 1075 gallons.
Labour charges at $\frac{1}{2}$ anna per man hour (at plantations in rural areas) for 74.67 man-	
▲ ·	= Rs. 2·333
Cost of fuel; Coal at Rs. 12 per ton for 392 lbs	= Rs. 2.100
Cost of water used for the condenser at 2 annas per 1000 gallons for 1075 gallons	= Rs. 0·134
Total cost of treating one ton of limes	= Bs. 4.567

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The above figures of cost are exclusive of the cost of limes, supervision charges, depreciation on equipment, repairs and replacements.

Table VI shows the comparative cost data when kerosene, coal or steam is used as fuel.

TABLE VI.

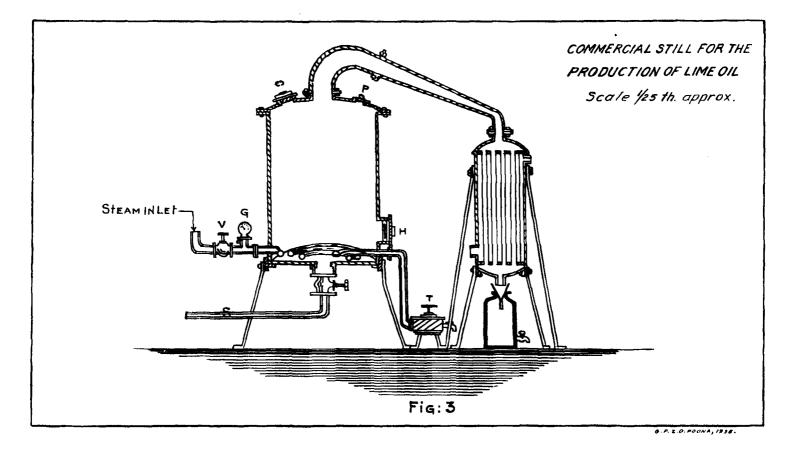
Kind of fuel used.	-	Kerosene.	Coal.	Steam.
		Rs.	Rs.	Rs.
Labour charges		3 · 27	3.74	2.33
Cost of Fuel		6.88	2.53	2.10
Cost of water used in the process	 	0.13	0.13	0.13
Total cost of treating one ton of limes	۲ 	10.28	• 6·40	4·56

• Comparative Cost Data when Kersosene, Coal and Steam are used as Fuel for the production of Lime Oil.

COMMERCIAL PRODUCTION OF LIME OIL.

The above partial data of the cost of production and encouraging 26. replies from the importers and users of lime oil, both in the United Kingdom and the Continent as well as the price offered by them and their willingness to buy the entire estimated production, clearly showed that the production of lime oil will positively be a paying proposition. The owner of a plantation at Utran, in the East-Khandesh District of the Bombay Province, was therefore induced to undertake the production of the oil on a commercial scale. A still capable of holding a charge of about 800 lbs, designed by us, was constructed by Messrs. Ripon Copper Works of Bombay under our guidance and supervision. It was then installed and put into working order on the pumping station of the plantation where a steam boiler had already been installed for the generation of power for running the pumps and oil mills. The steam generated in this boiler was used for the operation of the still.

27. Fig. 3 shows the diagram of the commercial still. It was designed more or less on the same lines as the experimental still except with certain modifications such as the size, cleaning and heating arrangements, etc. The still is designed to hold about 800 lbs. of charge at a time. It is, however, charged with about 750 lbs. of the material for usual routine distillation. As shown in the figure the steam from the boiler enters one end of the coil. The pressure of the steam is regulated by the steam valve V. The gauge G indicates the pressure of steam in the coil. The water that is condensed in the coil is discharged through the steam trap T. The still is charged through the opening O, which can be screwed



steam-tight after charging. There is one more opening P at the top which is used at times for adding water to the charge. After the distillation, the sludge is cleared through the sludge pipe S. The still is cleaned by directing a flow of water through the manhole H at the side and also through the openings O and P at the top.

28. Table VII shows the particulars regarding distillations carried out in the commercial still.

TABLE VII.

Yield of oil in the Commercial Still.

2 mount of water added to the pulp.	Ti requi to st dist		Ti	4 me		5	6	7	8	9	¹⁰ .	11
of water added to the	to st dist	ired		me								
			fc actu dist latio	red or mal il-		ta) ne ired.	Pressure of steam used Lbs. per sq. inch.	Amount of steam used.	Amount of distil- late collec- ted.	Water used for conden- ser.	Amount of oil recover- ed.	Yield of oil on the weight of limes
Gal.	H.	. 111.	н.	 ma.	н.	. m.	·	jbs.	Gal.	Gal.	Gms.	Per cent
3 8	0	55	1	15	2	10	815	180	8.2	300	1170	0 -350
39	0	50	2	00	2	50	6-15	160	2.6	300	1051	0 • 324
39	0	50	1	22	2	12	815	190	8 ∙5	300	1068	0 -329
39	1	00	1	5	2	5	· 815	180	2.1	300	1023	0 • 310
39	0	57	1	10	2	7	815	160	8.1	300	1093	0 • 326
39	1	00	1	10	2	10	8-20	180	3 ∙2	300	1056	0.315
40	1	5	1	3	2	8	8-20	180	3.2	300	1193	0 • 361
40	1	5	1	5	2	10	10-20	200	3.5	300	1123	0 .335
40	1	10	1	27	2	87	10-15	180	3.4	300	934	0 -314
40	1	36	1	33	3	9	5-15	200	2.8	312	981	0 -299
32	0	55	0	45	1	40	10-12	140	3.1	240	577	0 •300
20	0	85	11	15	1	50	5–10	140	2.4	240	600	0 •300
40	0	58	1	10	2	8	10-20	200	3.3	. 312	1193	0 •340
40	1	5	1	00	2	5	10-20	220	2.8	300	1091	0 •320
	-		-		-			}				0 • 300
	-		: -		_	-	t	1				0.305
	1 -	•	i –	• -	-	-	1					0 - 295
-	1		1		-							0 .305
40	1 -		1		_							0 ·316 0 ·330
	48 48 48 48 40 40	48 1 48 1 48 1 48 1 40 1	48 1 15 48 1 7 48 1 30 40 1 25	48 1 15 1 48 1 7 1 48 1 30 1 40 1 25 1	48 1 15 1 25 48 1 7 1 00 48 1 30 1 15 40 1 25 1 15	48 1 15 1 25 2 48 1 7 1 00 2 48 1 30 1 15 2 40 1 25 1 15 2	48 1 15 1 25 2 40 48 1 7 1 00 2 7 48 1 30 1 15 2 45 40 1 25 1 15 2 40	48 1 15 1 25 2 40 10-15 48 1 7 1 00 2 7 10-15 48 1 30 1 15 2 45 10-15 40 1 25 1 15 2 40 10-15	48 1 15 1 25 2 40 10-15 220 48 1 7 1 00 2 7 10-15 200 48 1 30 1 15 2 45 10-15 210 40 1 25 1 15 2 40 10-15 200	48 1 15 1 25 2 40 10-15 220 2 6 48 1 7 1 00 2 7 10-15 200 2 8 48 1 30 1 15 2 45 10-15 210 2 2 40 1 25 1 15 2 40 10-15 200 2 3	48 1 15 1 25 2 40 10-15 220 2 8 300 48 1 7 1 00 2 7 10-15 200 2 8 300 48 1 30 1 15 2 45 10-15 210 2 2 300 40 1 25 1 15 2 40 10-15 200 2 300	48 1 15 1 25 2 40 10-15 220 2 6 300 969 48 1 7 1 00 2 7 10-15 200 2 8 300 989 48 1 30 1 15 2 45 10-15 210 2 2 300 988 48 1 30 1 15 2 45 10-15 210 2 2 300 968 40 1 25 1 15 2 40 10-15 200 2 300 1049

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Average ... 0.319

It will be seen from Table VII that the average yield of oil from limes is 0.319 per cent. or 7.1 lbs. per ton of limes treated.

29. The cost of treating limes in the commercial still is as follows :---

Mincing 750 lbs. of limes; 6 men for 2 5 hours .. 15 man hours.

For looking after the distillation and sundry work such as cleaning and recharging the still, regulating the steam, cleaning containers, etc., 2 men for 4 hours ... 8 man hours.

Total man hours for treating 750 lbs. of limes ... 23 man hours.

Man hours per ton of limes treated	••	68 [.] 7.
Coal consumed through steam (1.5 steam $= 1$		
of coal) for one distillation	••	135 lbs.
Coal consumed per ton of limes treated	••	403 lbs.
Water used for condenser per ton of limes	••	1,000 gallons.
Labour charges at $\frac{1}{2}$ anna per man hour (at plan	ita-	
tions in rural areas) for 68.7 man hours		Rs. 2 15.
Cost of fuel, coal at Rs. 12 per ton for 403 lbs.	••	Rs. 2 16.
Cost of water used for condenser at 2 annas	per	
1,000 gallons for 1,000 gallons	• • •	Rs. 0.125.
Total cost of treating one ton of limes	• •	Rs. 4 '435.

The above figures of cost are exclusive of the cost of limes, supervision, charges, depreciation on equipment, repairs and replacements.

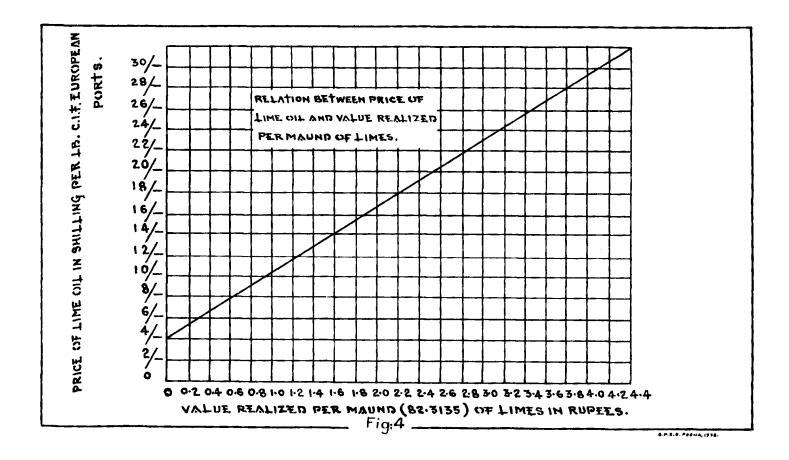
30. The oil distilled during the last three seasons in this plant has been sold at an average rate of 16 shillings per lb. C. I. F. European ports.

COST OF TREATING ONE TON OF LIMES AT UTRAN IN EAST KHANDESH DISTRICT OF THE BOMBAY PROVINCE ON THE BASIS OF TREATING 100 TONS OF LIMES PER YEAR.

31. From the observations recorded in the foregoing paragraphs regarding the working of the commercial still, it is possible to work out details regarding the actual cost of treating one ton of limes on a commercial scale at the plantation. The following data is based upon actual working of the plant and disposal of the oil in foreign markets :---

			Rs.			
Cost of picking limes at 1 anna per 100 lbs.						
Cost of hauling limes from plantation	on to the sti	ll at 🛓				
anna per 100 lbs.	••	••	0.20			
Cost of sorting at $\frac{1}{2}$ anna per 100 lbs	s	••	0.20			

Per ton.



				•	Per ton.
					Rs.
Labour charges for pulping a	and distil	ation		••	2.12
Cost of fuel	••	••		••	2.16
Boiler attendant	••	••		••	0.20
Cost of water	••	••		••	0.13
Depreciation of the still (ba of limes per year)	sed on tr	eating	100 t	ons	2.00
Repairs and replacements	••	• •		••	2 00 1 · 50
Supervision and control				••	3.00
Containers	• •	••		••	0.30
Packing cases	••	••		۰.	0.25
Drying the oil and packing	••	••		••	0.50
Railway freight to Bombay	••	••		••	0.25
Forwarding, insurance and fi	eight cha	rges to F	lurop	ean	
ports	••	••	-	••	2.50
Correspondence, postage, ca	bles, etc.	••		••	0.30
Total cost of treating 1 ton	Total cost of treating 1 ton of limes			••	17.84
			Say	••	18.00

32. The average yield of oil by distillation of limes in the commercial still is 0.319 per cent. or 7.1 lbs. per ton of limes treated. Taking into consideration the industrial and other unavoidable losses, 6.5 lbs. can be taken as an average yield of oil per ton of limes treated. The oil exported during the last three seasons was sold at an average rate of 16 shillings per pound. Taking 15 shillings per pound as the minimum, realisation C. I. F. European ports, 6.5 lbs. will fetch at least Rs. 65. Deducting from this Rs. 18 which is the total cost of treating one ton of limes, the net realisation per ton of limes treated will be (65-18=47)Rs. 47, excluding of course the cost of limes.

33. The graph in Fig. 4 shows the relation between the price of lime oil in shillings per pound C. I. F. European ports and the net realisation per maund of limes in rupees, at the plantation. From the graph it is possible to find out easily as to what will be the probable realisation that could be expected by treating one maund of limes for the production of lime oil. Deducting from this the cost at which limes can he had, it is possible to determine whether it would pay to produce the oil. For example, if the price realised in London market is say 16 shillings per pound, the net realisation will be Rs. 1 9 per maund of limes treated. If the price of limes is Rs. 1 5 per maund one would gain approximately Rs. 0 4 or about $6\frac{1}{2}$ annas per maund one would loose Rs. 0 35 or about $5\frac{1}{2}$ annas per maund.

34. Limes are either picked from the ground after they have fallen from the trees or plucked from the trees (Figs. 6 & 7). The limes so collected are brought to the still-house (Fig. 8), where they are sorted into two grades (Fig. 9), provided the market price of the better grade is sufficiently high. The limes set aside for the production of the oil are minced in handoperated mincing appliances (Figs. 10 & 11). The pulp thus obtained is put in the still alongwith about one-third of its volume of water and distilled (Figs. 12 & 13). The vapour generated during the distillation is condensed in a tubular tin-lined condenser and the condensate is collected in an aspirator bottle at the bottom of the condenser and allowed to settle. On settling two layers are formed. The upper layer is of the lime oil and the lower one is of water. The layer of water is removed through the stop cock at the bottom of the bottle. This water is added to the pulp in the subsequent charge in order to recover any oil emulsified with it. The last traces of visible water from the oil are removed by means of a separating funnel (Fig. 14). The oil is then filled in 24 oz. glass bottles which are well corked and kept in a cool dark place until five or six days before the oil is to be filled in tins. When the stock reaches 300 to 400 lbs. a little anhydrous calcium chloride is added to every bottle which is then corked air-tight. The bottles are shaken upside down 5-6 times a day for two days and then the contents are allowed to settle for two days more. Calcium chloride absorbs most of the water in the oil. The bottles are then opened and the oil poured through a strainer into a large tin-lined brass container. The container has an air-tight lid, and a cock about one inch above the bottom. Both the lid and the cock are so fitted that they can be locked and sealed if necessary (Fig. 15). The oil is allowed to settle for a day or so in the container in order to allow any fine particles of calcium chloride that might have gone into the container while the oil was poured from the bottles, to settle down. Specially made tin cans (14 in. \times 6 in. \times 6 in.), thoroughly washed and dried, are then filled with the oil and immediately closed and soldered to render them air-tight. Each can is filled with 14 lbs. of the oil. The tins are then packed in wooden cases, each containing two tins or 28 lbs. of oil in the case. The cases are marked according to the instructions of the buyers in the United Kingdom or the Continent (Fig. 16), and then handed over to the forwarding agents in Bombay for shipment abroad.

35. It has been stated in para. 17 that the yield of oil was less when the juice was first separated from limes and the rinds and juice were distilled separately, than when limes were minced without the separation of juice. A good deal of oil is lost due to evaporation during the process of cutting the limes and extracting the juice. In case the juice has to be used for concentration or preservation purposes it is necessary to separate it from limes before mincing. In order to establish a definite relation between the yields of oil when limes are minced without cutting and also when juice is first separated from limes before mincing, a series



Fig. 5. A General View of the Plantation.



Fig. 6. Picking of fallen Limes

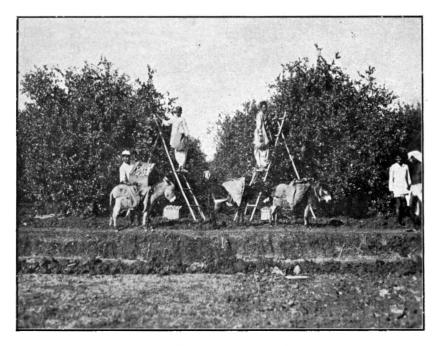


Fig. 7. Plucking of Limes from Trees.

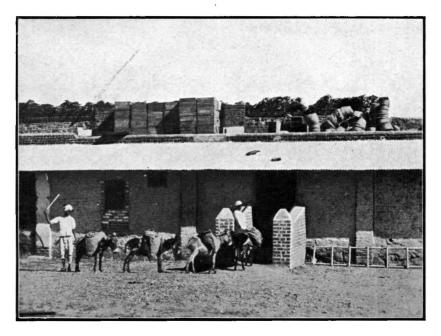


Fig. 8. Hauling of Limes from the Plantation to the Still House.

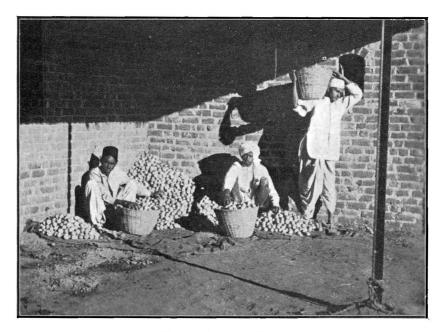


Fig. 9 Sorting of Limes

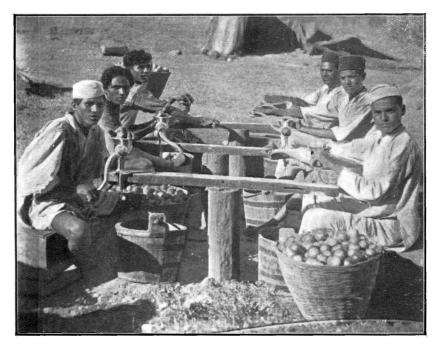


Fig. 10. Mincing of Limes in Progress.

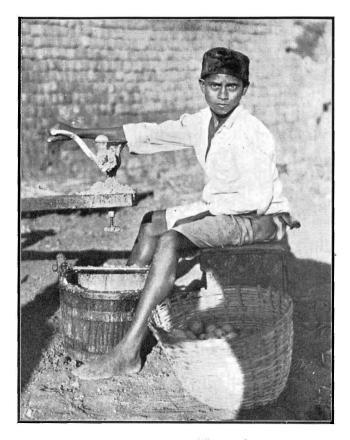


Fig. 11. Close View of the Mincing Operation.

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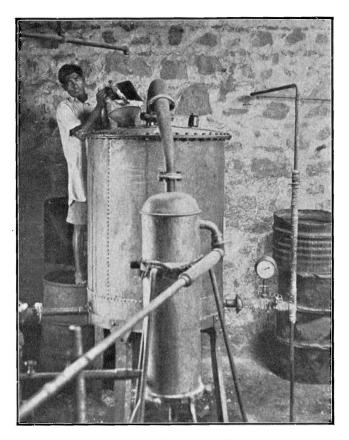


Fig. 12. Charging the Still.

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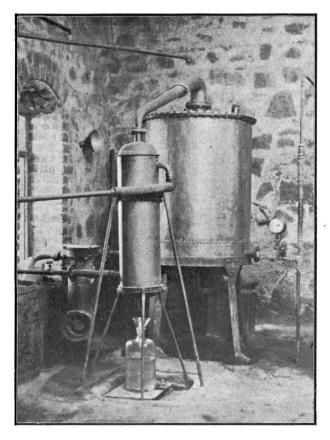


Fig. 13. Still in Operation.

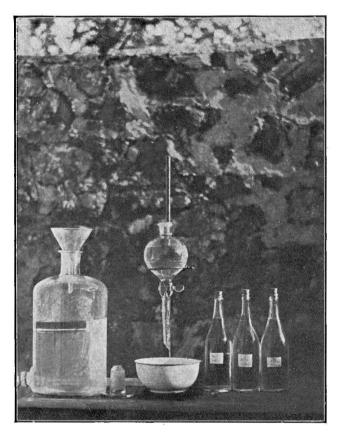


Fig. 14. Separation of Oil from Water.

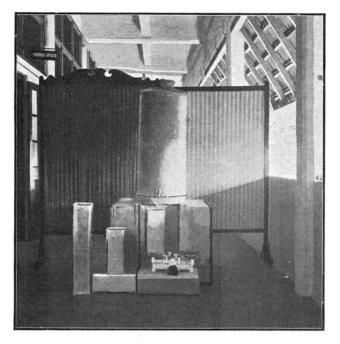


Fig. 15. Filling Tin Cans with Oil from the Oil Container.

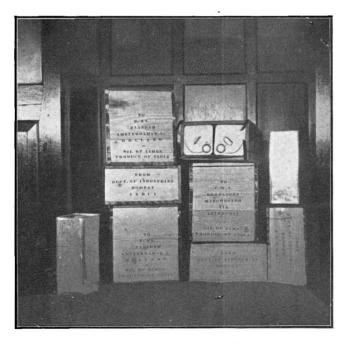


Fig. 16. Boxes packed and ready for export.



Fig. 17. Preparation of Calcium Citrate.

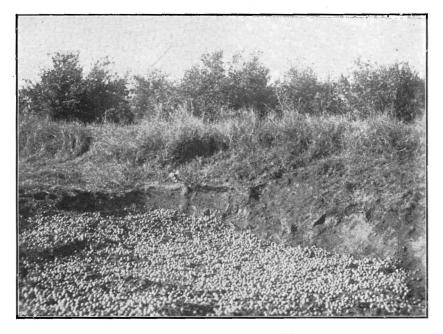


Fig. 18. Limes thrown away as Waste.

of experiments were carried out in the commercial still. Table VIII shows the yield of oil etc. when juice is first separated from limes and the rinds and juice are distilled separately for oil.

TABLE VIII.

Yield of Oil from the Juice and Rinds, distilled separately.

Weight of limes treated.	Amount of oil recovered from juice.	Amount of oil recovered from rinds.	Total amount of oil recovered.	Oil reco- vered from juice.	Oil resovered from rinds.	Total oli from lines,
lbs.	Gms.	Gms,	Gms.	Per cent.	Per cent.	Per cent.
615	74	458	532	0 -0265	0 .1642	0 •1906
757	68	676	744	0 •0198	0 •1968	0 • 2167
590	62	428	490	0 -0232	0 •1599	0 •1831
635	73	552	625	0 -0253	0 •1916	0-2169
1,063	96	909	1,005	0 -0199	0 •1886	0 • 2084
			Average	0.9229	0 .1802	0 -2081

It will be seen from Table VIII that the yield of oil is 0.2031 per cent. i.e., 4.5 lbs. per ton of limes treated, as against 6.5 to 7 lbs. per ton when juice is not recovered. This means a loss of about 2 lbs. of oil.

36. The cost of treating limes by the above method is very high as will be seen from the following cost data :--

Cutting 700 lbs. of limes into 2 halves : 4 men for
2 hours 8 man-hours.
Squeezing out juice from the halves on glass
cones: 12 men for 8 hours 96 man-hours.
Mincing the rinds : 3 men for 4 hours 12 man-hours.
Distillation of the pulp of rinds takes about 2 to
2.5 hours and the juice distillation takes about
Ith of an hour. For looking after distillations
and sundry work such as cleaning and recharg-
ing the still, regulating steam, cleaning
containers etc. 2 men for 4 hours
Total man hours for treating 700 lbs. of limes 124
Man hours per ton of 2,240 lbs
Coal consumed (through steam) for heating the
still
Coal consumed per ton of limes treated 512 lbs.
Water used for condenser 1,500 gallons.
Labour charges at ½ anna per man hour (at
plantations in rural areas) for 396 8 man hours. Rs. 12.40
Cost of fuel; coal at Rs. 12 per ton for 512 lbs Rs. 2.74
Cost of water at 2 annas per 1,000 gallons for
1,500 gallons Rs. 0.19
Total cost of treating one ton of limes Rs. 15.33 MO-II Bk Qa 15-2

It will be seen from the above data that the cost of treating limes by this method is about $(15 \cdot 33 - 4 \cdot 43 = 10 \cdot 9)$ Rs. 10 $\cdot 9$ more than when limes are minced without the separation of juice. Besides, the yield of oil is less by about 2 lbs., which means an additional loss of at least Rs. 20; that is to say the net realisation will be less by about $(20 + 10 \cdot 9 = 30 \cdot 9)$ Rs. 31.

37. One ton of limes yield about 1,000 lbs. of juice. This has to be concentrated to about $\frac{1}{8}$ th of its volume so that the concentrated product would contain about 50 per cent. of Citric acid. 12 to 13 gallons of concentrated juice will, therefore, be obtained from one ton of limes treated. This would be sold for about Rs. 14 at the present market rates. The cost of concentrating the juice, which would be about Rs. 3 must also be considered. If limes are to be treated profitably by this method the price of concentrated juice should be (31 + 3=34) Rs. 34 per 12 gallons or say Rs. 283 3 per 100 gallons of concentrated juice containing 50 per cent. citric acid.

38. All the reports on the oil produced in this investigation from purchasers of oil in the United Kingdom and European countries were almost unanimous in that the oil was about 10-20 per cent. inferior in quality to that imported by them from the West Indies.

39. In order to compare the oil produced from the Indian limes with that produced in the West Indies, one pound of lime oil produced in the West Indies was imported and its physical and chemical constants determined.

Table IX shows the physical and chemical constants of an average sample of lime oil from Indian limes produced in two different seasons in the year 1936 and those of the West Indian Oil.

TABLE IX.

Constants of two Indian and one West Indian Samples of Lime Oil.

		Indian Oil, 1936 July.	Indian Oil, 1936 December.	West Indian Oil.
Specific Gravity Optical Rotation		0.8533 at 30°C +45.22	0·8537 at 30°C +44·80	0.8563 at 30°C +41.16
Refractive Index	••	1.4710 at 30°C	1.4720 at 30°C	1•4728 at 30°C
Citral Content		0.94%	1.02%	1.73%
Acid Value		1.1	0:99	0.71
Ester Number		2•4	2.12	1.87
Non Volatile Residue	••	2.21%	2.35%	3.14%

From Table IX it may be said that the oil distilled in the second season (December) is better than that distilled in the first. Further investigation is, however, necessary to find out a relation between the quality of the oil and climatic conditions. The quality and quantity of manures used may also have an influence on the quality of the oil.

40. The West Indian sample had a somewhat better smell than the oil distilled here. It may perhaps be attributed to the difference in variety of the fruit, but more likely it may perhaps be due to the addition of a small quantity of hand-pressed oil to the distilled oil before sending it to the market.

PRODUCTION OF CALCIUM CITRATE.

41. Calcium citrate is a very important raw material for the manufacture of citric acid and as mentioned in paragraph 10, is one of the important citrus products in the international trade. A general method for the production of calcium citrate from lime juice is as follows :---

The juice is pressed out of the fruit by any of the usual methods and is then strained through felt bags so that it is practically clear. Any pulp left in the juice makes the subsequent washing and filtration of calcium citrate difficult and affects adversely the quality of the same. A marketable grade of calcium citrate should have at least about 62 per cent. citric acid content. If an appreciable quantity of pulp goes into calcium citrate it will be difficult to get a product having more than 60 per cent. citric acid content. The strained juice is boiled and then neutralised by gradual addition of a thin strained suspension of very high grade lime with constant stirring. The suspension of lime should be thin and free from big particles and lumps. Any lumps present in the suspension will not be reacted upon by the weak acid and will go down as such in the precipitate of calcium citrate. This will lower the percentage of citric acid and cause a loss of sulphuric acid in the process of manufacture of citric acid from the citrate. When the mixture reaches the neutralisation point—as shown by the litmus paper-the addition of lime is stopped. If the mixture shows any alkaline reaction, a little more juice is added so that ultimately the solution remains slightly acidic. The precipitate of calcium citrate is allowed to settle and the solution decanted while still hot. The precipitate is washed two or three times with hot water and finally dried either in the sun or in hot air ovens, after the removal of as much water as possible by filtration.

42. The commercial calcium citrate generally contains about 64 per cent. citric acid, the adjustment of higher or lower prices is based upon this figure. The citrate should not test less than 60 per cent. citric acid and should be free from magnesium citrate. The other requirements generally are that calcium carbonate content of the citrate should not be more than about 0.15 per cent. and iron and aluminium should not exceed about 0.1 per cent.

43. In this investigation calcium citrate was prepared by the above process from raw juice on a small scale in the laboratory and then on the

plantation on a large scale. Tables X and XI show the yield of calcium citrate on a laboratory scale and on a large scale respectively.

TABLE X.

Yield of Calcium Citrate from Juice of Limes on Laboratory Scale.

Gms.	Gms.	lbs.	
		lbs.	
100.0	135	90.2	
241.5	330	94.4	
301.0	430	95.8	
420.0	593	.94 7	
	301.0	301 ·0 430	

TABLE XI.

Juice treated.	Acidity of juice.	Acid present in terms of citric acid.	Weight of calcium citrate recovered.	Weight of lime used for neutra- lisation.	Limc used per ton of limes treated.	Yield of calcium citrate per ton of limes treated.
Gallons.	Per cent.	lbs.	lbs.	lbs.	lbs.	lbs.
7 -92	6 .95	5.52	6 -86	2.2	27.5	85 • 7
11 - 88	6 -95	8.27	9.60	3-4	28-3	80.0
11 -00	7 •00	7 • 72	9 ·20	3.2	28-5	82.1
11 .00	7 •00	7 • 72	00 [,] C	3 •1	27 •7	80 - 3
					Average	82.03

Yield of Calcium Citrate from Juice of Limes on Large Scale.

44. Preparation of calcium citrate from good clear juice does not involve any difficulty and claborate control in the operation. It is, therefore, comparatively easy to prepare calcium citrate from juice separated from limes before distillation. When limes are treated solely for the production of oil, that is, when they are minced without the separation of juice, and the pulp thus obtained is directly distilled, the residue left in the still is in the form of a thick viscous mass or sludge. It is colloidal in nature and cannot be easily filtered.

45. The sludge when stored in wooden barrels and allowed to settle for about 48 hours divides itself into two parts. The heavier and more viscous portion remains at the bottom leaving a cloudy watery liquid above it, which could be siphoned out or decanted. Out of the total acid present in the sludge about 45 to 50 per cent. is found to be present in this upper liquid. In order to get more acid in the decanted solution it was thought desirable to add some water to the sludge before allowing it to settle. The addition of water resulted in an increase in the amount of acid present in the decanted solution. With the addition of about 4th its volume of water, it was found that about 70 per cent. of the total acid present in the sludge can be brought into the decanted solution. The addition of larger quantities of water increases the yield of acid in the form of decanted solutions to some extent, but the resulting solution is too dilute for efficient and economic precipitation of calcium citrate. The addition of water to the sludge should, therefore, be restricted to 4th of its volume or even less. This will give the final solution of 2.5 to 3 per cent. citric acid content.

Table XII shows the per cent. recovery of acid from the sludge in the form of decanted solutions after the addition of water.

TABLE XII.

Amount of Acid present in the Solution after Decantation from the Sludge.

Sludge treated.	Water added.	Water added per 100 gallons of sludge.	Acid present in the sludge.	Acid present in the Decanted solution.	Acid present in the Decanted solution.
Gallons.	Gallons.	Gallons.	lbs.	lbs.	Per cent.
85 70 65 50 90	20 15 16 20 50	$ \begin{array}{r} 23 \cdot 5 \\ 21 \cdot 4 \\ 24 \cdot 6 \\ 40 \cdot 0 \\ 55 \cdot 5 \end{array} $	$21 \cdot 25 \\ 16 \cdot 10 \\ 14 \cdot 50 \\ 12 \cdot 12 \\ 22 \cdot 99$	14.62 11.23 10.26 8.57 16.60	68 82 69 76 70 76 71 12 72 21

The solutions were generally allowed to settle for about 48 hours. It was found that when allowed to settle for more than 48 hours there was no appreciable increase in the acid content of the decanted solutions excepting of course a little clarification of the same.

46. The solutions referred to above though clear to some extent were still colloidal in nature and one of the difficulties in precipitating calcium citrate from them was that the impurities precipitated along with calcium citrate. Further experiments on the precipitation of calcium citrate from dilute solutions obtained from the sludge showed that the impurities settled down as soon as the solution attained the neutralisation point. In the subsequent experiments it was found that the precipitation of impurities can be avoided if the addition of lime was stopped just before the neutralisation point was reached. In order, therefore, to prepare calcium citrate of marketable quality from the sludge obtained after the distillation of minced limes without the separation of juice, the following procedure was adopted.

The sludge was transferred to wooden barrels of about 150 gallons capacity and allowed to settle after the addition of about $\frac{1}{4}$ th its volume of water. After settling for about 48 hours the upper solution was siphoned off in a wooden tub of about 100 gallons capacity. This tub was used for the precipitation of calcium citrate (Fig. 17). The tub

was filled with 50 to 60 gallons of the solution of citric acid decanted from the sludge. It was heated by means of open steam. When the solution began to boil the thin lime suspension-previously prepared and strained through cloth-was gradually added to it. The force of steam kept the solution agitated to such an extent that it was not necessary to make any additional arrangement for stirring. The addition of lime was continued slowly and after every few minutes a portion of the reacting solution was withdrawn, cooled and 5 c. c. from it titrated against standard sodium hydroxide solution. When the acidity came down to about 0.1 per cent. the addition of lime was stopped and the solution allowed to settle for about 15 minutes. The supernatent liquid containing the suspended impurities was siphoned out and the precipitate of calcium citrate washed three or four times with hot water. The water from the washed precipitate was removed by transferring it to a filter made out of an ordinary cloth. The thick mass of moist calcium citrate thus obtained was dried either in the sun or in a hot air oven.

In precipitating calcium citrate from the solution obtained from the sludge, it is necessary to see that at no stage during the addition of the suspension of quick lime to the solution, the reacting mixture becomes neutral. In order to get a marketable quality of calcium citrate it is necessary that the addition of lime is stopped when the reacting mixture has 0.1 per cent. acid in it. If this point is overlooked the suspended pulp and other impurities in the solution, obtained from the sludge, coagulate and settle down with the precipitate of calcium citrate. The visible indication of stopping the addition of lime is that the solution at the point of contact with the suspension of lime becomes darkish brown in colour and the precipitate becomes greenish yellow, when the solution attains the acidity of less than 0.1 per cent. Table XIII shows the yield of calcium citrate from limes treated by this method.

TABLE XIII.

Lir	ght Of nes nced.	Solution obtained.	Acidity of the solution.	Acid present.	Acid remaining unneutra- lised.	Acid neutra- ìised.	Lime used.	Weight of Calcium Citrate recovered.	Yield of Calcium Citrate per ton of limes treated.
1	bs.	Gallons.	Per cent.	ibs.	lbs.	lbs.	lbs.	lbs.	lbs.
412		44	2.3	10 .12	1.02	9 ·10	4.7	11.5	62 ·52
718	••	56	3 • 1	17 .64	0 -92	16.72	8.0	21 ·0	65 ·48
777		56	3 • 4	19 .10	1.60	17.50	· 9·5	23 ·0	66 ·24
370	••	26	3.5	9 ·10	0.32	8.78	5.2	11 .3	68 ·31
790	• •	57	3.4	19 - 40	1.40	18-00	9-5	23 -0	65 ·21
671	+-	50	3 • 3	16.50	1 .20	15 30	7.5	20.0	60 •67
706	••	56	3.1	17 .36	1.98	15 ·38	11-0	20 - 5	64 •97
718	••	56	3.1	17 -64	0-90	16.74	10.2	21 .0	65 •48
								Average	65.61

Yield of Calcium Citrate from Sludge per ton of Limes treated.

From Table X1111 it will be seen that the yield of calcium citrate per ton of limes treated by this method is about 65 lbs. as against 93 lbs. shown in Table X when juice is separated from limes before distillation. The use of a filter press for filtering the sludge is sure to increase the yield of calcium citrate by this method. It will also facilitate the handling of the process.

Calcium citrate prepared by this method gave the following analysis :---

Citric acid	$= 62 \cdot 2$ per cent.
$\operatorname{Fe}_2 O_3 + \operatorname{Al}_2 O_3$	= 0.14 per cent.
CaČO ₃	= 0.11 per cent.
Moisture	= 0.08 per cent.

47. The cost of production and disposal of calcium citrate on a commercial scale (without the use of a filter press), on the basis of 700 lbs. of limes treated, works out as follows :--

Carrying sludge from still to settling barrels,	
4 men for 1 hour $\dots \dots \dots$	4 man hours.
Siphoning the solution, preparing lime paste, etc.,	
	2 man hours.
Decanting and washing the citrate, cleaning con-	
tainers, etc., 2 men for 1 hour $\dots =$	2 man hours.
Total man hours for treating 700 lbs. of limes $\ldots =$	8
Man hours per ton of limes treated $\dots =$	25.6
Coal consumed through steam $(1.5 \text{ lbs. of steam} =$	
1 lb. of coal)	60 lbs.
Coal consumed per ton of limes treated $\dots =$	192 lbs.
Lime (CaO) consumed for treating sludge from	
700 lbs =	
Lime (CaO) consumed per ton of limes treated $\ldots =$	28 lbs.
Labour charges at $\frac{1}{2}$ anna per man hour (at plantations in rural areas) for 25.6 man hours =	D. 0.9
Cost of fuel; coal at Rs. 12 per ton, for 192 lbs. =	
Cost of very high grade lime (CaO) at Rs. 3 per	RS. 1 U
	Da 0.75
cwt. for 28 lbs	NS. U 10
citrate	$\mathbf{p}_{\mathbf{a}}$ $\mathbf{h} \cdot \mathbf{g}$
Forwarding, insurance and freight charges to	1.8. 0 0
European ports	D. 1.95
	108.1 20
	Rs. 4·4
Total cost of treating one ton of limes for calcium	
citrate	Rs. 4.4

say .. Rs. 4.5

At an average market rate of about £ 23 per ton, 65 lbs. of calcium citrate will fetch about Rs. 8.8. Deducting from this Rs. 4.5 as the cost of production, the net realisation from calcium citrate would be Rs. 4.3 per ton of limes treated. The expected realisation from lime oil



if it fetches 15 shillings per lb. in the European market, is Rs. 47 per ton of limes treated. By adding to this sum Rs. 4'3 realised from calcium citrate, the total net realisation per ton of limes will amount to Rs. 51'3 or about Rs. 1'88 or about Rs. 1-14-0 per maund of limes treated. This figure will hold good provided at least about 100 tons of limes are treated per season at one place.

48. The figures of cost of treating the limes, etc., given in the foregoing paragraphs are for Utran in the East Khandesh District and so they are likely to vary from place to place depending upon the size of the still, the rates of wages for agricultural labourers in the locality and whether the still is heated by direct fire or with steam, etc. The cost of a still with a capacity to take charge of about 800 lbs. of minced limes capable of being heated with steam will come to about Rs. 800 complete, at the present (May 1938) market rates for copper sheets, etc. A second-hand boiler capable of running the still could be had for about Rs. 600. Steam connections, water tanks, water pumps, etc., would cost about Rs. 300. Miscellaneous expenses for mincing appliances, drums, casks, etc., would come to about Rs. 200 and Rs. 500 for the equipment and appliances for the preparation of calcium citrate. Adding Rs. 100 as the charges for erection etc. a sum of Rs. 2,500 will be required for putting up a plant for treating about 1 to 2 tons of limes per day provided a suitable building can be rented for installing the entire equipment. Working capital required will depend upon the quantity of limes handled during the season. Roughly speaking a sum of 3 to 4 thousand rupees ought to be sufficient for the purpose. This makes a total investment of about 6 to 7 thousand rupees for putting up a plant for the distillation of lime oil as well as for the production of calcium citrate. This is a very rough figure and will have to be modified according to the conditions prevailing in a particular locality.

For a very small unit, one can manage to produce the oil with a capital of about one to two thousand rupees provided the oil is sold to a party in this country and the value of oil is realised within 10 to 15 days after the delivery of the same. The value realised for limes in this case will obviously be very much lower than that realised by large scale operation Before, however, one finally decides to put up a small or big plant the distillation of the oil it is desirable for him to consult the Departn of Industries, Bombay, and find out whether his plan is likely to a success at a particular locality.

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 $\chi_{\mathcal{O}_1}(f^{(2)}; J^{(37211)}), 2^{(1)})$