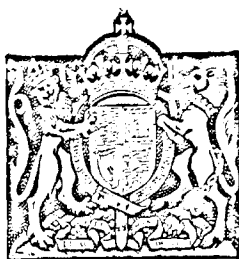


INVESTIGATION OF THE OIL OF
PUNNAL (CALOPHYLLUM INOPHYLLUM)

By

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Government of Bengal

Industries Department

Bulletin No. 43

Investigation of the
Oil of Punnal
(*Calophyllum Inophyllum*)

with reference to its Application in Soap-Making
specially of the Bengal type of Moulded Soap

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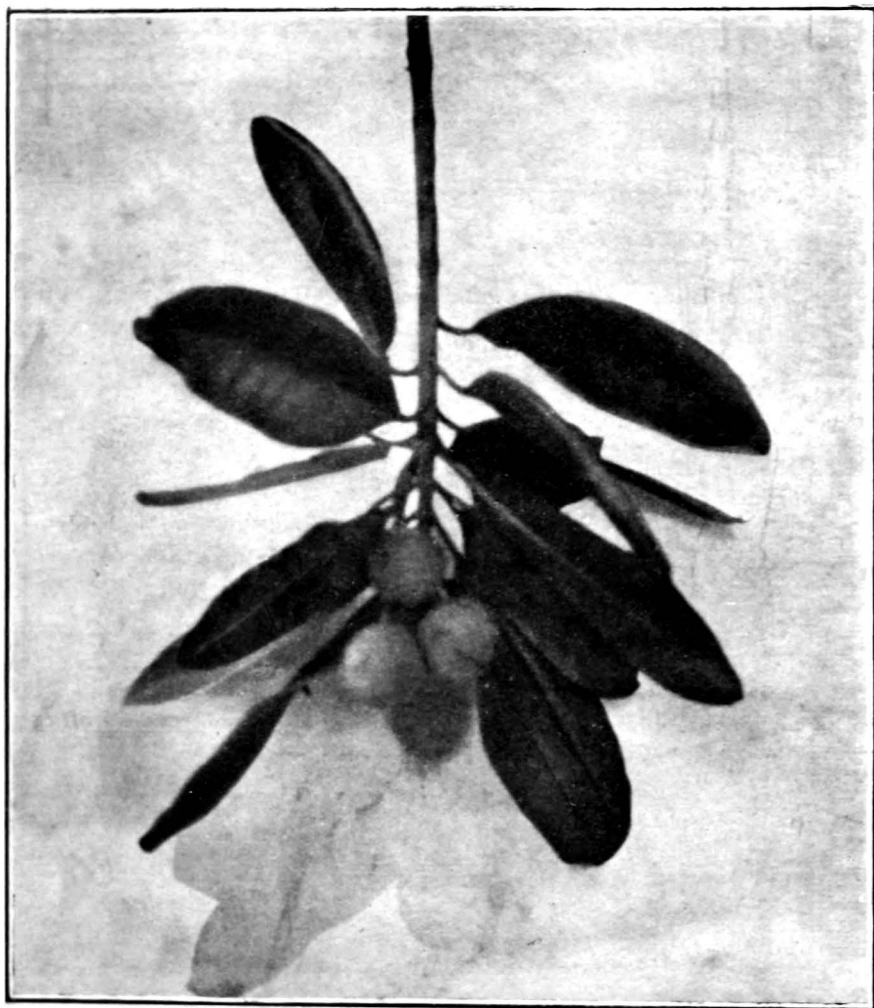


Illustration of the Punal leaves and fruits.

Investigation of the Oil of Punnal (*Calophyllum Inophyllum*) with reference to its application in Soap-Making, specially of the Bengal type of Moulded Soap.

Reason and object of investigation.—The Punnal tree (*Calophyllum Inophyllum*) has been known in Bengal and the oil expressed from its nut for use as an illuminant for a long time. The oil has of late been ousted from this field by kerosine. It was therefore felt that unless some other profitable use for the oil were found out and made known to the general public this prolific oil-producing tree would gradually disappear from the lands of Bengal, as it would then be considered good for nothing else than fuel. Investigation was therefore undertaken with a view to carrying out systematic researches to find out the properties and applications of the oil as a raw material for the soap industry, and the results obtained on experiments carried out with the oil in the Industrial Research Laboratory of this Department are herein published for general information. The oil is at present being used to a small extent in soap-making in Calcutta and elsewhere, the supply in the Calcutta market coming chiefly from the Puri district in Orissa and also from the Sandwip and Hatia islands in the Noakhali district, Bengal. The proper use of the oil as a soap stock is, however, little understood and hence it is being used only in the preparation of the cheap classes of soaps of indifferent qualities. It is expected that with the spread of the information contained in these pages regarding the exact nature and saponaceous properties of the oil on which no investigation appears to have been done before and published, the oil will come to be used in much larger quantities than at present and thereby its increased production will be encouraged. Most of the published data on this oil-bearing nut are old and antiquated, and without any substantial experimental facts to support them. Very frequently the observations of one writer have been reproduced by another, and where such is not the case the data are at wide variance with one another. For example, the actual yield of oil from the nut is very conflicting, some writers say that it is 60 per cent., while others hold that a given weight of the nuts gives one-third its own weight of oil. Data like these emphasize all the more the need for a systematic investigation on the subject.

2. The Punnal tree.—The tree is an evergreen one and is widely distributed along the coast of India. It is indigenous to the Western Peninsula, South India, Ceylon, Orissa, South-east Bengal, Burma and the Andaman Islands. It can be grown almost anywhere in the Indian Peninsula but thrives best near the sea coast. Dry sandy leeches where scarcely anything else will grow provide a suitable habitat for this tree. In such conditions it develops a large size with a heavy yield in fruit. In Bengal the tree is found quite abundantly in the Sandwip and Hatia islands in the Noakhali district. Judging from the coastal area and character of the soil of these two islands it is estimated that they are capable of producing large quantities of the oil, but are at present producing relatively small quantities almost all of which is sent out from the district to soap-making centres in Bengal. From the average yield of fruit from a mature tree reported from Sandwip an annual revenue of Ra. 20 per tree should by no means be uncommon. If, therefore, this tree can be established on

a plantation basis on waste land near the sea, or near, but not inundated with, saline waters, a high net return can be expected which may in all probability be more than that for many agricultural crops. The cultivation of this tree in the delta portion of Bengal and for the matter of that in other parts of India not far inland should also prove a very profitable proposition. It is, therefore, strongly recommended that enterprising men should take up such plantations and thereby increase the available supply of soap stock as also the producible wealth of the province inasmuch as considerable areas of land are now fallow or unprofitably utilised.

The advantages of winning oil from this nut are that the seed has a substantial size and weight and that its oil-content is exceptionally high, being 72 per cent. of the decorticated and dried seed. The oil fetches a good value which, under the present market conditions, ranges from Rs. 16 to Rs. 18 per maund.

3. **Seed germination and plantation.**—In Bengal the seed germinates in the rains. The unshelled seed is sown on loose soil but is not covered up by the same. Transplantation is not known to invigorate the young plant, and it is better not to have recourse to such a measure, but to sow the seed on the spot where the tree is to finally stand. In the early stages the cattle eat the plant and hence it should be protected by fencing. The tree comes to fruit in about ten years' time, which, however, is somewhat shortened in the case of trees in close proximity to the sea.

4. **Fruiting.**—The tree flowers in June and July and the nuts, round in shape, mature by the end of December or the beginning of January, the green nuts becoming yellowish on ripening. Frequently the outer shrunken skin of the ripe nut is eaten up by bats, so that when the nuts fall to the ground many of them have a clean surface, the hard, brittle shell enclosing the pale yellow kernel with a layer of brown brittle husk in between the two. The collection of the mature nuts offers no difficulty as they are simply picked up from the ground at the foot of the tree. The kernel is obtained by breaking off the hard shell, and the oil is expressed from it in the manner hereinafter described.

5. **The Punnal nut.**—The size of the nuts obtained from Sandwip varies from 1.4 c.m. to 3.1 c.m. in diameter, and the following table gives the weights of mature nuts of different sizes and the proportion of the kernel to the whole nut. In the table the weights are given in grammes, 28½ grammes being approximately equivalent to one ounce avoirdupois.

Serial No. of sample.	Weight of whole nut. (Grammes.)	Weight of kernel. (Grammes.)	Weight of shell and husk. (Grammes.)	Percentage of kernel on whole nut.
1	8.98	5.45	3.53	60.7
2	7.77	4.70	3.07	60.5
3	6.38	3.85	2.55	60.3
4	5.73	3.49	2.24	60.9
5	5.18	3.17	2.01	61.2
6	4.75	3.08	1.67	64.9
7	3.59	2.24	1.35	62.4
8	2.64	1.58	1.06	60.0
9	2.39	1.40	0.99	58.6
10	2.22	1.37	0.85	61.7
11	1.75	1.02	0.73	58.3
12	1.48	0.81	0.67	54.7

6. **The Bengal nut and the Travancore nut.**—It has already been mentioned that the tree is found in some other parts of India. For purposes of comparison, therefore, a consignment of the nuts was obtained from Travancore and examined in the Industrial Research Laboratory. The maximum diameter of a whole nut of the Travancore sample was 3.45 c.m. and the minimum 1.35 c.m., as against 3.1 c.m. and 1.4 c.m. respectively of the Sandwip variety. The total weight of the kernel of 336 Travancore nuts was 1,000.5 grammes and that of the same for an equal number of Sandwip nuts was 800.5 grammes. The average weight of the kernel of a Travancore nut was therefore 2.98 grammes, that for a Sandwip nut being 2.33 grammes. The shapes of the nut and the kernel of the Travancore variety are slightly ellipsoidal while those of the Sandwip variety are almost spherical. It will therefore be apparent that the nut from Travancore is slightly bigger than the Bengal nut.

7. **Decortication.**—At Sandwip the shelling of the nuts is usually done by placing them on the hard floor and pressing them with a wooden board which is kept sliding over them. Four to six nuts are thus shelled by each movement of the board. This method gives a slightly larger output than that obtained by breaking the nuts by means of a mallet or hammer. It has been found that by breaking the nuts one by one about 2 lbs. of kernel are obtained per hour. At Sandwip and Hatia the children and female members of the villagers do the shelling at their leisure time, and hence no account is taken of the cost of shelling. When, however, large scale plantations are organised, and all work including collection and shelling is required to be done by hired labour the question of shelling the nuts quickly and economically will assume considerable importance. A mechanical principle by the employment of which this can be done by machine has been practically tested with very good results. Practical application of this principle can only be possible when large quantities of the nuts are required to be decorticated at one and the same time such as what happens in large scale plantations. The design of a shelling machine embodying the above principle can be undertaken if the nut comes to be grown on such a scale.

8. **Drying.**—The kernel examined in the laboratory immediately after shelling the nut has been found to contain 27 per cent. of moisture. It has to be dried before the oil can be expressed from it. The drying in the sun does not take more than eight days, while in the shade it takes place slowly requiring a longer time for completion. Further, in drying in the shade the kernel must be spread in a thin layer if decomposition and fungus growth are to be avoided. Attempts to dry the kernel in steam-heated chambers or vessels immediately after shelling resulted in the exudation from the same of a very sticky resinous substance. The steam-drying is, however, quite satisfactory when it is preceded by partial drying of the kernel either in the sun or in the shade as aforesaid.

9. **Oil-content.**—After the removal of moisture by drying, the oil-content was determined in the laboratory by the solvent method, and it was found to be 72 per cent. of the dried kernel, which is about the maximum that can be found in any oil-bearing seed or fruit. The

proportion of oil in the undried kernel is thus 56.7 per cent. and in the whole nut 38.7 per cent., the percentage of kernel on the whole nut being, on an average, 59.

10. **Expression of oil—existing method.**—In Bengal the existing practice is to express the oil from the dried kernel by the country ghani but the results commonly obtained by the villagers are not satisfactory, the cake generally retaining fairly high percentages of oil. A sample of oil-cake from Sandwip gave, on analysis, 19 per cent. of oil, so that with the country ghani the actual yield of oil is only 53 per cent. out of the total of 72 per cent. contained in the dried kernel. The above result of the analysis may be taken as typical of what happens in practice in villages where the oil is expressed in countryghanis by village workers.

11. **Expression of oil—ideal method.**—The ideal method for obtaining the maximum yield of oil is to treat the kernel in the country ghani in the first instance and then to treat the resulting cake in the Expeller Press. It may incidentally be mentioned that on account of the spongy nature of the kernel very thorough expression cannot be effected in the Hydraulic Press.

12. **Manurial constituents in Punnal cake.**—The Punnal cake, like other oil-cakes, contains manurial constituents and a sample of cake from the country ghani containing 19 per cent. oil gave, on analysis, the following results which show that the cake has high manurial properties equalling those of the mustard cake:—

	Per cent.
Nitrogen	3.07
Phosphorus (as P_2O_5)	1.71
Potassium (as K_2O)	1.49

As for the other use of the cake, viz., as a food for cattle, it may be mentioned that the cake is poisonous and cannot be used for such a purpose.

13. **Physical and chemical constants of the oil.**—The oil as expressed has a light yellow colour with a greenish tint which deepens on standing. The oil has a tendency to deposit particles of solid fat. It has a characteristic odour, not very disagreeable, by which it can be identified unmistakably. On cooling, the oil begins to thicken at $5^{\circ}C$ and becomes a viscous solid at about $0^{\circ}C$. The freshly expressed oil gave on analysis the following constants:—

Specific gravity at $36.5^{\circ}C$	0.932
Acid value	36.3
Saponification value	194.0
Iodine value	89.9
Refractive Index at $31^{\circ}C$	73

14. **Physical and chemical constants of aged oil from Puri.**—A sample of the oil from Puri, several months old, gave the following constants:—

Specific gravity at $30^{\circ}C$	0.941
Acid value	44.5
Saponification value	197.8
Iodine value	91.6

15. **Unsuitability of the oil as a cold process soap-stock.**—Although the oil is at present being used by some soap-makers for the manufacture of soap by the cold process yet it cannot be pronounced as a suitable stock to be treated by that method. The presence of comparatively large proportions of free fatty acids and resinous matter in the oil interferes with the slow and progressive saponification of the stock, so necessary for success in the cold process, and thereby renders the complete saponification of the oil an impossibility. The resulting soap contains high proportions of unsaponified oil and is sticky.

16. **Properties and characteristics of Punnal soap.**—Regarding the soap-making qualities of the oil it has been found that the oil is saponified easily on boiling and the soap made entirely from this oil has, as already stated, a dark yellowish-brown colour. The soap is, however, free from odour and has a soft consistency. It has good cleansing properties and a very mild action on the skin. The lather is quick and abundant.

17. **Behaviour on saponification and graining.**—The behaviour of this stock in course of saponification and graining for the preparation of the Bengal type of moulded soap is described below.

The first addition of alkali produces a deep yellow colour and the original greenish colour of the oil disappears with the boiling of the mixture. As the saponification proceeds the colour of the solution gradually changes from deep yellow to dark brown with a cloudiness. On the completion of saponification the cloudiness disappears and a clear soap of a dark brown colour is obtained. After the addition of salt the graining of the soap proceeds normally though at times the soap shows a tendency to become much frothy. The froth gradually becomes less and less but a little of it persists at the top even after the completion of the graining. The thin layer of froth is then gathered in one side of the pan by a trowel leaving bare the neat soap. This soap is taken out by means of the trowel and put in earthenware cups which are used as moulds. The soap solidifying in the cups has a somewhat soft consistency and a dark brown colour in spite of the fact that a considerable quantity of the colouring matter is eliminated in the spent lye.

18. **Solidification point of the grained soap.**—The grained soap consists of the partially hydrated sodium salts of the fatty acids originally present in the stock, and the temperature for its solidification on the saline lye has an important bearing on the nature of the moulded soap produced. The solidification point of soap from this oil grained normally by means of common salt has been found to be 54°C. For purposes of comparison and for determining suitable mixtures with other stocks, the solidification points of the grained soaps of other standard stocks are given below:—

Tallow	66.5°C
Mowha oil	57.0°C
Groundnut oil	45.0°C

It will thus be seen that Punnal oil is a considerably softer stock than tallow but much less so than mowha oil. On the other hand it is harder than groundnut oil.

19. **Colour of the soap.**—The brownish colour which the soap develops is calculated to be a disadvantage in the use of the oil by itself. In admixture with other stocks, however, the oil can be used with great advantage in the making of all varieties of moulded soaps having high cleansing and lathering properties. By refining the oil, a process for which is described later on, larger proportions of this stock can be used in admixture with other stocks in the making of the abovementioned soaps. The refining eliminates most of the resinous matter present in the oil and considered to be responsible for the disadvantageous colouring substance.

20. **Proportion of the oil in combination with other stocks in making moulded soap—35 per cent.**—In making moulded soap of a fine quality producing an abundance of lather and having high cleansing properties a mixture of this oil with other stocks can be so judiciously made as to produce a soap having only a pale yellow colour. Examples of such combinations are given below using Punnal oil to the extent of 35 per cent. of the entire mixture, the consistency of the resulting soap decreasing in hardness as the serial number of the combination increases.

I.—Combinations using 35 per cent. Punnal oil.

		Parts.			Parts.
1.	Tallow ..	40	5.	Tallow ..	25
	Groundnut oil ..	10		Groundnut oil ..	15
	Mowha oil ..	15		Mowha oil ..	10
	Punnal oil ..	35		Punnal oil ..	35
2.	Tallow ..	35		Castor oil ..	8
	Groundnut oil ..	15		Cottonseed oil ..	7
	Mowha oil ..	15	6.	Tallow ..	20
	Punnal oil ..	35		Groundnut oil ..	20
3.	Tallow ..	30		Mowha oil ..	10
	Groundnut oil ..	10		Punnal oil ..	35
	Mowha oil ..	15		Castor oil ..	5
	Punnal oil ..	35		Cotton seed oil ..	10
	Castor oil ..	10	7.	Tallow ..	15
4.	Tallow ..	30		Groundnut oil ..	25
	Groundnut oil ..	10		Mowha oil ..	10
	Mowha oil ..	10		Punnal oil ..	35
	Punnal oil ..	35		Castor oil ..	5
	Cottonseed oil ..	15		Cottonseed oil ..	10

The above proportions can be varied within narrow limits to produce practically the same type of soap.

21. **Proportion of the oil in combination with other stocks in making moulded soap—25 per cent.**—If it is desired to have soaps of a paler colour, the percentage of Punnal oil should be reduced. Examples of such combinations are given below using the oil to the extent of 25 per cent., the same rule applying as regards consistency as in the first series of combinations.

II.—Combinations using 25 per cent. Punnal oil.

			Parts.				Parts.
1.	Tallow	..	35	5.	Tallow	..	20
	Groundnut oil	..	15		Groundnut oil	..	20
	Mowha oil	..	25		Mowha oil	..	20
	Punnal oil	..	25		Punnal oil	..	25
2.	Tallow	..	30		Castor oil	..	7
	Groundnut oil	..	20		Cottonseed oil	..	8
	Mowha oil	..	25	6.	Tallow	..	15
	Punnal oil	..	25		Groundnut oil	..	25
3.	Tallow	..	30		Mowha oil	..	20
	Groundnut oil	..	15		Punnal oil	..	25
	Mowha oil	..	20		Castor oil	..	5
	Punnal oil	..	25		Cottonseed oil	..	10
	Cottonseed oil	..	10	7.	Tallow	..	10
4.	Tallow	..	25		Groundnut oil	..	20
	Groundnut oil	..	25		Mowha oil	..	25
	Mowha oil	..	15		Punnal oil	..	25
	Punnal oil	..	25		Castor oil	..	5
	Castor oil	..	10		Cottonseed oil	..	15

The above combinations are only typical and as such admit of slight variations without producing considerable difference in the resulting soaps.

22. **Proportion of the oil in combination with other stocks in making moulded soap—50 per cent.**—Punnal oil to the extent of 50 per cent, and even more can be used if the brownish colour is not considered a disadvantage. Examples of such combinations are given below:—

III.—Combinations using 50 per cent. Punnal oil.

			Parts.				Parts.
1.	Tallow	..	50	4.	Tallow	..	20
	Punnal oil	..	50		Groundnut oil	..	15
2.	Tallow	..	30		Mowha oil	..	15
	Mowha oil	..	20		Punnal oil	..	50
	Punnal oil	..	50	5.	Tallow	..	10
3.	Tallow	..	30		Groundnut oil	..	20
	Groundnut oil	..	10		Mowha oil	..	20
	Mowha oil	..	10		Punnal oil	..	50
	Punnal oil	..	50	6.	Groundnut oil	..	25
					Mowha oil	..	25
					Punnal oil	..	50

23. **Purely white soap impossible with Punnal oil.**—Purely white soap cannot be made by the use of Punnal oil, but a near approach to that colour can be obtained by the admixture of this oil up to a limit of, say, 10 per cent, with other white-soap stocks.

24. **Proportion of refined Punnal oil in combination with other stocks in making moulded soap.**—Punnal oil refined by the method described below can be used in larger proportions without producing a deeply coloured soap. By the process of refining, the oil is freed from the resinous acids which carry most of the colouring matter. The refined oil can be used in any proportion, from 10 to, say, 80 per cent., in admixture with other common soap-stocks. If necessary, it can be used by itself without the admixture of any other stock. The soap made in this way will be quite good for ordinary purposes, but it cannot be expected to have all the properties of an ideal soap. The following combinations are given as examples:—

IV.—Combinations using refined Punnal oil.

	Parts.		Parts.
1. Tallow	26	4. Tallow	25
Punnal oil	71	Mowha oil	20
Rosin	3	Punnal oil	52
		Rosin	3
2. Tallow	30	5. Mowha oil	52
Punnal oil	66	Punnal oil	45
Rosin	4	Rosin	3
		6. Tallow	20
3. Tallow	25	Mowha oil	16
Mowha oil	12	Groundnut oil	20
Punnal oil	60	Punnal oil	40
Rosin	3	Rosin	4

25. **Purification of Punnal oil.**—The purification is carried out as follows: 100 parts (by weight) of Punnal oil are taken in an iron kettle and 300 parts of water containing 9 parts of sodium carbonate are added to the oil. The mixture is boiled and a resinous substance is thereby extracted by the alkaline water along with the colouring matter of the oil. It generally suffices to boil the mixture for one hour. It is then allowed to cool and settle when the light yellow coloured oil rises at the top. This is decanted off or let out by means of cocks provided at different levels of the pan. The boiling of the mixture can be continued for one hour more or even longer but very little further improvement in the matter of the removal of colour can be effected thereby. The sodium carbonate solution contains the colouring matter as well as the resinous substance and is coloured dirty green and has a cloudiness. The decrease in the weight of the oil by this treatment is generally 15 to 20 per cent. of which a large portion can be recovered by a process described later on. The treatment with sodium carbonate as described above removes the free fatty acids of the oil as evidenced by the very low acid value of the purified oil, and this is accompanied by a lowering of the density and the iodine value as the following constants of the refined oil will show:—

Specific gravity at 31°C	0.918
Acid value	1.50
Iodine value	82.7

26. **Character of soap from purified Punnal oil.**—The purified oil gives a soap which on salting and graining on lye has a solidification point of 55°C , which is higher by 1°C than that of the soap made from the original oil.

27. **Recovery of free fatty and resin acids.**—The free fatty acids and also the resin acids removed from the original oil remain as soaps in the greenish solution over which the purified oil forms a layer as described above, and are recovered therefrom by the use of sulphuric acid. The action of the sulphuric acid on the solution is firstly to neutralise the free sodium carbonate in the latter and then to decompose the soaps with the liberation of the fatty and resin acids. The addition of the sulphuric acid should be stopped as soon as the entire quantity of the soaps has been split up in this way. On allowing the mixture to settle the liberated liquid fatty and resin acids form the upper layer which has a dirty green colour. The operation can be carried out in iron pans provided no large excesses of sulphuric acid are used.

28. **Colour of soap from recovered fatty and resin acids.**—When the acids separated as above are subsequently saponified for actual soap-making their dirty green colour becomes deep yellow on the addition of alkali and the resulting soap has a deep yellow to brown colour. These acids can therefore be utilised along with other stocks for the making of cheap moulded soaps in which the yellowish brown colour is not an objection.

29. **Colour of soap from purified Punnal oil.**—In making soap from the Punnal oil purified in the above manner the light yellow colour which the oil retains even on purification is, in the most part, eliminated in the spent lye during the salting of the soap. In the process of saponification the yellow colour of the oil turns brown. On salting, the resulting soap has a pale yellow colour which appears paler on admixture with other stocks.

It is hoped that once the high qualities of the oil are recognised in the making of soap, particularly of the moulded variety, it will be in great demand and this will act as a powerful stimulant to the increased production of oil.

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