

these materials can generally be chopped fine and rendered fluffy, a thing which is not possible with laboratory mills when the oil content is high, and are then used in moisture and oil content determinations.

It was found that filtration is improved when some Polycel-RCB-80 or Super-cel filter aids are added to the contents in the container after trituration and mixed for a few seconds before pouring into the funnel. In some cases, it was also found, a funnel of coarse porosity worked better than the medium porosity funnel used for copra.

The removal of the solvent from the oil of 75-gm. samples of most of these seeds was accomplished in only 1/2 hour because these materials have generally a lower oil content than copra does.

Detailed procedures for the analyses of the various seeds have not been worked out because in our mill we seldom crush anything other than copra. Such details will be left to those who will find the method advantageous. Recently the Soybean and Flaxseed Advisory Committee voiced the need for quick and accurate determinations of oil content and quality of oil in oil seeds, stating that "such quick tests would facilitate trading and be a boon to sound production

and marketing practices" (12). The authors hope that the present method will assist in fulfilling that need.

REFERENCES

1. P. W. Tompkins. "Copra Sampling, Inspection and Analysis," Oil and Fat Industries, 4, No. 1 (Jan., 1927).
2. P. W. Tompkins. "Copra Quality Under New Rules," Oil and Soap, XVIII, No. 5 (May, 1941).
3. John P. Nielsen and G. S. Bohart. "Determination of Crude Lipid in Vegetable Matter," Industrial and Engineering Chemistry, Analytical Edition, 16, No. 11 (Nov., 1944).
4. R. B. Oesting and I. P. Kaufman. "Rapid Determination of Fat in Meat and Meat Products," Industrial and Engineering Chemistry, Analytical Edition, 17, No. 2 (Feb., 1945).
5. Joseph Hamilton and Seymour G. Gilbert. "Rapid Method for Determining Oil Content of Tung Kernels," Industrial and Engineering Chemistry, Analytical Edition, 19, No. 7 (July, 1947).
6. A. L. Ayers and J. J. Doodly. "Laboratory Extraction of Cotton Seed With Various Petroleum Hydrocarbons," The Journal of the American Oil Chemists' Society, XXV, No. 10 (Oct., 1948).
7. Lawrence Zeleny and D. A. Coleman. Rapid Determination of Oil Content and Oil Quality of Flaxseed, Technical Bulletin No. 554, United States Department of Agriculture, Washington, D. C., Feb., 1937.
8. International Chemical Union. "Minutes of Meeting of International Commission on Fats and Oils," Part II, The Journal of the American Oil Chemists' Society, XXVI, No. 3 (March, 1949). (London, England, July, 1947; translated by C. L. Hoffpauir.)
9. George S. Jamieson. Vegetable Fats and Oils (1943), p. 375.
10. Official and Tentative Methods of Analysis of the Association of Official Agricultural Chemists (1945).
11. Official and Tentative Methods of the American Oil Chemists' Society (1947).
12. "Soybeans and Flaxseed Advisory Committee," The Cotton Gin and Oil Mill Press, August 21, 1948, p. A-3.

[Received for publication May 6, 1949]

Oil From the Kernels of Lalob Fruit, *Balanites Aegyptiaca*¹

S. A. HUSSAIN, State of Hyderabad (India), and F. G. DOLLEAR and R. T. O'CONNOR, Southern Regional Research Laboratory,² New Orleans 19, Louisiana

Balanites aegyptiaca is a tree 20 to 30 feet high which is found principally in West Africa, Sudan, Uganda, Tanganyika, Palestine, and Arabia. The fruit of this tree is known locally by such names as "betu," "lugba," "heglig," and lalob. The kernel of the fruit, which contains about 46% oil, amounts to 9 to 10% by weight of the whole fruit.

The fruit, which usually falls to the ground on ripening, is often parasitized by the larvae of an unidentified noctuid moth. The caterpillar develops inside of the fruit and when ready to pupate, eats its way to near the surface, leaving a membranous window through which the adult moth emerges. This parasitism is responsible for considerable loss of kernels of wind-fallen fruit.³

Except for two anonymous reports (1, 2) from the Imperial Institute of London in 1908 and 1935, respectively, practically no information appears to have been published on the characteristics and composition of lalob kernel oil. Because of the current interest in exporting lalob kernels for crushing and the reported similarity of the oil to cottonseed oil, a comprehensive investigation was made of the fruit and oil by modern chemical and physical methods (3).

Experimental

Material: Lalob fruit used in this investigation was grown in the Fung district of Sudan.⁴ The fruit

resembles the date in size and appearance and consists of a loose, thin brownish-yellow skin, covering a brown sticky pulp, embedded in which is a hard woody shell enclosing a light yellowish kernel.

Components: The percentage distribution of the component parts of lalob fruit together with the contents of moisture and lipids made on 10 selected fruits weighing an average of 7 grams each is given in Table I.

TABLE I
Distribution of Principal Components of Lalob Fruit With Their Moisture and Lipid Content

Component	Proportion of whole fruit	Moisture	Lipids
	%	%	%
Outer covering.....	21.6	16.2
Sticky pulp.....	34.8	26.8
Shell.....	34.1	10.5
Kernel.....	9.5	5.7	46.5

Extraction of Oil: A large lot (8.8 kg.) of lalob fruit were dried in a vacuum oven at 158°F. for 20 hours to facilitate cracking and separation of kernels. Only 2.2% moisture could be removed by drying, and the pulp was still very sticky. The incompletely dried fruit was cracked and the kernels separated by hand. All worm-damaged fruit (12% by weight) were rejected, and only sound kernels were used for extracting the oil. The original lot of dried lalob fruit yielded 10.2% by weight of sound kernels.

The kernels (908 g.) were ground in a food grinder and extracted with commercial hexane (b.p. 146°F.) in a laboratory all-glass extractor. At the completion of extraction the bulk of the solvent was removed

¹ Presented at the 40th Annual Meeting of the American Oil Chemists' Society, New Orleans, La., May 10-12, 1949.

² One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

³ Information supplied by A. W. M. Disney, Department of Economics and Trade, Sudan Government, Khartoum.

⁴ The lalob fruit used in this investigation was supplied by the Greene Trading Company inc., New York, via the Sudan Trading Company Ltd., Khartoum, Sudan, Africa.

from the miscella by distillation and the last traces by heating under vacuum and stripping with nitrogen. The residual oil (425 g.) was clear light yellow in color.

Composition of Meal: The residual meal after desolventizing by spontaneous evaporation at room temperature was analyzed with the results given in Table II. The percentages of protein, ash, and crude fiber are similar to those previously reported in the literature (2) for this material.

TABLE II
Composition of Extracted Lalob Kernel

	As-received basis	Moisture-free basis
	%	%
Moisture.....	8.73
Nitrogen.....	8.06	8.83
Protein ($N \times 6.25$).....	50.37	55.19
Ash.....	6.30	6.90
Potassium.....	0.74	0.81
Phosphorus.....	1.02	1.12
Calcium.....	0.35	0.38
Crude fiber.....	4.60	5.04
Total sugar, calculated as invert sugar.....	7.15	7.84

Characteristics and Composition of Oil: The characteristics and fatty acid composition of the oil were determined by the methods prescribed by the American Oil Chemists' Society (3) except for the unsaponifiable matter (4), thiocyanogen value (5), and hydroxyl value (6), which were determined by the methods described in the references cited.

The characteristics of the present sample of lalob kernel oil are compared in Table III with the values previously reported in the literature (1, 2).

TABLE III
Physical and Chemical Characteristics of Lalob Kernel Oil

Source	Present sample	Literature values (1, 2)		
		Uganda	No. thern Nigeria	Sudan
Characteristic	Sudan	Uganda	No. thern Nigeria	Sudan
Specific gravity.....	0.9156 ^a	0.9220 ^b	0.919 ^b	0.9187 ^b
Refractive index, n_D^{20}	1.4643	1.4640
Lovibond color, 5 $\frac{1}{4}$ " cell.....	35Y-3.9R
Titer, °C.....	36.0	34.6	34.0
Saponification value.....	191.3	191.6	196.7	194.2
Iodine value.....	102.2 ^c	98.0 ^d	92.5 ^e	98.2 ^e
Thiocyanogen value.....	66.6
Hydroxyl value.....	2.9
Reichert-Meißl value.....	0.19	trace
Polenske value.....	4.27
Acid value.....	0.26	0.9	5.0	1.4
Unsaponifiable matter, %.....	2.0	0.3	0.6

^aAt 25°/25°C. ^bAt 15°/15°C. ^cWijs, 30 min. ^dWijs, 3 hrs. ^eHübl, 17 hrs.

The fatty acid composition was calculated from the iodine-thiocyanogen value and from spectrophotometric measurements of the oil before and after alkali isomerization for 25 minutes at 180°C. with an ethylene glycol-potassium hydroxide reagent, using nitrogen protection. Calculations from spectrophotometric data were made according to the equations of Brice *et al.* (7). The results are shown in Table IV. Reference to this table shows that the results obtained by the two methods are in close agreement. The saturated acids were also determined independently by the modified-Bertram oxidation method (8). These results are also included in Table IV. The present results are somewhat higher for linolein and somewhat lower for saturated glycerides than those previously reported for lalob kernel oil (1).

Fractional distillation of a small batch of methyl esters prepared from the saturated acids, which were

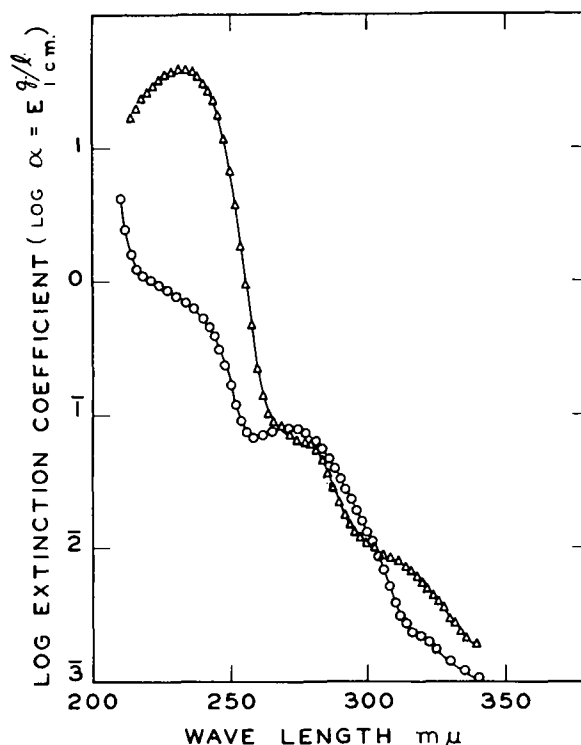


Fig. 1. Ultraviolet absorption spectra of lalob kernel oil, before (—○—) and after (—△—) alkali isomerization.

separated by low temperature crystallization, and determination of the saponification equivalent of the distilled ester fractions gave qualitative indications that the saturated acids of lalob kernel oil are composed mainly of palmitic and stearic acids.

Spectrophotometric Absorption: The absorption spectra of lalob kernel oil before and after alkali isomerization is shown in Figure 1, and the absorption spectrum of the oil in the near ultraviolet and visible region is shown in Figure 2. The observed values of the absorption maxima and minima are recorded in Table V.

Lalob kernel oil has a rather low absorption in the ultraviolet and visible region compared to other crude oils (9), indicative of the presence of only a small number of oil-soluble pigments. The sharp-banded absorption maxima of low intensity at 425, 450, and 478 $m\mu$ indicate the predominance of a specific carotenoid pigment. In order to characterize this pigment a solution of lalob kernel oil in isoctane was passed through a column of specially activated magnesium oxide.⁵ Under these conditions the magnesium oxide will retain the non-carotene carotenoids as well as all

⁵ Activated magnesia, Micron brand No. 2642 Westvaco Chlorine Products Company, Newark, Calif.

TABLE IV
Composition of Lalob Kernel Oil Calculated on a Glyceride Basis

Glyceride	Method of determination			Literature value ^a
	Spectrophotometric	Iodine-thiocyanogen	Bertram oxidation	
	%	%	%	
Olein.....	31.14	30.50	33
Linolein.....	42.96	43.84	33
Linolenin.....	0.027
Saturated.....	25.30	23.66	22.0	34
Unsaponifiable matter.....	2.0

^a Method of calculation not reported (1).

TABLE V
Spectral Absorption Characteristics of Lalob Kernel Oil

Minima		Maxima	
Wave length, $m\mu$	$E_{1\text{cm.}}^{F/A} \times 10^3$	Wave length, $m\mu$	$E_{1\text{cm.}}^{F/A} \times 10^3$
259	68.51	273	78.56
366	0.62	425	1.30
430	1.26	450	1.61
468	1.17	478	1.34
635	0.016	670	0.05

of the chlorophyll derivatives in the form of bands near the top of the column and the carotenes throughout the lower portions of the column. The solution passing through the column was found to be free of pigment.

The carotenes are adsorbed so loosely on this type of column that the addition of a small portion of a polar solvent will elute them without removing the other carotenoids or chlorophyll pigments (10). The column was developed by washing with a solution of isooctane containing 2% absolute ethanol. The solution passing through the column was colored bright yellow, and the column was almost completely decolorized by this treatment. Attempts to elute additional pigments from the column with other more polar solvents gave only weakly colored solutions, indicating that except for extremely small traces of other colored substances, the pigment of lalob kernel oil is a carotene.

The colored solution in the isooctane-two-per cent absolute ethanol solvent was examined with a Beckman DU spectrophotometer. The spectrophotometric curve (Figure 3) obtained was identical with that of α -carotene as shown in Figure 3. The triangular points on Figure 3 were obtained with a highly purified sample of α -carotene prepared by a previously described chromatographic method (11), when measured in the isooctane solvent containing 2% ethanol. The agreement between the absorption spectrum of the latter solution and that obtained with a solution of lalob kernel oil proves rather conclusively that the pigment of this oil is mainly α -carotene.

Summary

Lalob fruit from the Sudan has been examined by modern chemical and physical methods. The kernels which amount to 9 to 10% of the fruit contain ap-

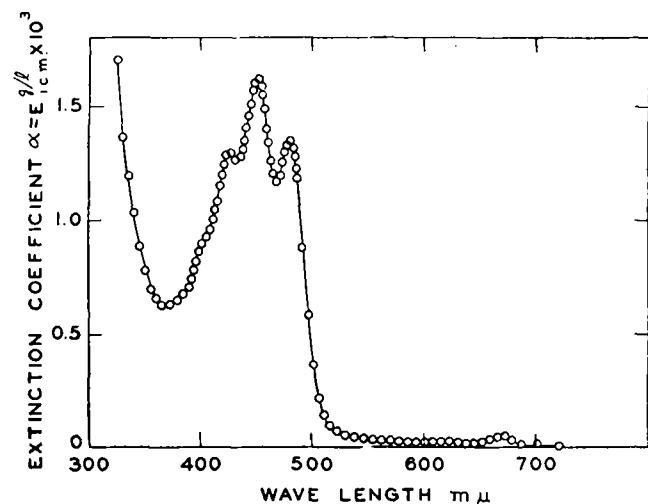


FIG. 2. Absorption spectrum of lalob kernel oil in the near ultraviolet and visible regions.

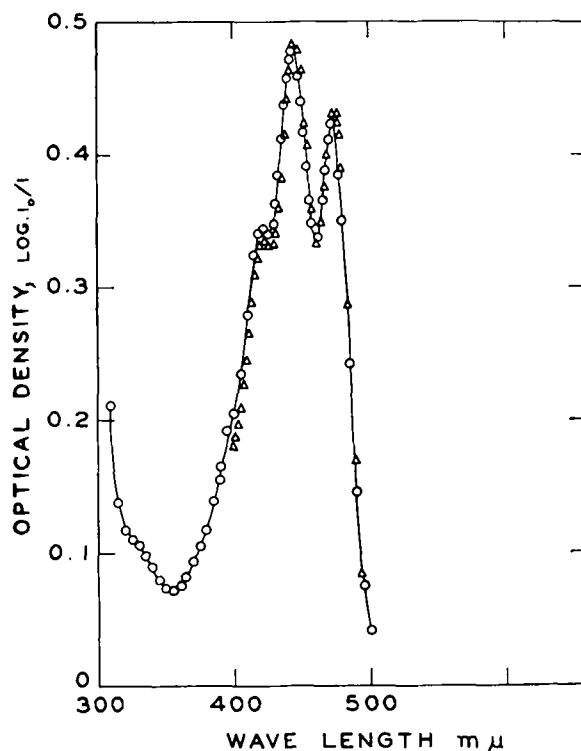


FIG. 3. Absorption spectrum of the pigment isolated from lalob kernel oil (-O-) compared with that for pure α -carotene (- Δ -).

proximately 46% oil. Extraction of the ground kernels with commercial hexane yields a light yellow oil, having a composition calculated from iodine-thiocyanogen values corresponding to 43.8% linolein, 30.5% olein, 23.7% saturated acid glycerides, and 2.0% unsaponifiable matter. Calculation of the composition from ultraviolet absorption measurements gave results in fairly close agreement with those obtained by the iodine-thiocyanogen method. It was shown that the color of the crude oil was due almost entirely to the presence of α -carotene.

Acknowledgment

The authors wish to thank E. T. Field for assistance in obtaining the spectrophotometric data reported in this paper and A. F. Cucullu, V. O. Cirino, E. R. McCall, and C. Leslie for the analysis of the lalob kernel meal.

REFERENCES

1. Anon., Bull. Imp. Inst., 6, 364-366 (1908).
2. Anon., Bull. Imp. Inst., 33, 274-277 (1935).
3. American Oil Chemists' Society, Official and Tentative Methods, 2nd ed., ed. by V. C. Mehlenbacher, Chicago, 1946.
4. Cocks, L. V., Report of the Subcommittee on Determination of Unsaponifiable Matter in Oils and of Unsaponified Fat in Soaps, Analyst, 58, 203-211 (1933).
5. Lambou, M. G., and Dollear, F. G., Oil & Soap 22, 226-232 (1945).
6. West, E. S., Hoagland, C. L., and Curtis, G. H., J. Biol. Chem., 104, 627-634 (1934).
7. Brice, B. A., Swain, M. L., Schaeffer, B. B., and Ault, W. C., Oil & Soap, 22, 219-224 (1945).
8. Pelikan, K. A., and von Mikusch, J. D., Oil & Soap, 15, 149-150 (1938).
9. O'Connor, R. T., Field, E. T., Jefferson, M. E., and Dollear, F. G., The Influence of Processing on the Spectral Properties of Vegetable Oils, presented at the 40th Annual Meeting of the American Oil Chemists' Society, New Orleans, La., May 10-12, 1949.
10. Official and Tentative Methods of Analysis, Association of Official Agricultural Chemists, 6th ed., Washington, 1945, pp. 600-606; rev. March 17, 1948, J. Assoc. Off. Agr. Chem., 31, 111-112 (1948).
11. O'Connor, R. T., Heinzelman, D. C., and Jefferson, M. E., Ind. Eng. Chem., Anal. Ed., 18, 557-562 (1946).