REGINALD R. LEWIS Lindsay, California

"O LIVE OIL foots or sulfur olive oil is produced by extracting with carbon disulfide olive pomace from which the expressable olive oil has been removed by hydraulic pressing. Sulfur olive oil is used exclusively in soapmaking." This quotation approximately summarizes the information on sulfur olive oil imparted by textbooks devoted to fatty oils. Thus apparently there is certain misinformation and a paucity of knowledge of the production and uses of sulfur olive oil, according to the writer's first hand information regarding this product.

The term sulfur olive oil has become a misnomer. Many years ago when the Mediterranean countries started to extract olive pomace carbon disulfide was more readily available and cheaper than hydrocarbon solvents and was used as the extracting solvent; hence the name sulfur olive oil.

There is no evidence, however, to indicate that California producers ever used carbon disulfide as a solvent and its use in Europe has greatly diminished. Commercial hexane is in almost universal use although a few small producers use the fireproof chlorinated solvents. It would therefore appear that extracted olive oil would be a more appropriate term than sulfur olive oil. The term olive oil foots, also applied to extracted olive oil, is unfortunate because of possible confusion of this product with the soapstock resulting from the refining of olive oil.

Despite the fact that carbon disulfide is not used as a solvent in the United States, the A.O.C.S. Silver Coin Test and the Silver Benzoate Test are used to detect the sulfur supposedly present in extracted olive oil. The writer has subjected genuine pressed olive oils to the above tests and obtained positive results which were attributed to sulfur-containing sprays used on olive trees.

Efficient pressing of olives produces an olive pomace of about 8% oil content, but a substantial percentage of California pomace contains as much as 12% to 13% oil. The olive pomace is ground, dried, and brought into intimate contact with hexane, the solvent distilled off leaving the extracted olive oil. Both batch and continuous extraction plants are in use in California.

Extracted olive oil differs from pressed olive oil because of the extraction of resins and gums from the pits and the extraction of relatively large percentages of chlorophyll from the skins and pulp. The unsaponifiable matter of extracted oil tends to be higher than in pressed oil; the iodine value lower; the color a much deeper green; and the apparent viscosity much higher.

"Sulfur olive oil is used exclusively for soapmaking." This statement is, to say the least, debatable. To the writer's knowledge, no pressed or extracted olive oil produced in California is used for soapmaking if it can be processed into an edible product. The scarcity and high price of olive oil during the war years gave especial impetus to perfecting refining techniques to convert all olive oil into edible oil. Like the processing of other vegetable oils, olive oil processing involves reducing the free fatty acid content, improving the color of dark oils, winterizing oils that deposit "stearine," and deodorizing those of inferior flavor and odor.

Oil pressed from fresh, sound olives is invariably low in free fatty acids ranging from a few tenths of 1% to as high as 1%. Ordinarily such oil requires no processing other than separating it from water and filtering. Oil pressed from damaged fruit (bruised, mouldy, or fermented) may run as high as 10% FFA, be of an unacceptable color, and possess poor flavor and odor. Pressed oil of this type presents no processing difficulties; it can be readily caustic soda refined, the loss running approximately twice the FFA content; it bleaches readily, and will deodorize to a bland, odorless oil.

It is extracted olive oil which presents difficulties in processing; neutralizing is troublesome, bleaching is often extremely difficult, and winterizing to remove gums and gelatinous materials is slow and arduous.

Oil which has been extracted from fresh pomace is generally fairly low in acidity, in the vicinity of 5%. However, during the olive crushing season pomace accumulates much faster than extraction plants can extract it and consequently most pomace undergoes adverse storage from a week to several months. During this storage fermentation and heating occur so that the oil extracted is high in acidity, ranging from 10% to 40% and even higher. Acidity of 20% would be near the average for a season's production.

Obviously, processing such an oil requires considerably different techniques from that employed on oils such as corn, cottonseed, and soya where acidities of 2% or 3% are considered high. Coupled with the difficulty of high acidity is the fact that olive oil is readily saponifiable so that excesses of alkalies in refining are dangerous and to be avoided.

Preliminary to neutralization in the conventional batch refining kettle, treatments to precipitate gums and resins are desirable. Washing the crude oil with a salt solution containing a small percentage of an organic acid such as oxalic acid is beneficial. This treatment removes certain emulsion-promoting substances.

Neutralization of an oil containing 20% FFA requires special procedures to promote a separation of the soap from the oil and subsequent settling of the soap in the refining kettle. A procedure which has proved successful is to heat the crude oil during agitation to 140° F. to 150° F., emulsify with 10% water and then add enough 45° Bé lye to exactly neutralize the free fatty acids. Agitation is discontinued immediately after the addition of the alkali. An alternate method involves heating the oil during agitation to 125° F., adding sufficient 20° Bé lye to exactly neutralize the free fatty acids, heating to 135° F. and adding 10% to 15% of a 10% soda ash solution, heating to 160° F. and then stopping agitation. The soda ash solution promotes "graining" of the soap and aids in settling.

The refining loss using the above methods has been somewhat more than twice the FFA content of the oil. Oils as high as 35% FFA content have been refined, but 35% is generally considered the maximum for refining by conventional methods.

The writer has perfected methods and equipment for the esterification with glycerol of the FFA in very high acid extracted olive oil. The esterification is continued until the oil has an FFA content of about 5%. The oil is next subjected to conventional high temperature, vacuum deodorization, not for the purpose of deodorizing at this point, but to convert mono- and diglycerides to triglycerides. Attempting to alkali refine esterified olive oil containing traces of mono- and diglycerides results in emulsions.

Bleaching extracted olive oil is often extremely difficult. In general, massive dosage of activated clays and activated carbon and very high bleaching temperatures are required. As an example, 10% activated clay plus 1% activated carbon at a temperature of 300° F. (under vacuum) has been necessary to secure a merchantable color. The addition of $\frac{1}{2}\%$ of a 50% solution of sulfuric acid to the oil-clay mixture has resulted in improved color in some instances. Oils which are objectionably red after bleaching may be agitated at 200° F. with $\frac{1}{2}\%$ to 1% of alfalfa meal and filtered. Oils so treated acquire a delicate green color characteristic of pressed olive oil.

Winterizing of extracted olive oil is necessary to produce an oil which will remain clear at room temperatures. This process is slow because, when chilled, the oil forms a gel rather than crystals. A paper dressed filter press and low filtration pressure is required for satisfactory processing.

Deodorization presents no special difficulties. Deodorization at 400° F. at 5 mm. to 10 mm. pressure for 4 or 5 hours yields a bland, odorless oil. To digress for a moment, the writer at one time was required to process an olive oil which had been accidentally contaminated with cottonseed oil. The oil showed a very positive Halphen test, a strong pink, indicating 1% or more contamination. After preliminary laboratory experiment, a 20,000-lb. batch of the oil was placed in the deodorizer together with 1 lb. of sulfur, 1 gallon of amyl alcohol, and 1 gallon of carbon disulfide. After 5 hours' deodorization the oil was bland and odorless and showed an absolutely negative Halphen test! Deodorization without the Halphen reagents did not destroy the Halphen reaction.

Refined extracted olive oil conforms closely with the chemical and physical characteristics of pressed olive oil but, of course, does not have the characteristic olive flavor for which olive oil is so highly valued. Consequently, refined olive oil is almost never marketed as such, but is blended with pressed olive oils with strong olive flavors and the blend marketed as "Pure Olive Oil."

Summary

Extracted olive oil is not used exclusively for soapmaking but in fact is used to the utmost extent in edible oils. General processing procedures are outlined for the production of edible olive oil from high acid extracted olive oil.

Edited by

M. M. PISKUR and SARAH HICKS

Abstracts

Oils and Fats

THEORIES OF AUTOXIDATION. ANTIOXYGENS. C. showed that t Paquot (Lab. Corps Gras). Inds. corps gras 3, 111-17, tion of eithe

140-3 (1947). THE USE AND ROLE OF FATS IN THE TANNERY. C. Gastellu (École Française de Tannerie). Inds. corps

gras 3, 102-10, 132-9 (1947). CONTINUOUS BUTTER PRODUCTION—A COMPARISON OF METHODS. F. H. McDowall (Massey Agr. Coll., Palmerston North, New Zealand). Food Inds. 19, 909-12, 1034, 1036, 1038, 1040 (1947). Six welldefined systems of continuous processing are described. These new methods apparently possess many advantages, but they also pose a number of problems in operation and distribution.

SOLVENT EXTRACTION OF COTTONSEED AND PEANUT OILS. E. L. D'Aquin, J. J. Spadaro, H. L. E. Vix, J. Pominski, L. J. Molaison, and E. F. Pollard (Southern Regional Research Lab., New Orleans, La.). Oil Mill Gaz. 51, No. 10, 17-19 (1947). The experiments were undertaken to obtain information required for the design and operation of pilot plant equipment and for the development of processes in solvent extraction. Crude cottonseed oils were produced on a pilot plant scale from a single lot of prime cottonseed by hexane extraction of the cooked and the uncooked meats and by standard hydraulic pressing of the cooked meats. Refining and bleached color tests showed that the crude oil obtained by solvent extraction of either the cooked or uncooked meats with removal of solvent at a temperature below 140° F., compared favorably in quality and grade to the oil prepared by hydraulic pressing. The lightest-bleached oil colors were obtained from the oil produced by solvent extraction of the uncooked meats. The solventextracted oils appeared to refine better and still give comparable color and refining tests. The oils produced from the 3 different processings were subjected for 1 hour to heating temperatures from 150-240° F. In all cases the bleached oils were darker in color as the heating temperature was raised. The oil extracted from the uncooked meats, although of lighter original bleached color, appeared to degrade in color more rapidly at the 150° and 240° F. temperature levels than did the oil extracted from the cooked meats; and its final bleached color was darker after heating at 240° F. The hydraulic-pressed oil was markedly less affected by heating than either of the 2-hexane-extracted oils and did not exhibit a sharp bleached color degradation as did the hexane-extracted oils. It is not known to what extent the darker bleached oil colors which resulted from heating the crude oil can be attributed to actual darkening of the oil in situ during heating or to fixation of the original pigments. (Chem. Abs.)