at the outlet of the column. The carotene content was determined by measuring the absorption at the wavelength of 450 mμ and taking readings of its concentration from the standard curve prepared previously. The above-described procedure was applied with all the determinations which were carried out. The carotene contents were expressed in its predominant component, i.e. beta-carotene.

The results obtained for the investigated rapeseed oils are given in Table III.

Further investigations are being continued on the accuracy of quantitative determinations of chlorophylls and pheophytins in rapeseed oil.

Studies on carotenes contents in rapeseed oil are also being continued.

REFERENCES

1. Kaufmann, H. P., and M. Vogelmann, Farbchemiker *61*, 6–10 (1959).
2. Kurita, K., Chem. Abs. *51*, 1597 2 e (1957).
3. Mingot, L., Anales Inv. Agron. *5*, 295 (1956).
4. Mackinney, G., and M. Joslyn, J. Am. Chem. Soc. *62*, 231–232 (1940).
5. Aronoff, S., Chem. Rev. *47*, 175–195 (1950).
6. Kaufmann, H. P., J. Baltes, H. J. Heinz and P. Roever, Fette und Seifen *52*, 35 (1950).
7. Glick, D., "Methods of Biochemical Analysis," New York, London, Vol. IV, 1957, p. 26.
8. Peach, K., and M. V. Tracey, "Moderne Methoden der Pflanzenanalyze," Springer Verlag, Berlin, Vol. IV, 1955, p. 145.
9. Harris, D. G., and F. P. Zscheile, Bot. Gaz. *104*, 515 (1943).
10. Zscheile, F. P., and C. L. Comar, Bot. Gaz. *102*, 463 (1941).
11. Falk, H., Planta *51*, 49 (1958).

[Received October 19, 1964—Accepted April 9, 1965]

A Simplified and Precise Flavor Evaluation Procedure for Determining Oxidative Rancidity in Vegetable Oils[1]

C. JANE WYATT and E. A. DAY, Department of Food Science and Technology
Oregon State University, Corvallis

Abstract

High correlations were observed between the flavor evaluation procedure and objective chemical tests for measuring oxidative deterioration in stored corn, cottonseed, safflower, and soybean oils. The high correlations observed are believed to result from the precision of the flavor threshold testing procedure. Therefore, when suitable flavor testing methods are employed, the chemical methods become more valuable in assessing the quality of an oil. Milk is recommended as the testing media because of its desirable properties.

Introduction

IN THE DEVELOPMENT, evaluation and quality control of edible oils, stability of the oils to oxidative deterioration is of primary importance. Sensory evaluation of the oils has long been the most sensitive method of assessing quality but it is well recognized that it generally lacks precision and reproducibility. Hence, many chemical methods have been developed to measure oxidative deterioration with the objective of correlating the data with flavor deterioration. Varied experiences have been reported in this regard and most researchers are still seeking the ideal test. While additional chemical methods certainly merit attention, we have observed that considerable improvement in research results is obtainable by applying a more precise flavor evaluation technique and by recognizing the reciprocal relationship of chemical data to the quantitative sensory evaluation data. This relationship was originally observed by Lillard and Day (3) in studies on milk fat. The purpose of this investigation was to ascertain if a similar relationship existed with vegetable oils.

Experimental Procedures

Cottonseed, soybean, safflower, and corn oil, containing no added antioxidants, were dispensed into 2 oz open vials and exposed to 100 ft-c of light at 30C for 16 weeks. Analyses were conducted at 2-week intervals on 2 oz of each oil.

Peroxide values were determined by the AOCS method (1) and expressed as milligram of peroxide per kilogram of oil. The 2-thiobarbituric acid number (TBA), was measured by the procedure of Sinnhuber and Yu (4), and the free carbonyls were measured as described by Keith and Day (2).

Flavor evaluations were carried out with a panel of 12 trained judges. The panel was selected from a group of 22 people by presenting milk, containing an oil with an oxidized flavor, at the approximate threshhold of detection, in a triangular test. Twelve panel members were selected after completion of ten triangular tests. The selected panel members were trained by using the aforementioned oils, to familiarize them with oxidative rancidity in the oils.

Samples were prepared for the flavor test in the following manner. The oil to be evaluated was emulsified in pasteurized skim milk to a level of 4% by means of a Waring blendor. The skim milk-oil sample was then mixed in different proportions with fresh pasteurized-homogenized milk containing 4% fat. The samples prepared in this manner contained a total of 4% lipid but different concn of the vegetable oil, depending on the amt required to give a perceptible oxidized flavor. A series of dilutions were thus prepared to cover the range of two above and two below

Name_____

Date_____

Can You Detect an Oxidized Flavor?

Sample No.					
Yes					
No					
Other Flavor (Comments)					

FIG. 1.

[1] Technical Paper No. 1833, Oregon Agricultural Experiment Station, Corvallis.

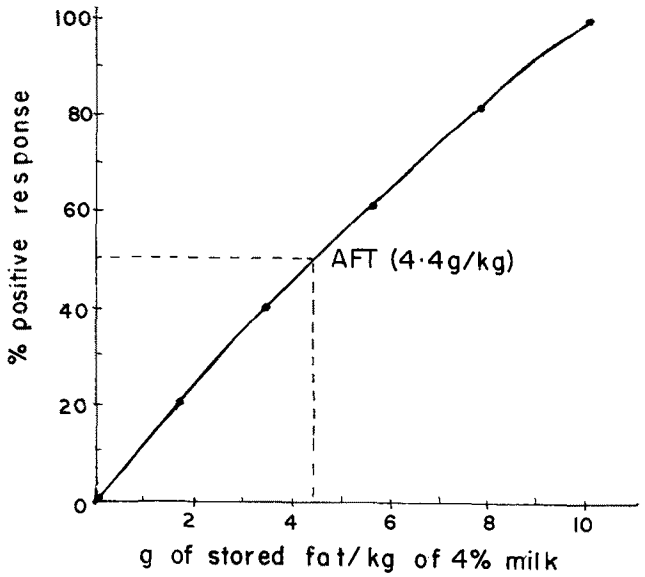

FIG. 2. Determination of the average flavor threshold.

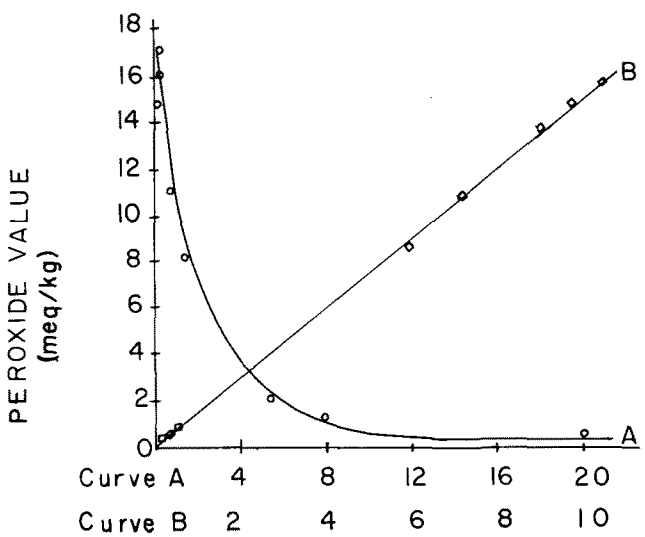

FIG. 3. Relationship between the AFT and peroxide value for oxidative deterioration (curve A is peroxide value × AFT and curve B is peroxide value × 1/AFT).

the average flavor threshold of the panel. A sample containing a mixture of 1:1 of 4% fresh vegetable oil in skim milk and fresh homogenized milk was used as a reference. Fresh homogenized whole milk was not suitable as a reference because each oil, in the fresh state, imparted its characteristic flavor to the milk. Sufficient quantities of the fresh oils were retained frozen in sealed vials to serve for preparation of reference samples. The reference oil samples were checked during storage by the chemical tests listed above and showed no signs of oxidative deterioration.

The samples were served at 21C in 6 oz glasses to the judges seated in individual testing booths. Each booth contained a sink with water available and a yellow-orange light to mask color differences between samples. For a specific test, a total of six glasses were served on a tray; these contained the reference, two sample dilutions above the approximate flavor threshold of the panel, one dilution at the threshold and two below. Each test was replicated twice, once in the morning and once in the afternoon of the same day. This gave a total of 24 responses for each dilution of a test. A sample ballot is shown in Figure 1. Note that the only decision required of the judge was "yes" or "no" relative to the presence of an oxidative rancidity type flavor. In the analysis of data, the 50% level of positive responses was taken as the average flavor threshold (AFT) of the panel. The AFT was expressed as grams of oxidized oil per kilogram of 4% milk. This is illustrated in Figure 2. As shown in the figure, the AFT was 4.4 g of vegetable oil per kilogram of milk. This means that 1 kg of milk containing 4% lipid would have 35.6 g of milk fat and 4.4 g of the vegetable oil. At this concn of oil, the panel could detect the oxidized flavor 50% of the time.

Results and Discussion

Analysis of the data for each oil during the 16-week storage period revealed an exponential relationship between the chemical tests and the average flavor threshold. The relationship is shown by Curve A of Figure 3 which is a hyperbola. Transformation of the data represented by Curve A, by taking the reciprocal of AFT (1/AFT), gives a straight line relationship when plotted against the peroxide values (PV). This is shown by Curve B. The same exponential relationship also was observed for the TBA No. and free carbonyl values. A comparable relationship was observed by Lillard and Day (3) during the study of oxidizing milk fats. The relationship represented by Curve B of Figure 2 was used for the statistical analysis.

The correlation coefficients for 1/AFT vs. the chemical tests during the storage of corn, cottonseed, safflower and soybean oil are given in Table I. The high correlations observed are believed to be due to the precision of the flavor threshold procedure. Precision is derived from simplifying the judge's decision. The judge is not concerned with remembering relative intensities of flavor from one judging session to the next. He merely responds to whether he can detect rancidity in the sample presented. Lack of correlation between the 1/AFT and the free carbonyls in soybean oil probably results from the presence of flavor compounds that are not detectable with the chemical tests employed. Compounds such as alcohols and ketones could be important in the reversion flavor and these would not be detected by the carbonyl procedure. Also the transformation of positional isomers in forming 2,4-dinitrophenylhydrazones, could be a contributing factor.

These data show that when suitable flavor testing methods are employed, the chemical methods become more valuable in assessing the quality of an oil. The flavor threshold method is relatively simple to apply. The use of milk or some other bland flavored diluent is desirable in that it improves the sensitivity of the judges and delays fatigue such as encountered when tasting oils. Milk is a desirable medium in that suitable emulsions can be made with the oils and the osmotic pressure of milk prevents it from introducing

TABLE I

Correlation Coefficients[a] Between 1/AFT and Chemical Tests with Corn, Cottonseed, Safflower and Soybean Oils Stored 16 Weeks at 30C.

Chemical test	Corn	Cottonseed	Safflower	Soybean
Peroxide value	0.970[b]	0.868[b]	0.963[b]	0.891[b]
TBA NO.	0.784[b]	0.771[b]	0.965[b]	0.986[b]
Free carbonyls	0.945[b]	0.622[c]	0.755[b]	0.530

[a] 9 degrees freedom.
[b] Significant at 1%.
[c] Significant at 5%.

various tactual factors that may be encountered with other media.

In the initial stages of this study, the vegetable oils were stored in screw-cap vials. The screw caps varied in their ability to maintain a complete seal and as a result, considerable variation was noted in the extent of oxidation from one vial to the next within a sampling period and between periods. Leaky seals might be responsible for the variation. When the oils were dispensed into pyrex vials, the vials degassed at 1 μ Hg and sealed at this pressure, stored samples showed no signs of oxidation over a 16-week period.

ACKNOWLEDGMENTS

Cottonseed and soybean oil from Durkee Famous Foods; safflower oil from Pacific Vegetable Oil Corporation; and corn oil from Corn Products Company. Mrs. Lois A. Sather and the flavorium staff conducted the flavor tests.

This investigation was supported in part by Public Health Service Research Grant No. EF-182 from the National Institutes of Health, Division of Environmental Engineering and Food Protection.

REFERENCES

1. AOCS, Official and Tentative Methods of Analysis, Tentative Method Cd 8-53, Chicago, Ill. (1958).
2. Keith, R. W., and E. A. Day, JAOCS *40*, 121–124(1963).
3. Lillard, D. A., and E. A. Day, J. Dairy Sci. *44*, 623–632(1961).
4. Sinnhuber, R. O., and T. C. Yu, Food Technol. *12*, 9–12(1958).

[Received August 28, 1964—Accepted March 8, 1965]

Flavor Evaluation of Natural Soybean Oils of High and Low Linolenate Content

C. D. EVANS, HELEN A. MOSER, D. G. McCONNELL and J. C. COWAN, Northern Regional Research Laboratory,[1] Peoria, Illinois; J. L. CARTTER and F. I. COLLINS, U.S. Regional Soybean Laboratory,[2] Urbana, Illinois

Abstract

Three varieties of soybeans, Crest, Grant, and Hawkeye, were processed in the laboratory to obtain edible oils containing 10.4, 9.4, and 5.2% linolenate, respectively. Taste panel evaluations were significantly in favor of low-linolenate soybean oils. Both high- and low-linolenate oils gave the typical off-flavors of aged soybean oil. Flavor results indicate that the linolenate content of soybean oil will probably have to be reduced below 5% to achieve a significant quality improvement in commercially processed oils. Soybean oils of excellent quality can be prepared by laboratory processing procedures.

Introduction

THE THEORY THAT LINOLENATE esters are a primary source of off-flavors in soybean oil is widely accepted by the edible oil industry. Elimination of linolenate is a basic and underlying processing trend in the development of improved edible soybean and rapeseed oils in both Europe and America. Reduction in linolenate content of the oil through genetic studies has not been particularly successful for soybeans (16, 18) although it has been accomplished in rapeseed where reductions of over 50% have been reported (3, 14). For soybean oil, practical results on lowering linolenate content have been obtained only by hydrogenation with reduction of total unsaturation. Almost complete removal of linolenate is obtained in shortening products, but in specially prepared liquid cooking and salad oils, 2 to 3% linolenate is retained after partial hydrogenation and winterization (4,11). Processing for removal of linolenate through polymerization, selective extraction, or selective hydrogenation has not been commercially successful.

Comparison of the flavor and oxidative stability of crambe, mustard, rape, and soybean, all linolenate-containing oils, has been reported by Moser et al. (13). Flavor characteristics observed for these four oils agree with the observations of Holm and co-workers (8,9) on the stability of soybean and rapeseed oils. This study was undertaken to determine if oil obtained from beans selected for their linolenic acid content would show a difference in kind or quality of flavor.

Materials and Methods

Three samples of soybeans were furnished by the U.S. Regional Soybean Laboratory at Urbana, Illinois, to members of the Soybean Processors Association, National Soybean Research Council, who were to cooperate in this study. These varieties were identified as Hawkeye, grown near Urbana, Illinois; Crest, near Fosston, Minnesota; and Grant, near Sabin, Minnesota.

The samples of beans, 14 lb each, were cleaned by hand, and weed seeds, dirt balls, and damaged beans removed. The cleaned beans were flaked in pilot-plant milling equipment. Processing consisted of cracking, screening, dehulling by aspiration, heat and moisture conditioning the grits, and flaking. The flakes were immediately placed in a 5-gal Pyrex carboy and extracted with redistilled petroleum ether. The flakes were completely covered with solvent and allowed to soak approximately 2 hr without agitation or stirring at room temperature. The solvent was removed by filtration through several folds of fine-mesh cheesecloth tied over the end of the carboy. Three such extractions were made plus a final washing of the flakes by hand shaking the entire carboy. The miscella was filtered through paper to remove fines and concentrated in rotating evaporators. Final concentration was accomplished by means of evaporation under diminished pressure, obtained from a mechanical vacuum pump while the oil was heated in a water bath. Oil from the Crest variety foamed badly and some was lost in the final stripping.

TABLE I

Processing Data

Variety	Condition	Moisture content, %	Oil yield, %	Oil content,[a] %
Hawkeye	Some bean damage	8.63	21.4	23.3
Crest	Some green, damaged, and broken beans	9.95	18.8	21.1
Grant	Beans rather small; cleanest of 3 lots, but contained some weed seeds	10.15	19.9	20.7

[a] Standard petroleum extraction—AOCS Ac 3-44.

[1] A laboratory of the No. Utiliz. Res. and Dev. Div., ARS, USDA.
[2] A laboratory of the Crops Res. Div., ARS, USDA.