Dilatometer Measurement Method as a Useful Tool in Fat Study

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Abstract

A dilatometer, previously described by Salway, has been employed in the study of fats and their composition. Reproducibility of data in measuring fat volume change with temperature has been demonstrated. Fats and fat mixtures of widely different composition have been studied. The results show that fat differences as reflected in component fatty acid differences can be detected by use of a dilatometer.

THE dilatometer method has been used previously in many types of research. Its application to the fats and oils field has been known previously. Normann[1] reported upon variations in volume changes with temperature change for a number of fats. Ravich[2] made use of a capillary method to determine the viscosity of fatty acids and various oils, including the progressive hydrogenation of a vegetable oil. This author mentioned that further study of factors such as viscosity, dilatation, iodine number, etc. might be useful in following the course of hydrogenation reactions. Vold[3] and co-workers reported on the use of a dilatometer in demonstrating new forms of anhydrous sodium palmitate. Recently, Willsmer[4] reported on the application of the dilatometer method of analysis in the manufacture of cosmetics.

A confectionery textbook by Jensen describes a dilatrometric method of test which was devised by Salway. The apparatus was recommended for use in the detection of cocoa butter adulteration. The structural simplicity of the apparatus obviously enhances its usefulness in research and routine control work. Therefore an investigation was undertaken in our laboratories to determine whether or not this apparatus could be conveniently used as a guide in manufacturing operations in producing various shortenings. Further, the study was extended to include component fatty acid data for some of the fats and fat mixtures studied. Thus this study sought to determine if fat differences, as reflected in component fatty acid differences, could be correlated with the differences in volume expansion experienced by the corresponding glycerides for a definite temperature range. The previously cited references have not included component fatty acid data for the fats studied.

TABLE No. 1

REPRODUCIBILITY OF RESULTS: VOLUME CHANGES OF HYDROGENATED SHORTENING — HEATING AND COOLING OF FAT

Temperature	Cooling of Fat Dilatometer Reading	Heating of Fat Dilatometer Reading
°C	(CM)	(CM)
65.0	21.85	21.65
60.0		19.60
59.0	19.35	
55.0		17.70
49.0		15.50
46.0		14.50
43.0		13.45
39.0	12.15	
35.0	9.95	9.90
29.0		7.35
24.0		5.75
6.5	—	—

APPARATUS AND METHOD

The apparatus consists of a 25 ml capacity glass cylinder, to one opening is fused a 50cm calibrated capillary whose bore capacity is 280 mm/cc water. A metric scale is attached to the capillary arm of the apparatus. At the other opening a glass stop-cock is fused.

Fats to be tested have been previously heated to 150° C to dispel air and then cooled in a vacuum dessicator. The water used has been recently boiled to dispel air.

TABLE No. 2

DILATOMETER DATA FOR VARIOUS FATS AND FAT MIXTURES

FAT	60-20°	20-8°	60-8°	65-25°	25-6°	65-6°	Expansion in cu. mm. 100 gms. Fat (Total Temperature Change)
1. Cottonseed Oil (C/S)	12.27 cm	2.08 cm	14.35 cm				2,110
2. Almond Oil	12.39	2.09	14.48				2,200
3. 16% Oleo Stearin-84% C/S Oil	14.02	2.23	16.25				3,460
4. 16% Veg. Stearin*-84% C/S Oil	14.25	2.46	16.71				3,800
5. 25% Veg. Stearin*-75% C/S Oil	15.45	1.80	17.25				4,180
6. (92% C/S Oil-8% — Coconut Oil)*				15.80	4.10	19.95	4,540
7. (92% (C/S Oil-8% — Coconut Oil)*				15.20	5.45	20.65	5,070
8. (100% C/S Oil)*	15.25	3.65	18.90				5,360
9. (100% C/S Oil)*				15.23	6.00	21.23	5,860
10. 75% Veg. Stearin*-25% C/S Oil	20.06	1.80	21.86				7,480
11. Tristearin (Mutton Tallow)	21.50	1.40	22.90				8,220
12. Vegetable Stearin*	21.36	1.82	23.18				8,440

*PH — Partially Hydrogenated.

Approximately 10 ml. 60°C water is placed in the apparatus. Then a definite weight of fat, such as 5 gms is added. This is conveniently accomplished by pipetting 60°C fat from a pipette calibrated for this purpose. A quantity of 60°C water is finally added which is sufficient to fill the cylinder and permit the water to rise about 30 cm in the capillary arm when the system has come to equilibrium in a 60°C bath. Entrapping of air while filling the apparatus is to be avoided.

The system is chilled to a temperature at which the fat is solid,5-10°C. Notation is made of this capillary reading and the volume changes are noted at various elevated temperatures, preferably 20°C and 60°C.

Each point of the data curves represents conditions in which the system under test has reached thermal equilibrium for the indicated temperature. The dilatometer data represent volume changes at each temperature as measured from a reference dilatometer reading.

TABLE No. 3

COMPONENT ACID DATA OF FATS AND FAT MIXTURES STUDIED**

	FAT	Saturated	Iso-Oleic	Oleic	Linoleic
1.	Cottonseed Oil (C/S)	24.1	0.3	30.9	44.7
2.	Almond Oil	1.7	0.7	86.9	10.7
4.	16% Veg. St.*-84% C/S Oil	29.6	1.2	31.6	37.6***
5.	25% Veg. St.*-75% C/S Oil	32.7	1.6	32.1	33.6***
10.	75% Veg. St.*-25% C/S Oil	50.0	4.4	34.4	11.2***
11.	Tristearin (Mutton Tallow)	98.2	1.2	0.6	—
12.	Vegetable Stearin*	58.6	5.8	35.6	—

*Partially Hydrogenated

**Calculated to 0.1%

***Calculated from C/S Oil — Vegetable Stearin data, Fats 4, 5 and 10 being mixtures prepared from 1 and 12.

RESULTS AND DISCUSSION

The data which are given in Table No. 1 and illustrated in Figure No. 1 demonstrate that dilatometer readings are reproducible within ± 0.1 cm for a hydrogenated fat. This degree of accuracy was considered to warrant further investigation of the method. The curve definitely indicates a transition point at about 40°C. This temperature approximates the Wiley melting point of the fat. Thus changes in physical state are shown to be detectable by use of this apparatus; this was to be expected. The time required for obtaining data for a fat under test was considered excessive for use of this type of apparatus for plant manufacturing control. The present day operators have other laboratory facilities which fulfill this need more quickly. However, the apparatus offers an excellent possibility for use in research study of fats and fat mixtures.

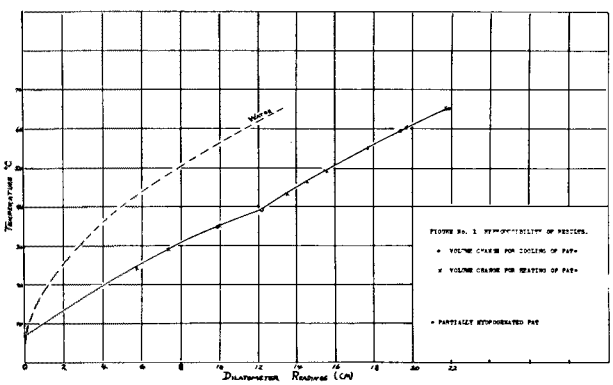
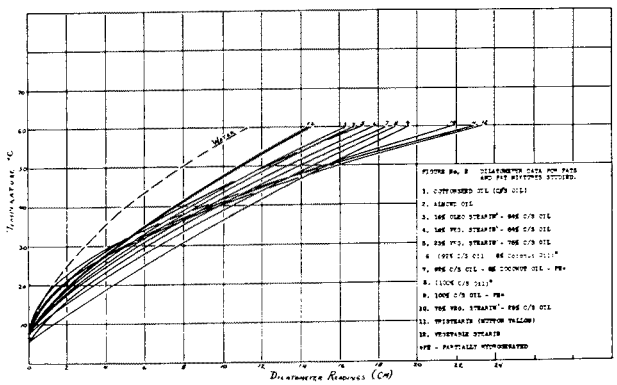


Table No. 2 shows the data obtained for the various fat-water systems studied; these are illustrated in Figure No. 2. The dilatometer readings at reference temperature, 20°C and 60°C were plotted for each fat studied; smooth curves were drawn between the points.

Obviously, the more saturated fats experienced greater volume changes with temperature than did the predominantly unsaturated fats, this is demonstrated in the data tables and figures. The fats studied are listed in Table No. 2 according to their increasing volume changes of fat-water systems with temperature.

In Table No. 3 the component fatty acid data are given for some of these fats. Each fat or fat mixture follows in logical sequence as to its volume change



with temperature with respect to the apparent summation of component fatty acid effects. In fats, natural or partially hydrogenated, the fatty acids are heterogeneously attached to the glycerol base; therefore, a calculation of the summation of expansion effects for each fatty acid type of a fat molecule would be most difficult to compute. However, it is possible to generalize and in so doing list the fatty acid types as to their volume change with temperature; they are first, saturated; second, "iso-oleic;" third, oleic and least in effect is lineoleic acid.

The study is considered to justify utilization of the method of test as a means of differentiating fats and fat mixture types. That is, predominately unsaturated fats can be distinguished from predominately saturated fats such as partially hydrogenated fats. Further, both types can be distinguished from compound type shortenings.

SUMMARY

A previously described dilatometer has been used in fat study. The ease with which reproducibility of results can be obtained has been demonstrated. The data show that differences in fat composition can be detected by this method.

1. Norman, Chem. Umschau Fette, Oele, Wachse Harze, 38, 17-22 (1931).
2. Ravich, Acta Phyiochim, U.R.S.S., 6, 205-12 (1937).
3. Vold et al, Oil and Soap, 16, 48 (1939).
4. Willsmer, Soap, Perfumery & Cosmetics, 12, 501-3,534 (1939).
5. Jensen, Chemistry Flavoring and Manufacturing of Chocolate Confectionery and Cocoa, pp. 214-221, P. Blakistone's Sons & Co. Philadelphia, Pa. (1931).