Modified Butyrometric Method for Rapid Determination of Fat in Seeds

Rashda Ali and **M. Nasir Khan**
Section of Food Science and Technology, Department of Applied Chemistry, University of Karachi, Pakistan

Butyrometers, the specific tubes for fat determination in dairy products, were used successfully in estimation of fats in commercially important seeds such as soybean, peanut, coconut, pinenut, walnut, sesame, almond and poppy seeds. The method is simple, rapid and economical; the results are reproducible and comparable to the standard methods for fat estimation such as the Soxhlet and solvent extraction.

The Soxhlet method is the oldest but still the most commonly used technique for the estimation of fat in food products, in spite of the disadvantage of the time it involves. Although originally modified, the method of Tavear has been used successfully by Hansen (1); its application in industry is limited, where quick results are required for prompt alterations in unit operation and processing. Other methods used for fat estimation are simple solvent extraction (2), Roese-Gottlieb (3) and modified Bolten methods (4). The modified volumetric methods for fat estimation have been reported to be more accurate than Soxhlet extraction. The fat has also been determined by gravimetric (6) and a multisequential method (7) which gives a significantly lower value of variation coefficient. Instrumentation techniques based on physical properties of fats and oils have been used in fat estimation. Prominent among them are the dielectric oil meter (8), hydrometer (9), IR-reflectometer (10) and optical activity measuring apparatus (11). Microanalytical techniques using sophisticated instruments have been developed recently. These include NMR [(pulsed (12), wide-line (13), transient (14))], GLC (15,16) and NIR-reflection spectroscopy (17).

Gerber's method has been applied (18) for rapid determination of the oil content of groundnut kernels using the milk butyrometer, which results in a deviation of 3% when compared with the Soxhlet procedure. Recently, the fat content of peanut, sunflower, avocado and olives also were determined by using the milk butyrometer (19). We have used the cream butyrometer, which has a scale from 0–70%, instead of the milk butyrometer, which is graduated from 0–10%, and the fat content was successfully estimated in such seeds of industrial importance as soybean, almond, sesame, coconut, poppyseed, peanut and pinenut. The results are compared with standard methods of the Soxhlet and solvent extraction.

EXPERIMENTAL PROCEDURES

Chemicals. All the chemicals were AR grade from BDH Chemicals Ltd., Poole, England. Sulfuric acid of specific gravity 1.7614 was purchased locally. A Funke-Gerber centrifuge, H. Jurgen and Co., Bremen, W. Germany was used at 1100 rpm.

The following stock solutions were prepared: (i) Modified Gerber acid was prepared by dilution of 700 ml sulfuric acid to IL; (ii) Modified Gerber's alcohol was prepared according to the method of Takahiro (20) by mixing isoamyl alcohol and isobutyl alcohol (7:3, v/v).

Sample preparation. The brown skin of peanut and pinenut was gently removed by rubbing and air blowing. The almonds were weighed, soaked in 0.05% KOH for one hr and the loose skin was then easily removed. Each sample of peanut (*Arachis trypogaea*), pinenut (*Pignolia*), walnut (*Juglans regia*), dried coconut (*Cocos mucifera*), poppy seed (*Papavera-ceae*) and almond (*Prunus amygdalus*) was dried using fluidized bed drier (RETSH, TGI, Nr 921/49169) at a temperature of 60°C with an air velocity of 1200 l/min for two hr. The seeds were then ground in an electric blender to a fine powder and properly mixed before weighing.

Modified butyrometric method. The powder (5 g) from each sample was taken separately in a 25-ml beaker previously weighed, and 10 ml of hot Gerber acid was added. The material was mixed thoroughly with a glass rod and transferred to a cream butyrometer. The beaker was washed twice with a total of 6 ml hot water; (washings added); then an isoamyl alcohol and isobutyl alcohol mixture (1 ml) was added, more hot water was added to bring the content to a level five mm below the shoulder of the butyrometer. The mixture was shaken vigorously after the stopper was replaced. The butyrometers were placed in a water bath at 65°C for five min and centrifuged at 1100 rpm for three min. The tubes were replaced in a water bath for two min and the percentage of fat was read directly on the scale.

The Soxhlet solvent extraction method. Each sample five g mixed with 100 g of coarse silica (previously washed with ether) was extracted with petroleum ether. Fat was extracted by the usual procedure.

The Gerber-digestion extraction gravimetric method (GDEG). The ground powder (5 g) of the sample was taken in a 25-ml stoppered tube. The modified Gerber acid (11 ml), hot water (10 ml), isoamyl alcohol and isobutyl alcohol mixture (1 ml) were added as previously described. The mixture was heated in a water bath at 65°C for five min and thoroughly stirred with the help of a test tube shaker. It was centrifuged, and the upper layer of the fat was collected by slightly tilting the tube in a tared plastic syringe. The percent of fat was calculated by the following formula:

$$F = [(W_2 - W_1) - A]\ 20 + 0.18 + C$$

Where F = percent of fat, W_1 = weight of the empty syringe, W_2 = weight of the syringe + oil, A = weight of one ml isoamyl:isobutyl alcohol, 0.8 = average loss of fat on the stopper, and C = correction factor = .66.

RESULTS AND DISCUSSION

The amount of fat of almond, soybean, pinenut, coconut, walnut, sesame and poppy seeds obtained by the modified Gerber method, Soxhlet method and Gerber-digestion extraction gravimetric (GDEG) method are compared in Table 1, which shows 0.05 as the mini-

TABLE 1

Percentage of Fat in Various Seeds

Samples	Modified Gerber method	Soxhlet method	GDEG method	Difference between highest and lowest values
Soybean (*Soja max*)	20	20.19[a]	19.53[b]	0.34
Sesame (*Sesam indicum*)	56[a]	55.37	50.07[b]	0.93
Peanut (*Arachis hypogaea*)	54[a]	53.33	53.02[b]	0.98
Almond (*Prunus amygdalus*)	53.5[a]	53.05	52.23[b]	1.27
Pinenut (*Pignolia*)	44.2[a]	43.95[b]	44.12	0.05
Walnut (*Juglans regia*)	54.0[b]	55.01[a]	54.41	1.01
Coconut (*Cocos mucifera*)	50.0	50.30[a]	54.98[b]	1.22
Poppy seed (*Papaveraceae*)	44.5[a]	44.0	43.5[b]	1.00

[a]Highest value obtained for fat.
[b]Lowest value obtained.

mum difference between the highest and lowest figures obtained, in pinenut, and 1.27 as the maximum variation, in almond. The average deviation calculated for the seven samples tested was only 0.52, indicating the reasonable compatability of methodology. It was interesting to note that our method usually gave the highest value among the three procedures. Of the eight samples tested, five showed the highest value, one gave the lowest fat and two had values in between. The Soxhlet method showed two highest, one lowest and five middle values, while GDEG gave six lowest and two middle values. It may be suggested here that a correction factor of +0.66 obtained by taking the average difference between highest and lowest values should be included in the formula given for calculation. The variation among the percent of fat in all the eight samples using three different methodologies is almost negligible, as shown in Table 1. The rapid butyrometric procedures might easily be adopted for routine estimation of fat in seeds, especially in the industries.

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