Rapid, Inexpensive Microwave Oven Method for Total Solids Determination in Fluid Dairy Products[1]

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ABSTRACT

Total solids determination in foods is one of the most often used tests in quality control. For some fresh fluid dairy products, total solids testing is one of a few analytical tests used prior to packaging and distribution. Traditional gravimetric, hot air drying oven methods take several hours to complete. Microwave methods only take several minutes, but automated microwave drying equipment may be prohibitively expensive. A suitable alternative is commercially available microwave oven and analytical balance, which is one-tenth the cost of the automated equipment.

A 1-g sample is evenly distributed in the bottom of a predried, tared styrofoam cup, 5.5 cm diameter and 5.5 cm tall. The sample is dried for 10 min at 520 W power, cooled in a desiccator, and weighed. Total solids is determined by difference.

At least 100 samples each of the following products were tested in duplicate: sour cream; cottage cheese dressing; and ice cream, ice milk, and sherbet mixes. Total solids results for these products obtained by the microwave method were compared with control results from a standard hot air oven total solids method. Total solids mean values from experimental and control groups were almost exactly the same. No statistically significant difference was found between methods for any of the products.

INTRODUCTION

Unless a testing procedure can be accomplished in 15 to 20 min, it is generally not used during processing of fresh fluid dairy products. Product quality data from traditional total solids tests, shelf-life tests, and microbiological methods are not available soon enough to be useful prior to subsequent product processing and packaging. Determination of milk fat and determination of total solids generally are the only tests that can indicate product quality in a timely fashion. There are several methods of rapid solids testing, but often these methods are complicated and expensive. The present study describes a rapid, simple, inexpensive method for determining total solids in fluid dairy products.

Moisture or water content of foods indicates quantity of water present in a given sample of food, revealing the total solids by difference. Because water tends to be the primary constituent of most foods, measurement of water is essential for quality control purposes. The importance of rapid, accurate, simple methods of moisture determination cannot be underestimated. Water in foods exists in both free and bound forms. Free water has little or no interaction with other food components, exists in the intercellular and interparticle spaces of foods, and keeps its characteristic physical properties, such as the colligative properties (9). Free water is removed during total solids tests.

For the purposes of this paper, bound water is defined as water that remains in an unchanged form when the food is subjected to a particular treatment (5). Bound water does not freeze at low temperatures nor is it available as a solvent. It requires more energy for removal than does free water when the amount of moisture is being determined.

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In the conventional method of moisture removal, the surface of food is heated by convection. Hot oven air contacts the food and is absorbed by a very thin surface layer of the food. The heat is then transferred to the interior of the food by conduction. Because the thermal conductivity of foods is relatively low, it takes a relatively long time for the interior portions of the food to be heated.

When microwave energy, rather than the conventional method of heating, is used, energy is transmitted throughout the food, and heating takes place more rapidly. Microwave energy can flow through many types of substances without being absorbed. Because the primary constituents of fluid dairy products are water and fats, however, dairy foods do absorb microwave energy (3) and convert it into heat energy. One of the problems of some food products is the uneven distribution of moisture and fat within the food and the subsequent uneven heating of the substance. This is not a significant problem in fluid dairy foods due to the homogeneity of the products. Thus, microwave heating can be an effective method for evenly heating fluid dairy products.

The next point to be considered in total solids determination is the mechanism of moisture removal from foods. There are two stages in the moisture removal or drying process: the constant-rate drying period and the falling-rate drying period. Constant-rate drying occurs as long as moisture is moving from the inside of the food to the surface at a rate equal to the rate of surface evaporation due to heating.

The falling-rate drying period is subdivided into two stages (Figure 1). During the first stage, some parts of the food surface are wet and other parts are dry. The second stage begins when the entire surface of the food is dry. The controlling factor during the second stage of the falling rate period of drying is the internal movement of water molecules in the food to the surface. The internal movement of water is not equal to the rate of evaporation.

The principles involved in this mass transfer of water are the same whether heating is accomplished by conventional or microwave energy applications. Drying of food is complete when very little if any moisture is being driven off. At this point, the weight of the food sample remains relatively constant. Even when no moisture is being removed by heating, however, a small amount of water that is chemically bonded to other food components, the bound water, remains. This water is generally not removed until the sample is ashed or burned. Bound water is not significant to total solids determination; however, it may lead to some variability in total solids results for some foods.

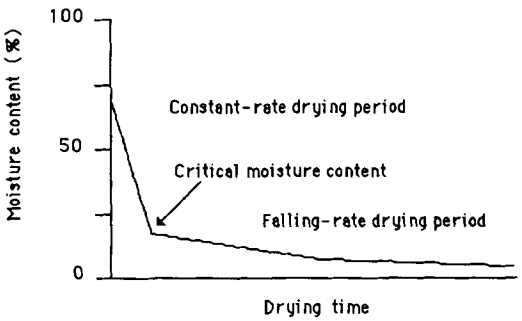

Figure 1. Phases in a food drying process [from (5)].

Both microwave and conventional total solids determination methods are presently being used in the dairy industry. Although traditional gravimetric, hot air drying oven methods take several hours, microwave methods only take several minutes. However, the cost of automated microwave drying equipment may be prohibitive. A suitable alternative to automated microwave drying equipment is a commercially available microwave and a standard analytical balance. Microwave solids testing with this equipment can be accomplished for one-tenth the cost of more highly automated systems. The Mojonnier test for solids has also been widely used in the dairy industry. However, it also takes longer to run than the microwave method and requires specialized equipment.

Lee and Latham (7) reported a rapid moisture determination method using a commercial microwave oven. This research was conducted on pet food. The investigators concluded that conventional and microwave moisture determination methods produced the same results.

Kipp and Spurgeon (6) described a method for accurately determining moisture in Swiss and cottage cheese. Results of microwave tests on these two types of cheese compared favorably with Mojonnier and AOAC tests. However, results from microwave testing on Cheddar, Mozzarella and cream cheese differed signifi-

cantly from the Mojonnier test results. The investigators stated that microwave test results were higher than Mojonnier test results but were lower than AOAC test results. Their conclusion was that this microwave method could be satisfactorily used for factory process control, cheese grading, and monitoring by regulatory agencies.

Several authors identified areas of caution in the use of the microwave method. Splattering of sample and subsequent loss of solids due to rapid heating was mentioned as a problem. This was overcome in several studies by placing the sample between two pieces of filter paper. Solids were effectively trapped between the layers of paper. This procedure provided an integral, thin layer of product with a large surface area, permitting better microwave energy absorption by the sample, more uniform increase in sample particle temperature, and better water diffusion through and out of the sample (2, 4, 7).

Sample burning was also cited as a problem. Microwave treatments need to be limited in intensity and duration so that samples are not charred. In addition, hot spots inside microwave ovens cause uneven drying of samples and difficulty in repeating results. Suggestions to minimize this problem included placing the samples in exactly the same spot in the microwave each time a test is run, placing the samples in the center of the oven each time, and placing the sample on a rotating sample table within the oven (4).

The objective of this study was to develop a highly reliable and valid, rapid method for determining total solids in fluid and semifluid dairy products. The method proposed would test for total solids, as do lengthier conventional methods, and would be inexpensive.

MATERIALS AND METHODS

Sample Descriptions

Samples of the following commercially produced dairy products were used in this study: sour cream; cottage cheese dressing; and ice cream, ice milk, and sherbet mixes. These products were made according to good manufacturing practices and, where applicable, followed the standards set forth by the Code of Federal Regulations for product identity and standards.

Samples were obtained directly from sampling valves or from open tops of holding vats and tanks after HTST pasteurization and homogenization of the products in a commercial dairy operation. Products were agitated at least 5 min prior to sampling. Sample sizes were approximately 500 ml each. Sample collection and testing occurred during a 1-yr period.

Once collected, samples were immediately taken to the quality control laboratory. There, each sample was individually blended to ensure homogeneity prior to testing. Blending was accomplished by pouring the sample back and forth between the original sample container and another clean, dry container at least three times prior to testing. During this blending, samples were poured in a manner that minimized air incorporation. Following blending, samples were immediately tested. Procedures for determining total solids were performed by trained technicians.

Experimental Testing Procedure

Styrofoam cups with a capacity of 118 ml were obtained from Fort Howard Paper Corp., Charlotte, NC 28290 (Stock Number FC4M). These cups measured 50-mm deep, 70-mm in diameter (i.d.) at the top, and 50 mm in diameter (i.d.) at the bottom. A Penney's microwave oven (Model number 5912) was used in the testing procedures. This microwave oven had a maximum power output of 650 W, a frequency of 2450 MHz, and power supply of 1.25 kw.

Empty styrofoam cups were numbered and preheated for 5 min at a power setting of 80% (520 W) in the microwave oven. After heating, the cups were immediately removed from the oven with forceps and placed in a desiccator where they remained for at least 1 min to cool. After cooling, empty cups were weighed to .1 mg on a Mettler analytical balance, Model Number AC 100 (Mettler Instrument Corp., Hightstown, NJ). The balance had been previously calibrated and standardized by an equipment manufacturer's technician. The tare of the sample cup was recorded and the balance was zeroed.

A sample of the product was pipetted into the cup and spread evenly over the bottom. Sample weight was 1.00 (± .03) g. This weight was also recorded. Using forceps, the cup was then placed in the microwave oven on the edge of a rotating sample table. The rotating sample table turned at a rate of approximately 2 rpm.

TABLE 1. Drying time of samples in microwave oven at approximately 520 W.

Number of samples dried	Duration of drying
	(min)
1	10
2	11
3	11.5
4	12
5	12.5
6	13

TABLE 2. Means and standard deviations for total solids percentages for selected fluid and semifluid dairy products as determined by microwave and conventional drying methods.

Product	n	Microwave		Conventional	
		$\bar{X}$	SD	$\bar{X}$	SD
Cottage cheese dressing	100	23.96	1.20	23.98	1.23
Lowfat dressing	35	22.04	1.18	21.95	1.21
Sour cream	27	21.74	1.26	21.67	1.28
Ice cream mix	47	38.91	1.05	39.12	1.16
Ice milk mix	32	34.66	2.76	34.67	2.70
Sherbet mix	30	32.95	1.89	33.05	1.92

Samples were dried at 80% power (approximately 520 W) according to the schedule found in Table 1. A maximum of six samples were dried at any one time. After the drying period, samples were immediately removed from the oven using forceps and placed in a desiccator for at least 1 min. After cooling, the cup and sample were again weighed on a Mettler analytical balance to .1 mg. The weight of the cup and dried sample was recorded.

The percentage of total solids was determined using the following formula: % Total solids = [{dish weight + dry sample weight − dish weight}/{(dish weight + wet sample weight) − dish weight}] × 100. Two different quality control laboratories performed this procedure using microwave ovens of the same make and model number. Samples were always run in duplicate, and the mean of the sample duplicate weights was used to obtain total solids content.

Control Testing Procedure

The same samples used for the experimental procedure above were also used for the control procedure. For the control testing procedure, a modification of the AOAC hot air oven method, Method I, No. 16.032 (1) was incorporated.

Aluminum weighing dishes measuring 5 cm in diameter and 1.5 cm in height (VWR number 25433-008) were numbered and predried in a Precision forced hot air oven (GCA Corp., Chicago, IL, Model Number 18) at 100°C for 10 min. After the predrying period, dishes were removed from the oven using forceps and were immediately placed in a desiccator to cool. Dishes were allowed to cool at least 1 min before being tared. The tare weight of each dish was recorded.

A sample of the product was pipetted into the dish and spread evenly over the bottom. Sample weight (2.5 to 3.0 g) was recorded. Dish and sample were then placed in the forced air drying oven for 4 h at 100°C. After the drying period, dish and sample were removed from the oven using forceps and were immediately placed in a desiccator to cool at least 1 min before being weighed. The weight of the dried sample and dish was recorded.

The percentage of total solids was also determined using the same formula as for microwave solids. Samples were run in duplicate, and the mean of the duplicate sample weights was used to obtain total solids content. Control samples from both laboratories were tested using the same hot air oven.

Statistical Methods

Means and standard deviations were calculated for control and experimental sample groups of all products. A *t* test was run between control and experimental groups for each product using Minitab (8).

RESULTS AND DISCUSSION

Means of total solids percentages did not differ significantly between the microwave oven method and the conventional hot air oven method for the following dairy products: sour cream ($P>.86$); cottage cheese dressing ($P>.93$); lowfat cottage cheese dressing ($P>.74$); ice cream ($P>.35$), ice milk ($P>.99$), and sherbet ($P>.83$) mixes (Table 2). Standard deviations for both methods did not differ by more than .11 standard deviation. Total solids

for these products ranged from about 13 to 40%.

The consistency and the agreement shown by both conventional and microwave methods for determining total solids indicates the efficacy of using the rapid, inexpensive microwave method. The probability values for ice cream mix may be low due to bound water. Hydrocolloids used in ice cream mix production chemically bind water, and if this water is driven off at varying rates then this may account for the variation in results. Judging from the wide variety of products tested in this study, almost any fluid or semifluid dairy product can be tested during production without delay of processing or packaging operations. With the addition of a relatively inexpensive commercial microwave oven, almost any food processing plant quality control laboratory can determine total solids in less than 20 min.

Several cautions should be noted in using this method. In preliminary studies not discussed here, technician training and experience were shown to be important variables. Consistent and reliable results between conventional and microwave methods were not obtained by an individual analyst until approximately 50 to 75 tests had been run. Once this training period was accomplished, there appeared to be no variability among analysts who ran the microwave total solids tests on different occasions. This training period consisted of an initial demonstration by a trained analyst and then subsequent individual practice. During this practice, duplicate samples were run by both the conventional and microwave methods. This allowed the trainee to crossreference results obtained from both methods. Prior to running the tests alone, each trainee was given a set of samples of each product that had been previously tested by an experienced technician. Differences between technicians' results did not vary significantly.

The other caution in using this method is that due to the small size of the samples, damage to the microwave oven may occur. It is strongly suggested that the microwave oven be checked regularly by qualified maintenance personnel for microwave radiation leakage. Damage to the microwave oven can cause inaccurate test results and, more important, may injure laboratory personnel.

One aspect not studied in this project was the use of this microwave method on particulate matter such as cheese. Although other studies have shown that microwave methods can be used in determining total solids in nonfluid foods (6, 7), the present method would probably need to be modified before being applied to solid particulate materials.

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