

A RAPID METHOD FOR THE DETERMINATION OF TOTAL SOLIDS IN MILK AND SOME DAIRY PRODUCTS USING MICROWAVE OVEN

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ABSTRACT

The total solids (TS) of milk, yoghurt, pickled Domiati and Ras cheese (20 samples each) were determined using microwave drying as compared to standard air oven method in order to establish the applicability of commercial microwave ovens. Preliminary experiments indicated that the suitable heating time in the microwave oven required for complete drying of milk, Domiati cheese and yoghurt was five minutes while that for Ras cheese was seven minutes. In addition, the location and number of samples in the microwave oven have no effect on the drying process. Moreover, partial drying of the experimented dairy products (cheese and yoghurt) at 105°C/30 min. was applied to prevent splattering and burning of the samples during the microwave drying. The drying time in the microwave oven was found to be 5 min. for milk, yoghurt and Domiati cheese, while it was 7 min. for Ras cheese.

Mean values for TS contents in milk, Domiati cheese, Ras cheese and yoghurt were 14.325 and 14.491%, 43.167 and 43.060%, 71.670 and 71.591% and 15.319 and 15.813% for air oven and microwave methods, respectively. Statistical analysis showed no significant differences between the two methods for any of the studied products. Consequently, microwave method is, therefore, recommended for the determination of TS in milk, yoghurt, Domiati and Ras cheese.

INTRODUCTION

Determination of the Total Solids (TS) content of food and dairy products is one of the most common tests done in quality control laboratories. Product TS determinations are routinely done for reasons of regulatory compliance and maintenance of product quality and product yield. In other instances, such as the determination of solids content of milk, results may be used as a base for payment to the farmer (Green and Park, 1980).

Both conventional and microwave TS determination methods are presently being used in the dairy industry. Although traditional gravimetric hot air drying oven method takes several hours, microwave method only takes several minutes. However, the cost of automated microwave drying equipment may be prohibitively expensive (Dzurec and Baptie, 1989).

In many applications, there is a need for rapid determination of moisture for on-line product quality control (Barbano and DellaValle, 1984). Therefore, the existence of a rapid, accurate and simple method for TS determination is of great importance.

Previous studies demonstrated the application of microwave drying to milk and dairy products (Thomasow, 1985; Dzurec and Bepide, 1989; Dzurec, 1991; Marquez et al., 1993; Marquez, 1995 and Hurskar and Vahcic, 1999). Several authors identified areas of caution in the use of microwave method such as splattering of sample and subsequent loss of solids, sample burning, differences in results from one microwave oven to the next (even when the same analysis conditions were used) and differences in results due

to differences of dielectric properties of sample (Lee and Latham, 1976; Hayward and Kropf, 1980 and Barbano and DellaValle, 1984). However, little attention has been given to this method in Egypt.

Since the suitable alternative for automated microwave drying equipment is the commercially available microwave oven which is inexpensive and commonly used, it was thought worthwhile to try and develop a reliable and valid method for TS determination in milk and some dairy products using commercially available microwave oven.

MATERIALS AND METHODS

1. Microwave oven:

Moulinex electric type 823 was used. The frequency of the radiation emitted in this oven was 60 Hz and when operated at full power it provides 1300 watt.

2. Sample descriptions:

2.1. Milk samples:

Twenty samples of raw milk were purchased from some retail shops at the Giza City. Each sample was well agitated to ensure homogeneity prior to testing.

2.2. Yoghurt:

Twenty commercially available yoghurt samples were purchased from the local market. They included samples from dairy investment companies, some small dairies and the milk processing unit of the Faculty of Agric. Cairo Univ. Each yoghurt package was thoroughly mixed until being homogeneous before testing.

2.3. Domiati and Ras Cheeses:

Twenty different samples of pickled Domiati cheese, as well as twenty different samples of Ras cheese were procured from different supermarkets in the Giza City. Each individual sample was ground in a porcelain mortar and transferred to a suitable glass vessel until analysis.

3. Experimental testing procedure:

Lower portions of petri dishes having 10 cm in diameter and 1.5 cm in height were numbered and preheated at 105°C/5 min. in an air oven and cooled in a desiccator. After cooling empty petri dishes were weighed to four decimal places and approximated to three, and this weight was recorded. A sample of milk (about 5 g.) was pipetted into the the petri dish and spread evenly over the bottom of this dish, then weighed and recorded. With the help of forceps, the petri dish containing milk sample was then placed in the microwave oven on a rotating sample table for different periods to establish the optimum period required for complete drying. After drying, sample was immediately removed from the oven and placed in a desiccator for cooling. After cooling, the petri dishes containing dry matter were weighed and all weights were recorded. All milk samples were always run in duplicate and the percentage of TS was determined.

Regarding determination of TS in Domiati and Ras cheese and yoghurt samples, preliminary trials were carried out to overcome the splattering of the sample particles and subsequently loss of solids as well as partial burning. Consequently, it was worthwhile to try and carryout some partial drying in an ordinary oven at 105°C for different periods as a mean for forming an integral thin layer of the sample before placing in the microwave oven. About 3.0 g of each product sample were weighed and evenly spread over the bottom of the petri dish with the help of flat edge glass rod and stepwise addition of 1.0:1.5 ml of distilled water. Partial drying in the air oven at 105°C was conducted for different periods to determine the most suitable partial drying time. Thereafter the partially dried samples were placed in the microwave oven (following the above mentioned method in drying of milk) and the suitable drying period in the microwave for each of the aforementioned products was studied. Following the suitable drying period, samples were removed from the microwave oven and placed in a desiccator for cooling. The petri dishes containing dry mater were weighed and the TS content of product was calculated.

4. Control testing procedure:

The same samples used for microwave drying method (milk, yoghurt, Domiati and Ras cheese) were used for the control procedure. The official method of the AOAC (1984) No. 16.032 was followed. Aluminum weighing dishes (5 cm in diameter and 1.5 cm in height) were used. samples were run in duplicate and the mean of the duplicate sample weights was used to estimate total solids content.

5. Statistical analysis

The statistical analysis were carried out according to Sendecor and Cochran (1967). An F test was run between the TS results of standard and microwave methods for milk and each product.

RESULTS AND DISCUSSION

1. Preliminary experiments:

Extensive work was done to determine the heating time, number of samples, and sample location inside the microwave oven that gave results that agreed with the official air oven method for each particular dairy product.

With respect to drying of milk using microwave oven a constant weight of milk samples (about 5g) based on the previous work of Barbano and DellaValle (1984) was used. The milk sample was placed in the microwave oven on the center of the rotating table for different periods ranging from 3-10 min. with one minute interval. The results obtained clearly indicated that heating time of five minutes was the most appropriate that give results similar to that of air oven method. The average TS of a milk sample from three trails was 14.61, 14.41, 14.32, 14.12, 13.82, 13.84, 13.80 and 13.82% when the heating time was 3, 4, 5, 6, 7, 8, 9 and 10 minutes respectively. The average TS of the same sample when air oven was used was 14.29% which is insignificantly different from that of the microwave oven for 5 min. (14.32%).

The effect of milk sample position in the microwave oven on the total solids content was also studied. Three samples of the same milk were placed in the microwave oven on the rotating table on a straight line, i.e. one on the center and the others two on the edges of the rotating table, then heated for 5 min. The results obtained showed no significant difference since the average of three replicates was 14.13, 14.12 and 14.04% when located on the center and edges of the rotating table in the microwave oven. The average TS of the same milk sample when dried in air oven at 105°C/3h. was 14.11%. Consequently, only 5 samples could be placed in the microwave oven in each run.

Regarding yoghurt, Domiati and Ras cheese, 3g of each product was weighed and placed in an air oven at 105°C for partial drying as formerly mentioned under "Experimental Procedure". The partial drying time required to form an integral thin layer of any of these products was 30 min at 105°C. This formation of an integral thin layer of the dairy products via even spreading and partial drying at 105°C/30 min. permitted better microwave energy absorption by the sample, uniform increase in sample temperature and better water diffusion through and out of the sample. Consequently splattering of the sample was prevented and a uniform drying was obtained.

The drying time of each product in the microwave oven for 5, 6, 7 and 8 min. showed that the optimum was 5 min. in case of yoghurt and Domiati cheese samples and 7 min. in case of Ras cheese samples. Deep browning of yoghurt and Domiati cheese particles was noticed if heating was extended. The average TS content of Ras cheese sample was 66.93, 66.70, 66.13 and 66.24% when heated for 5, 6, 7 and 8 min. in the microwave oven respectively. The average TS content of the same sample was 65.88% when it was dried at 105°C/3h. which corresponds to that of drying for 7 min. in the microwave oven. It was observed that heating more than 7 min. led to some browning.

2. Total solids determination
a. Raw Milk

Twenty different samples of commercially available raw milk were analyzed for TS content using the microwave and air oven. The results obtained were recorded in Table 1. It is clear that TS contents of different milk samples determined using the microwave oven were close to that determined using the conventional air oven. Total solids in raw milk samples analyzed ranged from 12.514 to 17.357% in case of conventional air oven drying method, while it was 12.859 to 17.719% in case of microwave drying method. The average of TS in the 20 samples of milk was 14.325% and 14.491% for the above mentioned two methods in order. These results are in agreement with those stated by Green and Park (1980) and Akuzawa et al., (1984) who found similarity in the average of TS in 20 and 12 samples of cows milk when determined by hot air or microwave oven drying methods.

Table (1): Total solids in raw milk as determined by hot air and microwave oven*.

Sample	Air oven	Microwave oven	Sample	Air oven	Microwave oven
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No.	moisture	T.S.	moisture	T.S.	No.	Moisture	T.S.	Moisture	T.S.
1	85.464	14.536	85.686	14.314	11	87.251	12.749	87.032	12.968
2	85.465	14.535	85.591	14.409	12	82.643	17.357	82.281	17.719
3	85.718	14.282	85.880	14.120	13	85.349	14.651	85.265	14.735
4	86.028	13.972	85.181	14.819	14	83.553	16.447	83.256	16.744
5	86.068	13.932	86.178	13.822	15	83.590	16.410	83.166	16.834
6	86.052	13.948	86.053	13.847	16	87.214	12.786	86.875	13.125
7	85.876	14.124	85.817	14.183	17	85.934	14.066	85.568	14.432
8	85.835	14.165	85.834	14.166	18	87.486	12.514	87.141	12.859
9	85.974	14.026	85.742	14.258	19	86.921	13.079	86.907	13.093
10	85.886	14.114	85.599	14.401	20	85.182	14.818	85.033	14.967

* Mean value = 14.325 and 14.491% for hot air and microwave oven, resp.

Statistical analysis were carried out to evaluate the variance between the two treatments and to find out the significance of these differences. It is obvious from the F test figures in Table 2 that there is no significant differences between the TS content of milk either at P₀₅ or P₀₁ levels when determined by microwave or air ovens. The insignificance of the differences between the TS content of milk when determined by either air or microwave oven method was confirmed by Akuzawa et al., (1984), Dzurec (1991) and Hruskar and Vahcic (1999).

Table (2): Analysis of variance of TS in raw milk*

Source of variance	d.f.	SS	MS	F-value
Between treatments SS	1	0.3601	0.3601	0.4661
Within treatments SS	78	60.2592	0.7726	
Total SS	79	60.6193	0.7673	

* Tabulated F value is 6.95 and 3.956 at P₀₁ and P₀₅ levels, resp.

b. Total solids in cheese and yoghurt

Data in Table 3 show the TS content of pickled Domiati cheese (20 samples) either determined by microwave or air oven drying method. It is clear from these results that the average of TS in pickled Domiati cheese was 43.1669 and 43.0598% for hot air and microwave oven drying methods, respectively which almost exactly similar. However the TS content in cheese samples ranged from 36.8090 to 50.7343% for microwave drying method or 36.4692 to 49.6574% for hot air drying method.

Analysis of variance presented in Table 4 exhibited no significant differences between the two methods used for TS determination in pickled Domiati cheese, since the estimated F-value was lower than that found in the F-table at the level of P₀₅ or P₀₁. These results are in line with the results obtained by Marquez et al., (1995) who worked on soft cheese (Brie-type) and Hruskar and Vahcic (1999) who worked on processed cheese.

Table (3): Total solids in pickled Domiati cheese as determined by hot air and microwave oven*.

Sample No.	Air oven		Microwave oven		Sample No.	Air oven		Microwave oven	
	moisture	T.S.	moisture	T.S.		Moisture	T.S.	Moisture	T.S.

1	57.547	42.453	58.229	41.771	11	65.022	43.978	56.742	43.258
2	50.343	49.657	49.266	50.734	12	57.599	42.401	58.034	41.966
3	54.728	45.272	57.587	42.413	13	55.642	44.358	55.125	44.875
4	55.150	44.850	54.057	45.943	14	63.087	36.913	62.632	37.368
5	55.509	44.491	55.255	44.745	15	60.753	39.247	61.177	38.823
6	53.739	46.261	53.110	46.890	16	57.730	42.270	58.267	41.733
7	58.877	41.123	58.436	41.564	17	61.184	38.816	60.809	39.191
8	55.979	44.021	56.673	43.327	18	57.150	42.850	57.775	42.225
9	53.929	46.071	54.934	45.066	19	54.488	45.512	54.247	45.753
10	63.531	36.469	63.191	36.809	20	53.675	46.325	53.079	46.921

* Mean value = 43.167 and 43.060% for hot air and microwave oven, resp.

Table (4): Analysis of variance of TS in Domiati cheese*

Source of variance	d.f.	SS	MS	F-value
Between treatments SS	1	8.2511	8.2511	1.4982
Within treatments SS	78	429.5730	5.5073	
Total SS	79	437.8241	5.5421	

* Tabulated F value is 6.95 and 3.956 at P₀₁ and P₀₅ levels, resp.

Table 5 shows the TS content of Ras cheese (20 samples) either determined by the conventional air oven drying method or the microwave oven. It is obvious that the total solids of Ras cheese ranged from 64.8673 to 79.2163% with an average of 71.6692% for conventional method, while it was 65.9943 to 79.3005% with an average of 71.591% for the microwave oven method. These figures clearly show the exact similarity of the two methods when used for TS determination in Ras cheese.

Table (5): Total solids of Ras cheese as determined by hot air and microwave oven *

Sample No.	Air oven		Microwave oven		Sample No.	Air oven		Microwave oven	
	moisture	T.S.	Moisture	T.S.		Moisture	T.S.	Moisture	T.S.
1	35.133	64.867	33.867	66.133	11	30.844	69.156	30.482	69.518
2	31.255	68.745	32.509	67.491	12	27.320	72.680	27.644	72.356
3	30.670	69.330	32.919	67.081	13	24.117	75.883	23.712	76.228
4	26.427	73.573	25.714	74.286	14	31.445	68.555	30.992	69.008
5	33.353	66.647	34.712	65.228	15	34.123	65.877	34.005	65.995
6	28.303	71.697	27.930	72.070	16	26.310	73.690	26.800	73.200
7	20.784	79.216	20.978	79.022	17	28.706	71.294	28.336	71.664
8	29.448	70.552	29.295	70.705	18	30.289	69.711	30.491	69.509
9	24.098	75.902	24.069	75.931	19	20.870	79.130	20.699	79.301
10	30.628	69.372	30.636	69.364	20	22.488	77.512	22.259	77.741

*Mean value = 71.669 and 71.591% for air and microwave oven, resp.

Statistical analysis as shown in Table 6 illustrate that there is no significant differences between the two methods either at the P₀₅ or P₀₁ levels.

Table (6): Analysis of variance of TS in Ras cheese*

Source of variance	d.f.	SS	MS	F-value
Between treatments SS	1	0.0365	0.0365	0.0041
Within treatments SS	78	699.9595	8.9738	
Total SS	79	699.9960	8.8607	

* Tabulated F value is 6.95 and 3.956 at P₀₁ and P₀₅ levels, resp.

With respect to TS content in yoghurt, 20 samples were analyzed using the microwave and conventional methods. The obtained results (Table 7) indicated that the average of TS content in yoghurt was 15.319% for the conventional method and 15.813% for microwave method. The range of TS in yoghurt commercially available was 13.105 to 19.460% for conventional and 12.986 to 20.470% for microwave methods respectively. This clearly illustrate the similarity of both methods. The statistical analysis data presented in Table 8 confirmed this finding, since there is no significant difference between the two methods as shown from the F-value at the P₀₅ or P₀₁ level.

Table (7): Total solids of yoghurt as determined by hot air and microwave oven *.

Sample No.	Air oven		Microwave oven		Sample No.	Air oven		Microwave oven	
	Moisture	T.S.	Moisture	T.S.		Moisture	T.S.	Moisture	T.S.
1	80.540	19.460	79.534	20.466	11	80.567	19.433	80.173	19.827
2	85.062	14.938	84.417	15.583	12	84.525	15.475	83.887	16.113
3	84.411	15.589	83.436	16.564	13	84.290	15.710	83.629	16.371
4	85.418	14.582	84.486	15.514	14	85.902	14.098	85.081	14.919
5	86.895	13.105	85.494	14.506	15	86.434	13.566	86.188	13.812
6	80.594	19.406	80.808	19.192	16	85.908	14.092	85.363	14.637
7	83.989	16.011	83.356	16.644	17	86.002	13.998	85.691	14.309
8	84.143	15.857	83.558	16.442	18	86.300	13.700	85.388	14.612
9	85.576	14.424	85.542	14.458	19	85.130	14.387	85.540	14.460
10	85.639	14.361	87.014	12.986	20	85.812	14.188	85.163	14.837

*Mean value = 15.319 and 15.813% for air and microwave oven, resp.

Table (8): Analysis of variance of TS in yoghurt*

Source of variance	d.f.	SS	MS	F-value
Between treatments SS	1	2.4365	2.4365	1.2853
Within treatments SS	78	147.2573	1.8879	
Total SS	79	149.6938	1.8949	

• Tabulated F value is 6.95 and 3.956 at P₀₁ and P₀₅ levels, resp.

These results are in accordance with those reported by Hruskar and Vahcic (1999) and Dzurec (1991) who found insignificant differences between these two methods when TS content of yoghurt was determined and is in accordance with Marquez et al., (1995) who stated that precision of TS determination by microwave oven was not satisfactory for the yoghurts (plain and flavoured).

In conclusion, it is suggested that the microwave method could be used as a rapid and accurate method for total solids determination in milk, yoghurt, Domiati and Ras cheese.

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طريقة سريعة لتقدير الجوامد الكلية في اللبن وبعض منتجاته باستخدام أفران الميكروويف

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تم تقدير النسبة المئوية للجوامد الكلية في اللبن، الزبادي، الجبن الدماطي المخزن، الجبن الرأس (عشرون عينة من كل) باستخدام أفران الميكروويف مقارنة بالطريقة القياسية ($105^{\circ}\text{م}/3$ ساعات) لتقييم مدى إمكانية استخدام أفران الميكروويف المنزلية في تقدير % للجوامد الكلية. وفي هذه الدراسة تم عمل تجارب مبدئية لتحديد أنسب وقت للتجفيف داخل الفرن وكذلك أفضل وضع للعينات داخل الفرن، بالإضافة إلى تحديد عدد العينات داخل الفرن.

كما تم عمل تجفيف جزئي لعينات الزبادي، الجبن الدماطي والرأس في الفرن العادي على $105^{\circ}\text{م}/30$ دقيقة كتعديل للتغلب على تطاير أجزاء من العينة أو احتراق أجزاء منها.

وقد وجد أن زمن التجفيف داخل فرن الميكروويف 5 دقائق لكل من اللبن، الزبادي، الجبن الدماطي كان 7 دقائق في حالة جبن الرأس

وقد أوضحت النتائج المتحصل عليها أن متوسط % للجوامد الكلية باستخدام الطريقة القياسية وطريقة الميكروويف للين هو 14.325، 14.491 % وللجين الدماطي 43.167، 43.060 % وللجين الرأس 71.670، 71.591 % وللزبادي 15.319، 15.813 % على التوالي مما يوضح تطابق النتائج في الطريقتين.

كما أوضحت النتائج المتحصل عليها عدم وجود فروق معنوية بين النسبة المئوية للجوامد الكلية في كل من اللبن الخام، الجبن الدماطي، الجبن الرأس، الزبادي سواء تم تقديرها بواسطة الطريقة القياسية أو باستخدام أفران الميكروويف.

وعلى ذلك توصي الدراسة بإمكانية استخدام أفران الميكروويف المنزلية لتقدير الجوامد الكلية في هذه المنتجات كطريقة سريعة ودقيقة في نفس الوقت.