Trabajos originales

Moisture determination in cereals and cereal products by microwave heating and analysis of hydroxymethylfurfural (HMF) formed

Determinación de humedad en cereales y derivados por calentamiento con microondas y análisis de hidroximetilfurfural (HMF) formado

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RESUMEN

EL horno microondas ha sido usado para evaluar el tiempo y precisión en la determinación de humedad de granos de trigo y arroz, harina de trigo, pan blanco y galletas.

Los resultados fueron comparados con el metodo estandard de estufa de aire. El tiempo para las muestras de granos de trigo y arroz fue de 40 y 60 minutos respectivamente y de 15 minutos para el resto de los productos. Sin embargo, la precisión obtenida para todas las muestras, fue menor que con estufa de aire.

Se determinó HMF en muestras de harina desecadas por ambos métodos y los resultados mostraron valores considerablemente más elevados en la desecación mediante microondas.

Palabras clave: Humedad. Cereales. Microondas. Hidroximethilfurfural.

ABSTRACT

A study was conducted to evaluate the use and accuracy of domestic microwave oven for quick moisture determination of wheat and rice grains, wheat flour, sliced white bread and biscuits. The results were compared with the standard air-oven method. A decrease in drying time was achieved using the microwave oven. For wheat and rice grains the drying times were 40 and 60 min repectively, and 15 min for the cereal products, although coefficients of variation were lower in the air oven.

Hydroxymethylfurfural (HMF) was determined in flour samples dried by both methods. The results were considerately higher by microwave oven.

Key words: Moisture. Cereals. Microwave. Hydroxymethylfurfural.

Recibido: 20-2-1995.
Aceptado: 6-4-1995.
BIBLID [0004-2927(1995) 36:2; 207-213]

INTRODUCTION

Measurement of the moisture content of cereal grains and derivatives is on of the most important parameters in measuring quality and predicting storability

In general, moisture determination methods are chosen for either their rapidity or accuracy, although obtaining both simultaneously is the ultimate goal (1).

Microwave energy has provided the heating technique for moisture determination in a variety of vegetable products. Gorakhpurwalla *et al.* (2) determined the moisture content in corn and sorghum in 12 and 17 min respectively. Click and Baker (3) studied corn, soybeans, alfalfa and potato chips samples, and showed that considerable time was saved by using a home microwave oven. Davis and Lai (4) compared air-oven and microwave oven for wheat flour. Noomhorm and Verman (5) conducted studies on grains of wheat and rice which compared the official AOAC method with electrical and microwave-oven methods. They observed that the electric methods gave less accurate results.

The objectives of this work were to study a procedure for determining the moisture content of cereal grains and derivatives using a home microwave oven and to compare the results with standard oven methods. Bread and biscuit samples were also used since the bibliography consulted yielded no studies on these products.

At the same time, the HMF produced as a result of the browning reaction has been determined, which gives us information on changes in the food constituents and on possible fluctuations in the determination of moisture.

MATERIAL AND METHOD

Apparatus

Grains were ground in a Thomas-Wiley mill. The samples were heated in a Moulinex microwave oven (Model MF 460, output 1300 W, frequency 50 MHz) with a rotating plate. The samples were uniformly spaced around the outer edge of the plate. A Heraeus air oven (Model, RT 360) was used for the standard method.

Sample preparation

Rice grains, wheat flour (75% grade extraction), sliced white bread, and biscuits were bought in local stores, while wheat grains were supplied by a Spainsh manufacturer. Cereal grains and biscuit samples were ground to a size 30 mesh in a Thomas Wiley mill and the bread was sliced 1 cm thick and quartered. 5g samples (2 g for bread) were placed in glass containers (height 30 mm, diameter 60 mm) for both methods.

Standard method

Moisture determinations were performed according to the Official Spanish Method (6). The initial drying time was 1 h followed by 30 min intervals for all samples except wheat grains, for which 3 h intervals were employed.

Microwave oven

Preliminary tests with 11 replicate samples of each product were used to generate drying curves and establish necessary times for constant weight by microwave drying; constant weight was 0.5 mg differente in two successive samples. 100% power level was used for the rice, bread and biscuit samples and were cooled and weighed at 3 min intervals. The power level was reduced to 75% at 5 min intervals for the wheat grains as 100% power level burned the samples.

Then a single set of six replicates (the maximum number which could be dried together in the microwave-oven) of each product were dried and compared with air oven method. The conditions for each sample in the microwave oven are shown in Table 1.

Table 1.—Drying conditions in the microwave oven.

SAMPLES	Initial time *(min)*	Intervals *(min)*	Power *(%)*
WHEAT	10	10	75
RICE	5	5	100
FLOUR	5	15	100
BREAD	15	5	100
BISCUITS	15	5	75

Determination of HMF

Hydroxymethylfurfural was measured only in flour samples since, as we determined, the flour contained no HMF to start with. The samples were then dried using both methods.

In order to obtain estimable quantities of HMF in the air-oven samples, it was necessary to start with six samples of 5 g each. They were subjected to Soxhlet extraction for 8 h with 150 mL of organic solvents (chloroform and ethyl acetate mixture 1:1 v/v). Distilled water (40 mL) was added to the extract and the organic solvent was eliminated by distillation. The aqueous extract obtained was clarified with Carrez I and II solutions (0.5 mL of each), diluted to 50 mL with distilled water, then filtered. The HMF concentration was obtained from spectrophotometrical absorbances at 284 nm, according to the procedure outlined by White (7) for honey.

Statistical method

A regresion curve was used for calibration of the microwave oven (preliminary study). Variance around the means of each six data points was expressed as coefficients of variation (%), and significance of differences was assessed by Student's t-test.

RESULTS AND DISCUSSION

In the preliminary study, we determined the correlation between the effect of the drying time on the moisture content. Exponential equations were determined for the wheat and biscuit samples treated by microwave, giving R^2 values of 0.972 and 0.995 respectively. Verma and Noomhorm (8) reported slightly lower correlations in wheat and rice grains, possibly due to their using lower-strength microwaves in their experiments. Hyperbolic equations were determined for the bread and flour microwave samples with R^2 values of 0.947 and 0.988 respectively.

That study allowed us to calculate the correlation between the relationship moisture content and drying time as well as drying conditions in the microwave oven. Difficulties were encountered when attempting to establish the final drying times, since constant weights were not always obtained on consecutive weighings. In the final drying intervals, some samples actually showed an increase in weight, probably caused by transformations in the food constituyents.

Wheat grains

The average time to complete the moisture test using a domestic microwave oven was 40 min compared with 270 min for the air-oven drying method (Table 2). However, Verma and Noomhorm (8) reported lower drying times (24 min) when using an oven preheated for 5 min. The moisture content was higher using the microwave oven, although the difference in moisture content between the two methods was only 1%. Both results were shown to differ significantly ($p < 0.001$).

Table 2.—Average moisture content and statistical analysis obtained by both methods.

	AIR OVEN (130 °C)			*MICROWAVE*		
	Time (min)	$\bar{x} \pm SD$ (%)	*CV (%)*	*Time (min)*	$\bar{x} \pm SD$ (%)	*CV (%)*
WHEAT	270	9.72 ± 0.01	0.01	40	10.72 ± 0.13	3.73
RICE	120	13.20 ± 0.04	0.30	60	13.78 ± 0.11	0.80
FLOUR	90	11.84 ± 0.03	0.27	15	12.57 ± 0.16	1.28
BREAD	90	35.37 ± 0.90	2.54	15	36.05 ± 1.69	4.68
BISCUITS	90	2.59 ± 0.09	3.47	15	2.93 ± 0.03	10.24

Mean of six replications

Rice grains

The microwave oven was faster than the air oven (Table 2), although faster testing times were obtained by Noormhorm and Verma (5), who recorded results of 30 min at 100% power level for samples of rice with aproximately similar moisture content. The statistical comparison of both methods shows a significant difference ($p < 0.001$).

Sliced bread

The drying time is reduced to 15 min with the microwave oven (Table 2). The percentage of moisture in the samples differed by 0.68% for the two methods, and the variation coefficients were high, as well. The samples analysed were produced to have a long shelf-life, and it is thus possible that the incorporation of humectants caused these deviations. The two methods showed no statistical differences.

Biscuits

Here we are dealing with a product with a low moisture content (approximately 2%). The difference in the moisture values in the two methods was 0.34%. The variation coefficients turned out to be 1.02 in the microwave and 3.47 in the air oven. A comparison of the two methods shows statistically significant differences ($p < 0.05$).

Wheat flour

The results yielded indicate that the microwave consistently gives slightly higher values for moisture when compared to the air oven. The degree of difference is on the order of 0.7%. However, Davis and Lai (4) showed higher values using an air oven. CV% was low in both methods and the drying time was 15 min in the microwave oven, while a comparison of the two methods showed significant differences ($P < 0.001$).

Hydroxymethylfurfural

HMF is commonly used as an indicator in controlling the degree of heating for certain products. We have used it as an indicator to determine to what extent the drying method may favour the production of HMF.

After obtaining the results from drying the flour samples in the microwave oven, the hydroxymethylfurfural content was determined using the two methods tested. The average rate for HMF was 0.09/100 g in the air oven and 25.61 mg/100 g in the microwave.

The HMF variation in the samples is shown in the values of standard deviation. The positioning of samples in the microwave may not cause this variation, since we used a microwave oven with rotating plate and the sample were uniformly spaced around the onter edge of the plate. This variation can ought to the non uniformity of source radiation emission.

Flour samples dried in the two ovens yielded very different amounts of HMF, with the microwave producing considerably larger concentrations (Table 3). This suggests that browning reactions resulting from microwave treatment could result in loss of precision and accuracy in the measurement of moisture by microwave as compared with the air oven.

Table 3.—Determination of moisture and HMF in flour samples.

ASSAYS	*AIR OVEN (130 ± 2 °C, 2h, 30 min)*		*MICROWAVE OVEN (100% power, 15 min)*	
	Moisture (%)	*Free-HMF (mg/100g)*	*Moisture (%)*	*Free-HMF (mg/100g)*
1	12.24	0.12	12.47	24.05
2	12.19	0.11	12.12	7.37
3	11.44	0.06	13.31	40.37
4	11.36	0.04	13.25	30.65
5	11.70	0.11	12.31	20.61
6	11.76	0.07	13.61	42.47
MAX	12.24	0.12	13.61	40.37
MIN	11.36	0.04	12.12	20.61
MEAN	11.78	0.09	12.84	27.50
SD	0.11	0.03	0.61	12.71

Finally, the higher concentration of HMF in the microwave dried samples (given by the HMF parameter) is evidence of greater transformation of the carbohydrate than occurs in the air-oven samples.

CONCLUSIONS

The moisture content for all samples analysed was higher in the microwave than in the air oven. The microwave-oven method described has definite advantages due to the rapidity of analysis in determining moisture in cereal grains and derivatives. This speed of analysis makes the method appropiate for routine process controls in the manufacturing of this type of products. On the other hands, the air-oven CV were lower than in the microwave for all samples making this the more suitable method where precision of analysis is needed.

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