THE INFRARED ANALYZER MA35 SARTORIUS APPLICATION FOR MOISTURE DETERMINATION IN DRIED APPLES AND CARROTS

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Abstract: A comparison of the moisture content measurement results in dried apples with moisture from 2.5% to 28% (wet base) and dried carrots with moisture from 4% to 15% (wet base) obtained by infrared drying rapid method and classical vacuum oven method is presented. It was shown that IRD results depend both on the drying temperature and on the moisture level in the tested samples. We determined optimal drying conditions/ temperature for analyzer application being of 82 °C for dried carrots, and 82 °C or 98 °C for the dried apples with a brittle (and hard) or elastic texture, respectively. The systematic measurement errors that appear for dried samples with different moisture content at the same IRD temperature have been corrected due to reference values data using appropriate graphs and empirical equations.
Keywords: moisture determination; dried apples and carrots; infrared drying method; Sartorius Analyzer application.

Introduction

Rapid methods for determining the moisture are needed for a proper technological process management and to obtain the dried product of a standard quality. The Sartorius (Germany) moisture analyzer is widely used in analyzing the moisture content of chemical products, pharmaceutical, paper, cosmetic, textile, food and animal feed industry. In moisture determination the Sartorius application data for dried fruit and vegetables is very limited.

This is a thermogravimetric IR analyzer. The results obtained by infrared drying can be reproduced if the drying parameters are carefully chosen, as already reported in the literature [1, 2]. When the infrared dryer/analyzer is properly calibrated, it is well suited for application in the food manufacturing process [3]. The calibration is product-specific [4].

The aim of this work was to find out infrared drying conditions/temperatures to match the mass loss results with the moisture content of dried apples and carrots determined by classical vacuum oven method, used as a reference method.

Materials and methods

Samples preparation. Fresh apples (*Malus domestica Borkh*) of two varieties, Golden Delicious and Prima, from local market, 14-15° Brix, were used in this study. The apples, each variety separately, cleaned, sliced and mixed, were divided randomly into 7 portions. They were processed to obtain commercial quality dried apples samples in the range from 2.4% to 28% (wet base) moisture content, with 4% increments.

Each portion was dried in a pilot-plant tunnel air drier at the temperature of 62 ± 2 ºC and 2.5 m/s air velocity. Moisture loss during the drying was measured by weighting the sample at the beginning and periodically during the drying process, till the required mass and, therefore, the corresponding moisture content was attained.

Fresh carrot roots *(Daucus carota L.),* from the local market, having the average dry matter content of 14.7 %, were used in this study. The cleaned roots were cut into long thin strips and steam blanched at 100 °C, atmospheric pressure, for 5 minutes. They were cooled and drained on a sieve for a couple of minutes, mixed and divided randomly into several portions. They were processed to obtain commercial quality dried product in 4% to 14% (wet base) range. They were dried under the same process parameters as the apples.

Both apples and carrots portions were broken up to 2 – 4 mm uniform size bits, ground in an electric coffee mill, avoiding to be turned into fine powder. The ground samples were packed and tightly closed.

Reference method (RM) for moisture determination

The test portions of 2.5 ± 0.1 g for brittle and hard product and 3.5 to 4.0 g for elastic texture were placed in non-corrosive metal containers, provided with well-fitting lid, about 45 mm of diameter and dried in an electric vacuum oven under the pressure of

10 mm Hg (1.33 kPa). Cyclic weighings at approximately 10% of the total drying time were performed. The drying was carried out at constant temperature of 70 ± 1˚C for apples and 60 ± 1˚C for carrots to the constant mass point (At this point, the reduction in weight is less than 0.0010g in 2 hours of drying for apples and 6 hours of drying for carrots.).

The moisture content is defined as the mass loss between before and after drying measurements expressed as percentage of mass. Five parallel determinations for each analysis were carried out on the same test sample. Their average arithmetic value was taken as a final result.

Rapid method for moisture determination by IRD

A Sartorius MA 35 analyzer was used for infrared drying (IRD) rapid method. For sample heating, the MA35 is equipped with two 360 W metal tubular heating elements. These elements give more of an oven effect, providing intense heat throughout the whole heating chamber, and allowing for better drying of all sides of the sample. A built in balance with 1mg resolution do the test portions weighting.

The device offers two modes of operation, automatic and time mode. In the automatic mode the cyclic moisture content measurements are carried on till no change in mass is registered during a certain short time interval. In the time mode the operator is choosing the drying time from the beginning. He results will depend on the experience and reproducibility of drying curves. In both modes the drying temperature is to be chosen.

When decomposition reactions occur, resulting in values too high for the mass loss, a lower temperature has to be applied.

The preliminary experiments revealed that the determination of moisture, using the time mode led to unsatisfactory results. We used the automatic mode in our experiments. The first measurement results were excluded from each row of data, as reflecting an operating transitory state that appears at the beginning of the experiment [3]. Two-minute intervals between parallel measurements were set [1].

Two series of experiments were performed in this work. In the first series we varied the temperature and the sample moisture. Each analysis was carried out at least three times. The arithmetic average value was taken as a final result.

In the second series, the selected temperature was kept constant for each of three groups of the samples tested: the samples of dried apples with a brittle or hard texture and the samples of dried carrots were measured at the temperature of 82 ºC; the samples of dried apples with an elastic texture were analyzed at the temperature of 98ºC. Each analysis was carried out at 5-9 times. All tests were performed by the same operator. *Statistical analysis*

There were six procedures for statistical analysis of IRD experimental data in this study:

- calculating the average values ($\bar{y}_j$) and precision estimations (s_j) of the measurement results (y_{jk}) in the conditions of repeatability, for each level j of moisture (ISO 5725 -2);
- measurement results examination for consistency and outliers, using the Grubbs` tests, described in ISO 5725 -2, with the recalculation the statistics if data are rejected;
- investigation whether s_j depend on the average $\bar{y}_j$, and if so, the functional relationships determination, using the methods described in ISO 5725 -2;
- checking by the Fisher - test (with 95% confidence) whether the dispersions (s_j^2) calculated for various moisture levels are similar or dissimilar. If the null hypothesis (no difference) is accepted, the weighted average value of dispersions ($s_{n,q}^2$) and coresponding precision ($s_{n,q}$) are determined [5];
- repeatability limit (r) value determination (with 95% confidence) by ISO 5725-6;
- measurement results bias estimating ($\hat{\Delta}_j$) at each moisture level is expressed as an absolute value by ISO 5725 - 4:

$$\hat{\Delta}_j = \bar{y}_j - \mu_j , \tag{2}$$

where μ_j is an accepted moisture reference value (%), wet base;

The 95% confidence interval for the systematic error ($\hat{\Delta}_j$) of measurements is approximating calculated as follows:

$$\hat{\Delta}_j - A\, s_j \le \hat{\Delta}_j \le \hat{\Delta}_j + A\, s_j \quad (3) \text{ or } \hat{\Delta}_j - A\, s_{n,q} \le \hat{\Delta}_j \le \hat{\Delta}_j + A\, s_{n,q} \quad (4)$$

Here, A is a factor used for calculating the bias estimate uncertainty:

$$A = 1.96/\sqrt{n} \quad \text{or} \quad A = 1.96/\sqrt{N_{n,q}} \tag{5}$$

If the confidence interval includes zero, then the systematic error is not significant (α-level =0.05), otherwise it should be considered significant.

Results and discussion

Moisture determination. The objective of the first series of experiments was to find out a proper working value for the drying temperature.

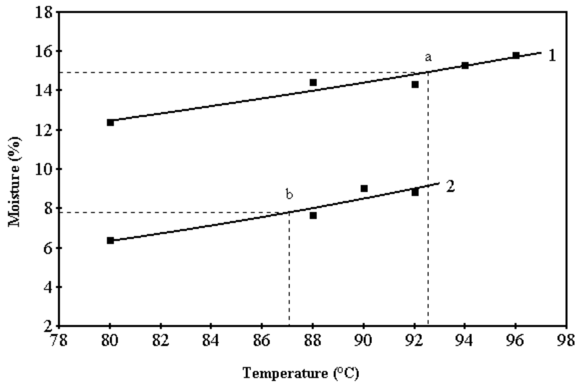

Fig. 1. Influence of IRD temperature on measured moisture value ($\overline{y}_j$) for different carrot moisture level (%): 1– 14.96, 2 – 7.82

We have presented the rapid method measured moisture results as a function of IRD temperatures for seven portions of dried apples; the most suitable temperature parameters have been found as the 82 °C and 98 ºC for testing dried apples below and above 7.5 % moisture content, respectively [6]. Figure 1 displays the rapid method measured moisture results as a function of IRD temperatures for two portions of dried carrots. We determined two values for most suitable temperatures of 87 and 93 ºC for dried carrots with the true moisture content of 7.82% and 14.96%, respectively. We expect lower temperatures required for dried carrots samples with moisture below 7%. Based on the dried apples experiment results [6], temperatures around 80ºC would be good for this case. As the carotenoids in carrots are unstable to heat, to avoid thermal degradation resulting in distortions of IRD measurement results, an 82 ºC temperature for the whole range of sample moisture was applied in all our experiments with dried carrots.

Statistical analysis. The statistical analysis of measurement results, for the first and second series in common, for three groups of products: dried apples with a brittle or hard texture, dried apples with an elastic texture, and dried carrots showed that (Table 1):

- no functional noteworthy relationship between values s_j and $\overline{y}_j$ or μ_j was detected for any of the tested groups of products;
- no significant differences were found between the values of s_j within the same group of samples, which allowed us to unite in one batch all statistics results obtained for the same group of product and to calculate weighted average of standard deviation and repeatability limit (Table 2);
- the measurement bias at 82 °C for four levels of brittle or hard dried apples were found to be insignificant values in two of the levels and significant values for the other two. The measurement bias for 98 °C – dried apples and at 82° C – dried carrots were considered significant values for all of the indicated levels with one single exception. A systematic error in IRD moisture determination for all three groups of tested dried samples appeared. Based on this fact, correlations of $\overline{y}_j$ with μ_j was studied using linear and non-linear least square regression analysis. The correlations were good:

for dried apples

$Y(x) = 2.1507e^{0.1660x}$, R^2=0.995, s=0.1638 in the 2.5% – 7.5% moisture range;
$Y(x) = 1.5115 + 0.9380x$, R^2=0.9976, s=0.3274 in the 7.5%–28% moisture range;
for dried carrots
$Y(x) = 0.6258 + 0.8450x$, R^2=0.975, s=0.5187 in the 4.1%–15% moisture range.

Table 1. *Statistical estimation of IRD moisture values*

Statistical indicators								
j	n_j	s_j	$\overline{y}_j$	μ_j	$\hat{\Delta}$	The cnfd. interval (4)		Δ
						$\hat{\Delta}$ -	$\hat{\Delta}$ +	sgn
obtained at 82 °C for brittle or hard samples of dried apples								
1	3	0.0611	4.20	4.24	0.04	- 0.06	0.14	no
2	3	0.1305	7.57	7.67	0.01	- 0.09	0.11	no
3	5	0.1297	7.56	7.41	0.15	0.05	0.25	yes
4	5	0.2969	3.35	2.54	0.81	0.16	0.36	yes
obtained at 98 °C for samples of dried apples with elastic texture								
1	3	0.31953	8.46	7.67	0.79	0.7	0.88	yes
2	3	0.26514	14.17	13.29	0.88	0.79	0.97	yes
3	3	0.40004	18.92	18.06	0.86	0.77	0.95	yes
4	3	0.22605	23.49	23.73	0.24	0.15	0.33	yes
5	3	0.03214	25.03	25.47	0.44	0.35	0.53	yes
6	3	0.63540	28.01	28.02	0.01	- 0.08	0.10	no
7	8	0.14870	9.30	8.70	0.60	0.51	0.69	yes
8	8	0.43398	15.60	14.75	0.85	0.76	0.94	yes
9	7	0.41457	19.26	18.49	0.77	0.68	0.86	yes
10	7	0.20254	23.82	23.70	0.12	0.03	0.21	yes
11	6	0.14774	25.46	25.90	0.44	0.35	0.53	yes
obtained at 82 °C for dried carrots								
1	8	0.3731	4.36	4.09	- 0.27	0.11	0.43	yes
2	4	0.4361	6.72	7.82	- 1.10	0.94	1.26	yes
3	7	0.5438	9.14	9.49	- 0.35	0.19	0.51	yes
4	9	0.4656	9.36	9.61	- 0.25	0.09	0.41	yes
5	4	0.4053	12.89	14.96	- 2.07	1.91	2.23	yes

The IRD measurements performed at different times could be subjected to environmental changes influence. It follows that if s_j is a precision estimate in terms of repeatability, then $s_{n,q}$, according to ISO 5725-3, may be regarded as the standard deviation of the intermediate precision with differences in the "time" factor, i.e. $s_{I(T)}$. Therefore, the absolute value of the difference between the results of two measurements obtained by IRD in the conditions of intermediate repeatability (at the differences in the factor "time"), should not exceed, with 95% confidence or probability, the value of $r_{n,q}$ specified in Table 2. There are relatively low. This is concluded by comparing them with ones standardized for oven drying method (the r values of 1% when samples are drying together and 3% when samples are drying at different times or in different laboratories are included in the national standard of the Republic of Moldova).

Table 2. Statistical indicators for the united batch of various types of product

Sample				
Dried apples with brittle and hard texture	16	12	0.2099	0.58
Dried apples with elastic texture	54	43	0.3212	0.89
Dried carrots	32	27	0.4532	1.25

Conclusions

Moisture content data obtained by infrared drying rapid method and classical vacuum oven method for dried apples with 2.5% to 28% moisture and dried carrots with 4% to 15% moisture were studied. It was shown that IRD method results depend on the drying temperature as well as on the level of moisture in the tested samples.

In analyzing the dried apples IRD method results we used different temperature behaviour approach for different dried apple textures: 7.5% to 28% moisture dried apples have an elastic texture, below 7.5% moisture have a hard to brittle texture.

The systematic measurement errors that appear for dried samples with different moisture content at the same IRD temperature have been corrected due to reference values data using appropriate graphs and empirical equations.

The statistic studies confirm that the infrared drying rapid method can be used to measure moisture content in dried fruit/vegetables industrial and commercial control units under strict conditions. For valid results, the moisture level of tested product should be taken into consideration.

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