RESEARCH ON WATER CONTENT OF JONATHAN APPLES BY CLASSICAL AND INNOVATIVE METHODS

CERCETĂRI PRIVIND DETERMINAREA CONȚINUTULUI DE APĂ PENTRU MERELE DIN SOIUL JONATHAN PRIN METODE CLASICE ŞI INOVATIVE

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Abstract. *This paper presents results on the moisture determination on the Jonathan apples by oven drying method and lyophilization. Statistical interpretation of experimental results allowed the comparison of the two methods, selection of the most convenient in terms of simplicity, speed and cost as well as achieving a hypothetical model on the percentage variation of water forms with applicability for the conservation of food substrates.*
Key words: water, Jonathan apples, lyophilization, FTIR spectroscopy.

Rezumat. *În lucrare sunt prezentate date privind determinarea umidității la merele din soiul Jonathan prin metoda uscării la etuvă şi liofilizare. Interpretarea statistică a rezultatelor experimentale a permis compararea celor două metode, selectarea celei mai convenabile din punct de vedere al simplității, rapidității şi a costurilor precum şi realizarea unui model ipotetic privind variația procentuală a diverselor forme de apă cu aplicabilitate pentru conservarea substraturilor alimentare.*
Cuvinte cheie: apa, mere Jonathan, liofilizare, spectroscopie FTIR.

INTRODUCTION

The scientific literature contains information on the likely forms of water from supply substrates.

Content of principal forms of water (or proportional ratio) provides practical information for agriculture, horticulture, biology, medicine.

Free water/bound water proportional ratio can be a useful index for assessment of physiological processes-organs specific, or for metabolic characterization of living organism (plant/ animal).

Currently scientific literature presents the characterization of water forms as a general concept level (Baucour et al., 2000, El Sayd et al., 2010) with limited focus depending on the specialization approached field (physiological, physical, chemical, biochemical, technology).

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Development of analytical methods will provide characterization and determination of the principal forms of water from food supply with applicable results in practice (Derossi et al., 2007, Trincă, 2004).

It is accepted that by reducing the water content of fruits and vegetables it is possible to preserve them for a long time, hight contents of water (90-98%) affecting their perishability.

In practice the drying processes are used to inhibit the growth of microbial flora and to limit the effects of degradation reactions (Rahman, 2006).

This paper aims to determine physico-chemical water content and to highlighte the presence of chemically bound water from Jonathan apples structure by various methods and to carry out statistical correlations of experimental data.

MATERIAL AND METHOD

In early November 2011 JONATHAN apples were purchased from the city market with weight ranging between 81-103 g g and normal, healthy appearance.

Periodic measurements for various types of water monitoring were carried out for 60 apples (by considering the lot of ten apples stored under the same conditions of temperature and humidity).

Determination of free water was performed for twenty days by weighing (every five days) the apples, the difference in mass being attributed to the percentage of free water. Drying of the samples was carried out by two methods: the oven drying and lyophilization drying .

For oven drying 5 g sample of Jonathan apple (cuted into pieces, minced, and pressed to get the juice) have been subjected to drying at 90 ^{0} C temperature until constant mass ($\Delta < 10^{-2}$ g).

Water content was determined according formula:

$$H_2O\ \% = \frac{m - m_1}{m_2} \times 100$$

m = mass dish + mass sample before drying, $\mathbf{m_1}$ = mass dish + mass sample after drying, $\mathbf{m_2}$ = mass of the sample.

Lyophilisation is a process of dehydration by cold : water freezes faster than other components and is removed in the form of ice, without changing the structure of the food substrate.

Lyophilisation has occurred with Freeze Dryer ALPHA type 1-4 LD p l u s. 5 g sample of apple (cuted into pieces, minced, and pressed to get the juice) were subjected to freeze-dryer drying at a temperature of – 50 -60 C^0, and 0.02-0.03 mbar pressure until constant mass.

Spectral analysis by FTIR spectroscopy highlights functional changes of the structure sample substrate in relation to the standard.

FTIR spectra were recorded in KBr pill using DIGILAB-EXCALIBUR SDS 2000 spectrometer fitted with a heating device.

Working parameters were: spectral range between 4000-400 cm^{-1}, resolution 4 cm^{-1} and the number of scans 24.

By MATLAB function the correlation coefficients have been identified for apples samples (day five and day twenty) for free water, physico-chemical water (determined by drying in the oven and lyophilization for the Apple (cuted into pieces, minced, and pressed to get the juice).

For the statistical analysis of the recorded differences it was applied the method of analysis report variances (X) and Pearson correlation coefficient, determination gradient has been calculated (by considering the case $p < 0.05$ statistically significant). Statistical evaluation was performed using SYSTAT 13 (SYSTAT SOFTWARE, Inc. CHICAGO).

RESULTS AND DISCUSSION

Our study provide that drying methods can be used in the determination of water content.

Table 1

Free water content (%) dynamics variation during experimental period

Parameter	Day five	Day ten	Day fifteen	Day twenty
MEAN±ST DEV	1.423±0.148	2.878± 0.546	3.671±0.668	5.363±0.944
MINIMUM	1.199	2.051	2.833	4.234
MAXIMUM	1.565	3.442	4.420	6.427
VARIANCE	0.110	0.435	0.556	0.792

Free water content increased statistically insignificant on the day twenty compared to day five, which reveals a moderate loss of water in the food substrate for Jonathan apples variety compared to other varieties (Vesali et al., 2011, Gradinariu et al., 2002).

Table 2

Dynamic variation of physico-chemical water content (%) determined by lyophilization during experimental period

Parameter	Day five	Day ten	Day fifteen	Day twenty
MEAN±ST DEV	78.340±3.248	78.025±1.211	77.923±0.960	77.528±1.185
MINIMUM	75.432	77.020	77.202	75.457
MAXIMUM	83.854	79.888	79.216	78.340
VARIANCE	10.551	1.467	0.921	1.404

Water percentage of samples was statisticaly significant when compared drying by oven to drying by freeze.

Physico-chemical water content determined by lyophilization decreased by 1.04 % on day twenty compared to day five, the same sense of variation (1.17 %) being registered for the juice resulted by pressed apples.

Statistical processing highlighte a positive correlation between physico-chemical water-bound determined by lyophilization and physico-chemical water-bound determined by drying in oven the juice resulted by pressing the apples ($r^2 = 0.757$).

Statistical processing of individual results for each of the ten apple revealed linear correlations for day twenty between free water and physico-chemical water determined by lyophilization ($r^2 = 0.376$).

Table 3

Dynamics of physico-chemical water-bound variation content (%) determined by drying in oven the juice resulted by pressing the apples

Parameter	Day five	Day ten	Day fifteen	Day twenty
MEAN±ST DEV	80.424 ±1.982	80.262 ±0.484	80.209±2.324	79.482±1.366
MINIMUM	77.998	79.543	76.556	77.349
MAXIMUM	83.372	80.655	82.965	80.871
VARIANCE	3.931	0.234	5.403	1.866

Statistical processing of individual results for each of the 10 apple revealed for twenty day linear correlations between free water and to physico-chemical water determined by drying in the oven the juice resulted by pressing the apples ($r^2 = 0.280$).

Satistical analysis of the results showed through statistical modelling interrelationships between the two forms of water investigated.

Table 4

Dynamic variation of physico-chemical water content (%) determined by drying apple pieces in the oven

Parameter	Day five	Day ten	Day fifteen	Day twenty
MEAN±ST DEV	79.801 ±0.587	79.543 ±1.699	77.690±3.885	77.193±2.848
MINIMUM	78.925	76.865	71.048	73.166
MAXIMUM	80.578	80.655	80.580	81.208
VARIANCE	0.345	2.887	15.097	8.116

Physico-chemical water content determined by drying minced apple in the oven decreased by 0.01 % on day five, and by 0.47 % on day twenty.

In day five hydration state of the food substrate ensured a good resistance of cellular walls compared to day twenty, in wich the significantly elimination of free increased the amount of physico-chemical water pool (table 3).

In the same time the dehydration process stressed the destruction of cellular walls and release of intracellular water.

Table 5

Dynamic variation of physico-chemical content (%) determined by drying minced apple in the oven

Parameter	Day five	Day ten	Day fifteen	Day twenty
MEAN±ST DEV	79.802 ±5.106	79.384 ±1.035	78.925±1.612	77.666±2.379
MINIMUM	71.998	77.693	77.075	76.105
MAXIMUM	86.264	80.274	80.133	81.653
VARIANCE	2.608	1.073	2.599	5.662

Statistical processing of the results for the apple pieces and minced for day five does not revealed a linear correlation between the individual parameters.

This fact can be explained by consideration of crushing as a process of cellular destruction not so easy to quantify in terms of infracelular water released.

In the case of minced apple it was produced the destruction of cellular walls (process largely difficult to quantify) which caused the release of intracellular water.

Appropriate analyses of experimental data of twenty day highlighted positive correlation (r^2 = 0.354) between physico-chemical water of apple pieces compared to minced wich sustains the hypothesis that increased dehydration produce interconversion water forms.

Comparative analysis of methods used in this trial highlighted the advantage of accessibility for drying oven method while freeze-drying method provides better results by cancelling the potential errors of drying oven such as evaporation of volatile compounds or destruction of the walls of the food substrate.

Determination of chemical water related by FTIR spectroscopy

In figures 1 and 2 are presented FTIR spectra for apples (pieces and minced) in day five and day twenty.

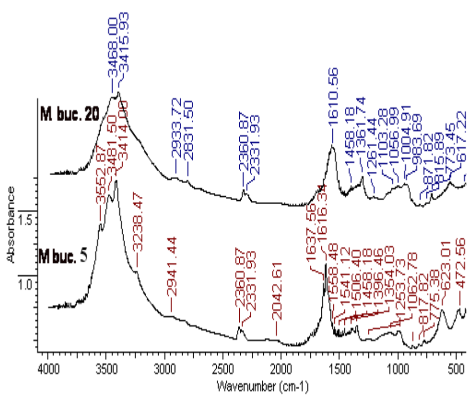

Fig. 1 - FT-IR Spectra of apple pieces samples in day five and day twenty

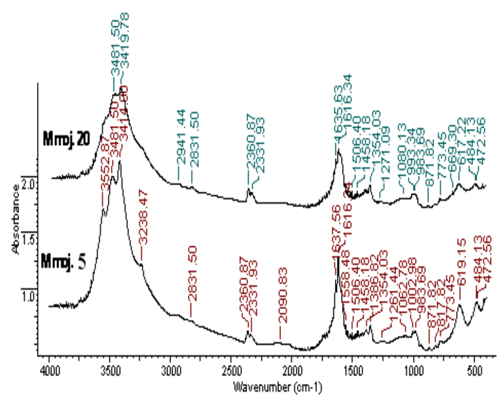

Fig. 2 - FT-IR Spectra of minced apple samples in day five and day twenty

The corresponding signals of water in the food substrate are given by symmetric and asymmetric bands of excitation of phenolic groups -OH present in the area 3552-3540 cm^{-1}. These signals are specific to chemical bound water from the substrate structure. Thus, from these spectra can be notice that intense signals appears in the area just in the case of the samples analysed in day five. Spectra recorded for samples of day twenty day presented low intensity signals in this area maybe because of the existence of a smaller percentage of chemical bound water in raw samples. In this case the signals appear only in the aria of 3480-3420 cm^{-1} both for apple pieces and minced samples.

CONCLUSIONS

1. Determination of apple moisture samples revealed that drying by lyophilization ensures a high content of physico-chemical water because this method does not cause the destruction of the substrate.

2. FTIR analyses confirmed the presence of chemical bound water (specific signals for are powerfull in day five while in day twenty can be traged their absence in certain areas of the absorption).

3. Statistical data sustain for day five linear correlations only between free water and physico-chemical water determined by drying in oven of apples pieces fact that can be explained by corresponding hydraotion state in day five.

4. In day twenty the free water content is related to physico-chemical water-bound (both for apple pieces, minced apple and juice resulted from pressing the apples samples) determined by the oven drying and lyophilization.

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