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DETERMINATION OF SUGAR CONTENT IN MILK POMADE SWEET BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY AT THE SHELF LIFE

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Abstract

The objective of the research was to evaluate the carbohydrate content changes in milk pomade sweets (sherbet) during the storage time. The samples were hermetically packed in modified atmosphere (MAP) consisting of 100% CO_2 applying three types of packaging materials (OPP, Multibarrier 60 and PP). Packaging in air ambiance was used as a control. The samples were stored at the room temperature +21±1 °C, moisture content and cutting force of samples were analyzed before packaging (day 0) and after 2, 4, 6, 8, 10, 12, 14 and 16 storage weeks. Carbohydrate content changes in milk pomade sweets (sherbet) were analyzed before packaging (day 0) and after 4, 8, 12, and 16 storage weeks.

Carbohydrate content in the sherbet samples was determined by high performance liquid chromatography (HPLC). The total sugar content analyze showed that excepting basic composition of sucrose the sherbet comprises also sugars like fructose, glucose, turanose, maltose, maltotriose and melecitose. Fructose, glucose and melecitose were found in a relatively small quantity and were less than 1%.

Statistical processing of the data showed that used packaging materials do not have an impact on the total sugar composition in sherbet samples during the storage time.

Key words: *sugar content, milk pomade sweets, packaging, shelf life*

1. INTRODUCTION

The confectionery industry is enormous. It ranges from small shops to branches of the largest companies in the food industry. The industry is divided into three classes: chocolate, flour and sugar confectionery. Manufactures of most modes of confectioneries are not science-based professionals an exception to this is development of products resembling sugar confectionery but free of sugar, where more scientific efforts have been required. (Quinton and Kennedy, 2002)

Sugar confectionery has been developed over the centuries with increasing sophistication and it exists in countless formats with different degrees of sweetness, flavours and aromas, textures and mouthfeel. Confectionery serves a very simple purpose; the rush of sweetness coupled with pleasant flavours, aromas and mouthfeel provokes an almost instantaneous feeling of well-being and happiness. Sugar confectionery by definition is meant to include products that contain predominantly one form or another of the following sugars: sucrose (usually cane or beet sugar); dextrose (otherwise known as glucose, usually corn sugar); fructose (often referred to a fruit sugar) or lactose (otherwise known as milk sugar). (Zumbé, 2001)

Milk pomade sweets are one of the sugar confectionery products. Milk pomade sweets usually contain sugar, glucose syrup, water, condensed milk; it may also contain sugar and nuts depending on product

category. The shelf life of milk pomade sweets depends on several parameters including: storage temperature and humidity, availability of oxygen in the immediate environment, directly related to packaging material used, as well as the addition of other ingredients such as fats, nuts etc.

Scientists Mexisa S.F., Badekaa A.V., Riganakosa K.A. and Kontominas M.G. mention, that the chocolate shelf-life is affected by similar factors.

Shelf life is the duration of that period between the packaging of a product and its consumption, when the quality of the product remains acceptable to product user or the shelf life refers to the time when a food can satisfy on both the retailer's and consumer's requirements before it becomes unacceptable. Product shelf life can be controlled by three factors: product characteristics, the environment to which the product is exposed during distribution, and the properties of the packaging material. (Raisi and Aroujalian, 2007)

The shelf life of sugar confectioneries is limited by a qualitative decay due to severe hardening of the internal paste. This alteration could be due to both the redistribution of water that leads to the crystallization and loss of water into the surrounding environment. In fact, almond all pastry cookies are high in sugar and low in moisture. The sucrose recrystallization causes release of water and subsequent redistribution of moisture. The water could be absorbed by other components and lost from cookies. The hypothesis that sucrose recrystallization is responsible in part for the firming of soft cookies was demonstrated by Belcourt and Labuza. (Romeo et all, 2010)

Controlling crystallization of sugars in confectionery products is critical to develop their proper appearance, texture and storage stability. Sugar crystals in the product may be desirable (e.g. sugar shell, fondant, toffee and fudge), or undesirable (e.g. hard candy, jelly and caramel). (Ben-Yoseph and Hartel, 2006)

In a fondant system, the sugar crystals are normally developed during the heating process of sucrose/glucose syrup to a high temperature and by following cooling down. The correct sizes, shapes and numbers of sugar crystals are dependent on the proper control of crystallization process, which is easily affected by external factors such as heating temperature, cooling rate, agitation speed, beater shape, and contaminants that can act as nuclei of crystallization. (Lau and Dickinson, 2007)

Hardness of confectionery products can be affected not only by moisture content in product, but by other parameters as well. As have been indicated by R. Ergun, R. Lietha and R. W. Hartel, in sugar-based confections the water content is generally governed by the boiling point relationship of the sugars present in the formulation. The final water content has a significant impact on texture and shelf life, with lowers moisture content leading to hardening of candies that typically have longer shelf life. However, water content by itself is not sufficient to completely characterize the quality of candies and shelf life. Water activity, or the relative vapor pressure, of candies is an important parameter often used to describe microbial stability, texture, and water migration during storage. (Ergun et all, 2010)

However, the end of the shelf life due to moisture loss or gain, with subsequent changes in textural and other properties, is often the main problem of candies. Thus, an understanding of water activity is important for control of shelf life and stability of candies. (Ergun et all, 2010)

The changes in water content due to moisture migration can affect both chemical reactions and physical properties of candies. These changes may be desirable, causing development of flavors and textures, but most often, the changes are detrimental and decrease consumer acceptability of the product. Whether the moisture picks up or loss in candies depends from water activity (a_w) and relative humidity (RH) in storage ambience could be ascertained. (Ergun et all, 2010)

The objective of this research was to evaluate the carbohydrate content changes in milk pomade sweets (sherbet) during the storage time, when hermetically packed in modified atmosphere (MAP) consisting of 100% CO_2 applying three types of packaging materials (OPP, Multibarrier 60 and PP).

2. MATERIALS AND METHODS

2.1. Experimental design

Experiments were carried out at the Department of Food Technology, Latvia University of Agriculture. The object of the research was milk pomade sweet ***sherbet*** with crunchy ***peanut*** chips, produced by stockholder *Laima*, Latvia. Ingredients of sherbet: sugar, peanuts (24%), condensed milk with sugar, water, glucose syrup, wafers (wheat flour, egg mass, baking agent (E500), emulsifier (soya lecithin), salt. Dimensions of one piece of sherbet in average was 40 x 40 x 8 mm, mass on average 30±1g.

2.2. Packaging and storage of samples

The study involved preliminary preparation of polymer pouches (80 x 120 mm) applying three types of packaging materials: OPP, Multibarrier 60 and PP, following packaging of sherbet samples in modified atmosphere (MAP) containing 100% of CO_2 and in air ambiance and carton boxes as control and storage in room temperature at +21 ± 1 ºC (controlled by MINILog, Gresinger electronic) and about 40% RH for 16 weeks under day and night conditions. The materials for experiments were selected with different water vapour transmutation rate and various thicknesses in order to asses whether hardening can be ascribed to water loss from sherbet sample or from redistribution of the moisture inside the product or by combination both of moisture loss and redistribution. Two pieces of sherbets were placed in each package. The total product mass in each package – 60 ± 1.0 g. The results were reported as averages of all determinations. Moisture content and cutting force of samples were analyzed before packaging (day 0) and after 2, 4, 6, 8, 10, 12, 14 and 16 storage weeks. Carbohydrate content changes in milk pomade sweets (sherbet) were analyzed before packaging (day 0) and after 4, 8, 12, and 16 storage weeks.

2.3 Physico-chemical analyses

The following chemical and physical characteristics were analyzed:

- Moisture content and accordingly at the storage time was determined by using verified balance KERN (Germany) with precision ±0.001g ; mass loss (%)were determined by weighing the samples on the electronic scales;
- Hardness for freshly manufactured sherbet samples was determined as cutting force (in N) by using TA-XTplus Texture Analyser. Cutting force was determined for six small sherbet samples from each it piece. For the sample cutting force determination a special probe with knife edge for a cut test HDP/BSKBLADE SET WITH KNIFE was applied. The maximum cutting force (in N) was detected at the deformation rate 10 mm/s and distance 10 mm. The samples were cut right through, in order to check whether any different structural characteristics ***(peanut pieces)*** were present under the knife inside the product or on the surface. Samples for cutting were placed centrally under the knife edge. Plotting force (in N) versus storage time (in weeks), the hardness change of sherbet stored in each gas composition in the package as well as for each packaging material was calculated. The maximum cutting force (N) was used as an index for the cut test.
- The sugar content of milk pomade sweet – ***sherbet*** with crunchy ***peanut*** chips was determined by applying the method of high performance liquid chromatography (HPLC). The method is

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based on the fact that the chromatographic separation of glucose, fructose and sucrose is based on their delayed time differences (Kūka, 2008), which enables to determine the quality and quantity analysis of the sample. Liquid chromatography is a method of separating and analyzing mixtures of substances in which the mobile phase is a liquid. By the method of liquid chromatography it is possible to separate fixed and thermally non-persistent substances. In the liquid chromatography, separation of substances takes place in a constant temperature which most often is close to room temperature. In the liquid chromatography, the mixture to be separated is forced through columns and the mobile phase is let in.

By injecting the sample in the HPLC, with the mobile phase it flows to the column. In order to guarantee high efficiency, correct injection of the sample, its quantity and volume have essential importance. After the extraction of carbohydrates they are determined by using refraction detector which monitors composition of elution flown out of the column. The signal coming from the detector is electronic; therefore it can be processed by using electronic data processing systems. For determination of carbohydrates in berries, light refraction coefficient detector is used. It is all-purpose and can be used in cases when the substance does not absorb UV radiation, for example, in determination of carbohydrates. The pikes are identified according to the time of a particular carbohydrate flowing out from the chromatography column. Composition of carbohydrates is calculated by using standard method according to the pike square (Kúka, 2008).

Fundamentals of the standard method are construction of calibration curve by using sugar standard-solutions of diverse concentrations. By using the obtained calibration curves, concentrations of diverse sugars are found in the analyzed sample. Using potentialities promoted by Shimadzu LC-20 Prominence with light refraction coefficient detector of the RID-10A brand liquid chromatography software, analyses of the obtained results of the calibration curve and the analyzed sample is carried out automatically.

The research was made according to the standard method. The reagents used: glucose, fructose, maltose, sucrose, acetonitrile and deionised water. Preparation of the basic solution – weighed out 2±0.0002 g of fructose, glucose, maltose and sucrose in 10 ml volumetric flasks, filled with deionised water up to the mark and mixed thoroughly (concentration of the obtained mass was 0.2 g ml^{-1}).

Parameters of the method:

- column: amino column (- NH_2);
- isocratic regime;
- temperature of the column and detector: 25 °C;
- mobile phase: acetonitrile/water (80:20 V/V);
- capacity of the injection sample: 10 µl;
- total time of the analysis (glucose, fructose, sucrose, maltose): up to 30 minutes;
- rate of the flow: 1.3 ml min^{-1}.

Liquid chromatography of the carbohydrate calibration:

the chromatography data processing system fixes the composition of glucose, fructose, sucrose, maltose in berry by comparing the berry chromatography with the chromatography of sugar standard-solution mentioned above.

Composition of carbohydrates in the analyzed samples was calculated in the form of percentage expression. The following formula was used in calculations:

$$W\,\% = \frac{CxV}{m} \times 100, \qquad (1)$$

Where:

C – concentration read, g l^{-1};

V – capacity of the extraction solution (total), l;

m – weighed mass, g

2.4. Statistical analysis

The results were processed by mathematical and statistical methods. Statistics on completely randomized design were determined using the General Linear Model (GLM) procedure SPSS, version 16.00.

3. RESULTS AND DISCUSSION

The aim of this work was to assess the effect of MAP composition (100% CO_2) during the storage time on the carbohydrate content and their changes in ***sherbet*** with crunchy ***peanut*** chips samples affected by moisture losses and hardening in different polymer packaging and compare with storage conditions in air ambiance.

Initial moisture of samples was 3.21±0.01 %. As can be seen (Figure 1) the moisture content during 16 weeks storage decreased. Significant differences in moisture content values during the 16 weeks storage among all sherbet samples (B, C, D, E, F and G) packed in different types of polymer materials with barriers properties were not found ($p>0.05$). Experimentally it was established that moisture content of the product samples packed in air ambience in cardboard boxes (control sample – A) decreased from up to 1.3% and it differs from those packed in all other types of polymer pouches ($p<0.05$), in which moisture content decreased on average from 2.2 up to 2.7%.

The changes of cutting force of sherbet samples packed in various types of polymer packaging materials in 100% CO_2 ambiance (MAP) and in air ambience and, compare to packaging in cardboard boxes (control sample – A) are presented in Figure 2.

Sherbet texture becomes harder irrespective of used packaging material type and corresponding moisture cont changes. The major reason can be water vapour migration through the packaging material and subsequent sucrose crystallisation, which promotes hardening. The initial cutting force of all samples determined at the beginning of the experiment was 53.6±2.0 N. Mouth feel, texture and eating qualities are adversely affected by the loss of moisture (Sabdo, 2009). For our tested sherbet samples, mouth feel of all samples packed in polymer materials during the storage time was acceptable. Experimentally it was established that cutting force of the product samples packed in air ambience in cardboard boxes (control sample – A) during 16 weeks of storage increased from 53.6±2.0 N up to 540.24±1.5 N and it differs significantly from those packed in all other types of polymer pouches ($p<0.05$). Significant differences in cutting force during 16 weeks storage among all sherbet samples (B, C, D, E, F and G) packed in three types of polymer materials in 100% CO_2 (MAP)

and air ambience was not found ($p>0.05$) – after 16 storage weeks they showed increase in the cutting force on average 218.65±1.5 to 336.97±2.0 N.

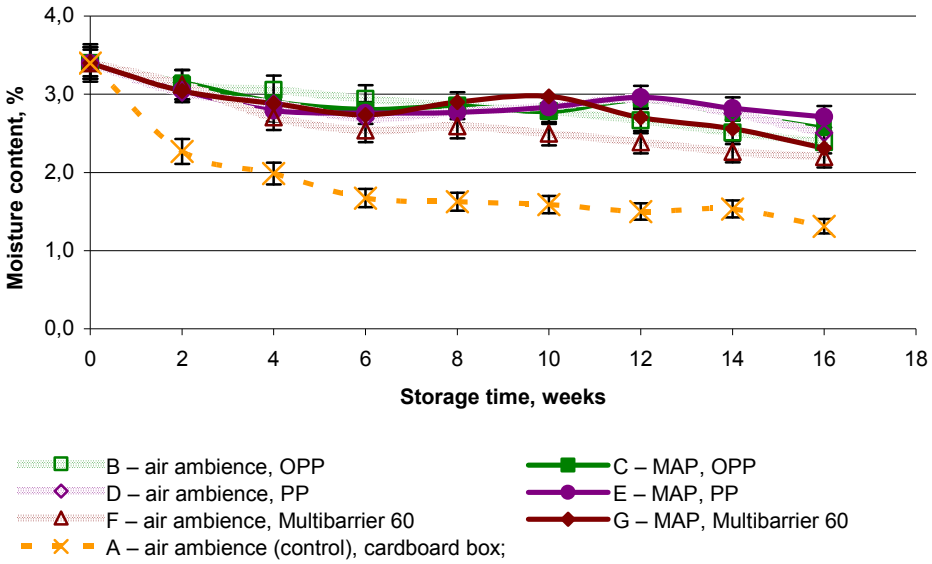


Fig. 1. Moisture content in air ambience and in MAP packed sherbets during the storage time.

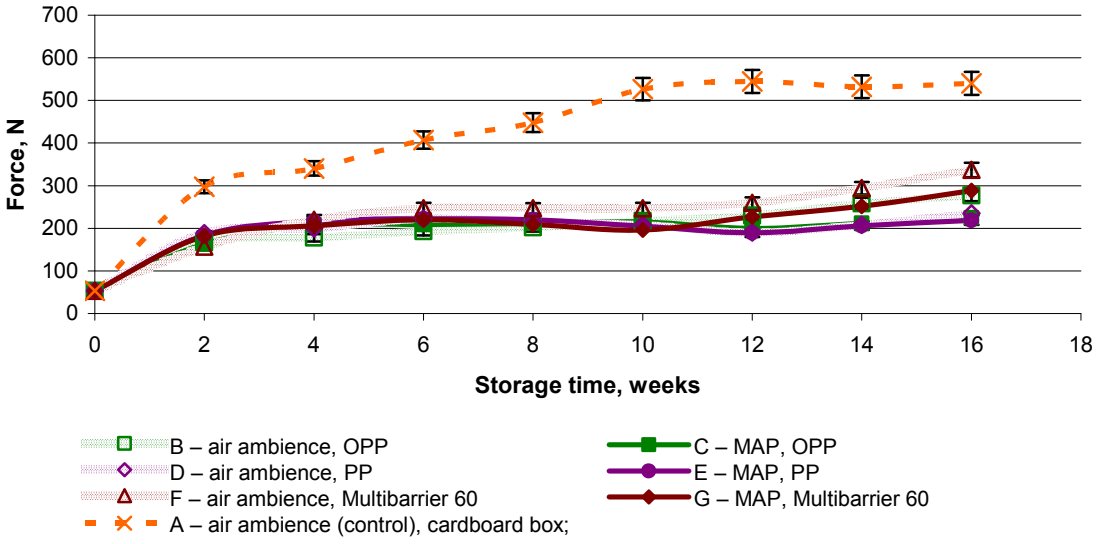


Fig. 2. Cutting force dynamics of air ambience and MAP packed sherbets during the storage time.

When sweets are stored without proper packaging, especially in ambiance of high humidity, the sucrose can crystallize, making the sweets sticky and grainy. Traditional packaging materials (paper

based) are often used for sweet's wrapping. However, this kind of packaging does not provide sufficient protection during long time shelf-life because their week barriers to water vapour migration, they cannot be securely sealed as well. Alternatively, individual wrapping should be made from waxed paper, polymer laminate with aluminium foil, and cellulose film, or a combination of those. In most cases, the sweets have been hand wrapped, but for higher production capacities, semi-automatic wrapping machines are available. For further protection, the individually-wrapped sweets could be packed in flow packs in heat-sealed polymer material bags.

The total sugar composition in investigated sherbet samples before packaging was 60%. As it was found in this study, sucrose (Figure 3) is the primary component of sugars (53.5±2%) in ***sherbet*** with crunchy ***peanut*** chips. A significant disparity between sucrose content (from 53.5±2.0% till 60±5%) in air ambience and MAP packed sherbet samples in all applied packaging types during storage up to 16 weeks ($p>0.05$) was not observed.

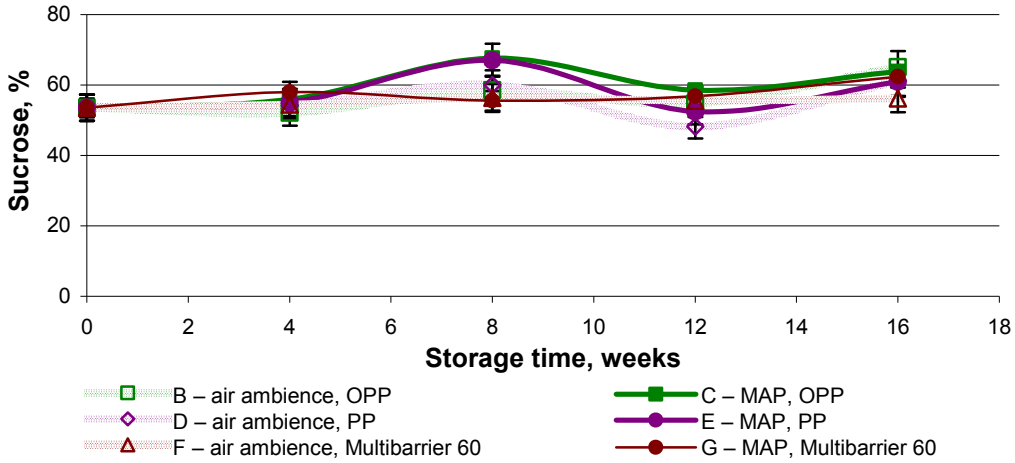


Fig. 3. Sucrose dynamics in air ambience and in MAP packed sherbets during the storage time.

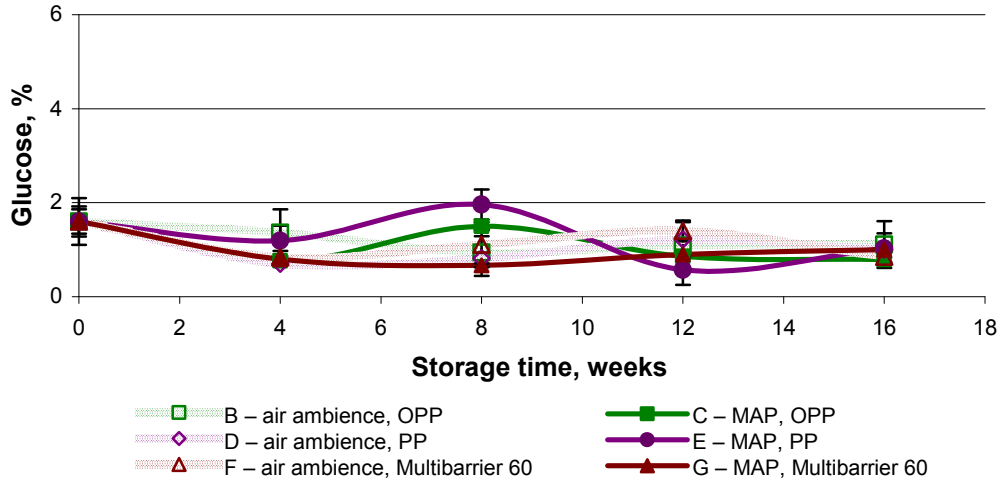


Fig. 4. Glucose dynamic in air ambience and in MAP packed sherbets during the storage time.

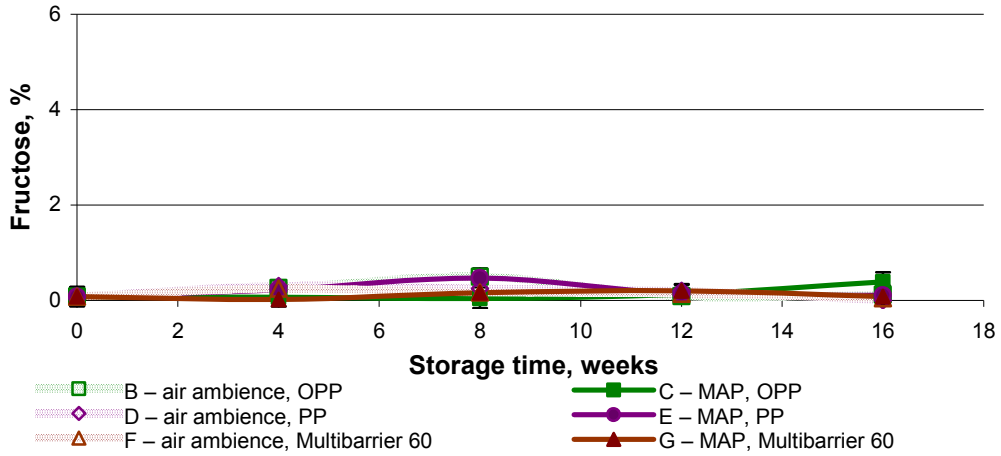


Fig. 5. Fructose dynamic in air ambience and in MAP packed sherbets during the storage time.

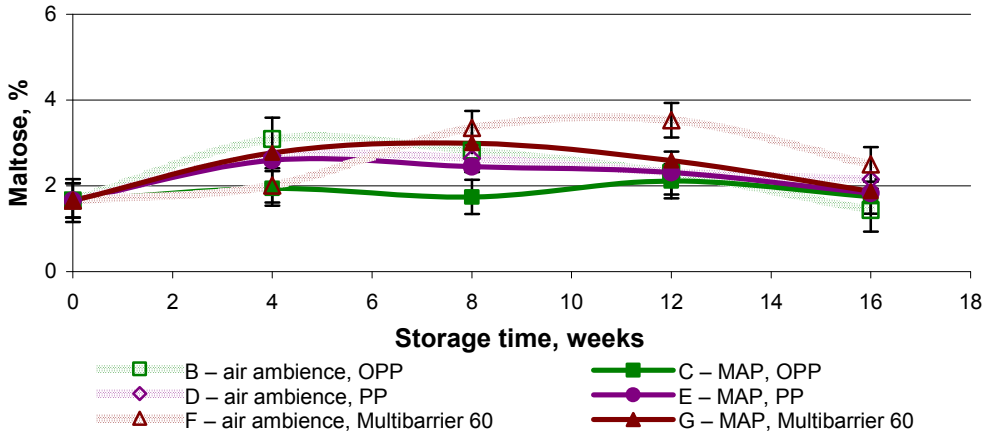


Fig. 6. Maltose dynamic in air ambience and in MAP packed sherbets during the storage time.

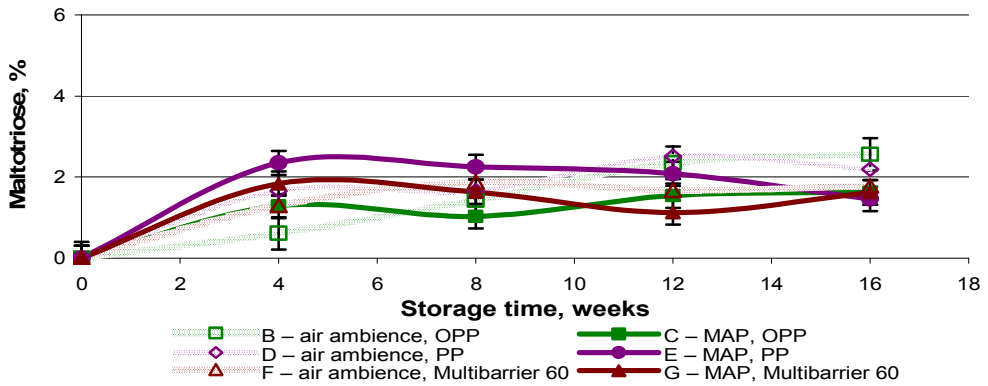


Fig. 7. Maltotorise dynamic in air ambience and in MAP packed sherbets during the storage time.

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Glucose, fructose, maltose maltotriose, melecitose and turanose was minor sugars in sherbet (less than 4%) (Figs 4; 6; 7 and 9). Fructose and melecitose were found in a relatively small quantity and were less than 1%. (Figs 5 and 8). The changes of those sugars was not significant during the all storage time ($p>0.05$).

The monitoring of the changes of maltotorise can be seen in Fig.7. The maltotorise was not found in the fresh prepared sherbet samples, however content increased till 1.8–2.2% during the 16 storage weeks, though this increase is not significant ($p>0.05$).

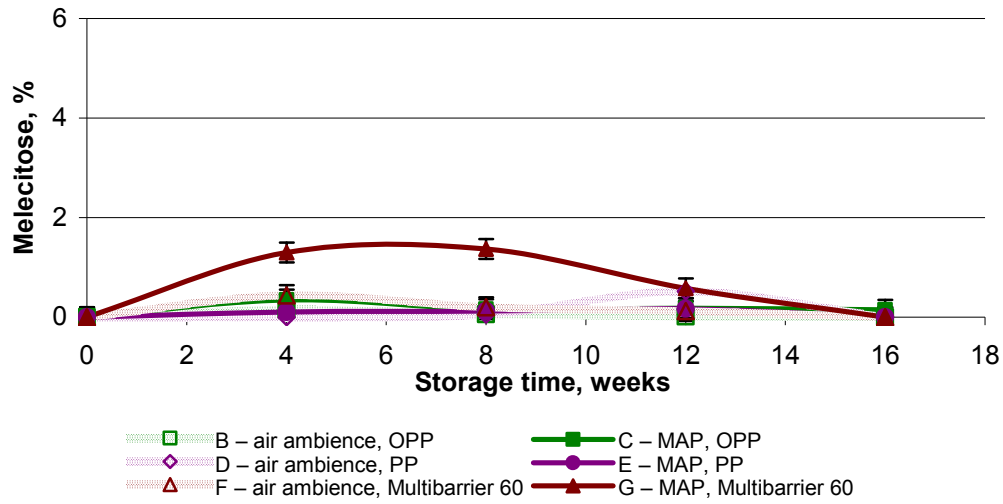


Fig. 8. Melecitose dynamics in air ambience and in MAP packed sherbets during the storage time.

The melecitose of analyzed samples B, C, D, E, F does not change at the storage time (Figure 8), however certain changes of melecitose was observed in sherbet samples packed in Multibarrier 60 film in 100% CO_2 ambience (sample G) within storage period from 4 to 10 weeks, during the storage time up to 16 weeks there was not found any changes in melecitose content.

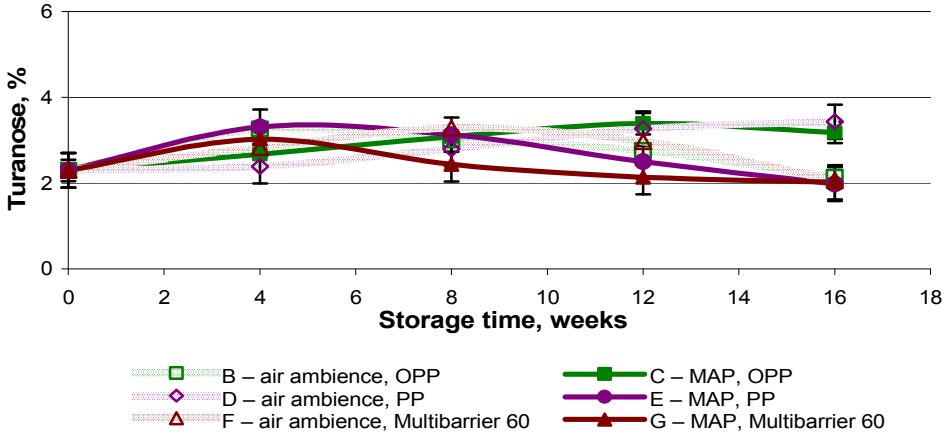


Fig. 9. Turanose dynamics in air ambience and in MAP packed sherbets during the storage time.

The dynamic of the turanose can be seen in Fig.9. Significant differences in turanose during 16 weeks storage among all sherbet samples (B, C, D, E, F and G) packed in three types of polymer materials in 100% CO_2 (MAP) and air ambience was not found ($p>0.05$) – after 16 storage weeks they was till 2.0±0.2–3.5±0.2%

4. CONCLUSIONS

- Cutting force of the sherbet samples packed in air ambience in cardboard boxes (control sample – A) during 16 weeks of storage increased and it differs significantly ($p<0.05$). From those packed in all other types of polymer pouches in air and 100% CO_2 ambience.
- Significant differences in cutting force among all sherbet samples (B, C, D, E, F and G) packed in three types of polymer materials (OPP, Multibarrier 60 and PP) in 100% CO_2 (MAP) and air ambience during 16 weeks of storage was not found ($p>0.05$).
- In this study was found, that sucrose is the most important component of sugars (53.5±2.0%) in ***sherbet*** with crunchy ***peanut*** chips.
- Significant differences in sugars during 16 weeks storage among all sherbet samples (B, C, D, E, F and G) packed in three types of polymer materials in 100% CO_2 (MAP) and air ambience was not found ($p>0.05$) However, the observed upward trend of the sugars in the sherbet during storage time.

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