

# Influence of Active Packaging on the Quality of Pumpkin - Rowanberry Marmalade Candies

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**Abstract**—Experiments with pumpkin-rowanberry marmalade candies were carried out at the Faculty of Food Technology of the Latvia University of Agriculture. The objective of this investigation was to evaluate the quality changes of pumpkin-rowanberry marmalade candies packed in different packaging materials during the storage of 15 weeks, and to find the most suitable packaging material for prolongation of low sugar marmalade candies shelf-life. An active packaging in combination with modified atmosphere (MAP, CO<sub>2</sub> 100%) was examined and compared with traditional packaging in air ambiance. Polymer Multibarrier 60 and paper bags were used. Influence of iron based oxygen absorber in sachets of 500 cc obtained from Mitsubishi Gas Chemical Europe Ageless® on the marmalade candies' quality was tested during shelf life. Samples of 80±5 g were packaged in polymer pouches (110 mm x 110 mm), hermetically sealed by *MULTIVAC C300* vacuum chamber machine, and stored in a room temperature +21±0.5 °C. The physicochemical properties –moisture content, hardness, a<sub>w</sub>, pH, changes of atmosphere content (CO<sub>2</sub> and O<sub>2</sub>), ascorbic acid, total carotenoids, total phenols in headspace of packs, and microbial conditions were analysed before packaging and in the 1<sup>st</sup>, 3<sup>rd</sup>, 5<sup>th</sup>, 8<sup>th</sup>, 11<sup>th</sup> and 15<sup>th</sup> weeks of storage.

**Keywords**—Active packaging, marmalade candies, shelf life

## I. INTRODUCTION

**H**ISTORICALLY, marmalades, jams and jellies may have originated as an early effort to preserve fruit for consumption in the off-season. Processing of different fruits into juice, marmalade or jam has been important for ensuring fruit availability during all year.

This research has been prepared within the framework of the Project “Formation of the research group in food science” Contract Nr.2009/0232/1DP/1.1.1.2.0/09/ APIA/VIAA/122.

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Jellies, jams and marmalades are primarily classified according to the form from which their fruit is incorporated.

Marmalades are mostly jellies from fruit purée and sugar-acid-pectin gel or low-methoxyl pectin-calcium gels. [1],[2]. Definition “fruit marmalades” for human consumption is specified in the Codex Alimentarius International food standards. According the definition, non-citrus marmalade is the product prepared by cooking fruit, whole, in pieces, or crushed adding foodstuffs with sweetening properties to obtain a semi liquid or thick liquid. The product shall be produced such that the quantity of fruit ingredient used as a percentage of the finished product shall not be less than 30% [3].

Numerous snack products and pastries are prepared with a variety of jams and jellies, and thus their quality is a very important factor for the quality of many other products [1], [4].

Consumers are demanding high-quality healthy products in various innovative forms and for competitive prices [5]–[8]. Therefore it is very important to choose raw material with high content of bioactive compounds. There is still a lot of potential to improve the product quality using new, non-traditional fruits and vegetables with high contents of bioactive compounds as raw materials for production of marmalades, jams and jellies. Such raw materials are also pumpkins and sweet rowanberries.

Pumpkin is vegetable that meet the requirements of healthy nutrition. It is a tasty and valuable vegetable crop, contains a lot of biologically active substances and is distinguished for its dietary qualities. There are three common types of pumpkin world-wide, namely *Cucurbita pepo*, *Cucurbita maxima* and *Cucurbita moschata* [9]. Pumpkins provide a valuable source of carotenoids and ascorbic acid, which have major roles in nutrition as provitamin A and as an antioxidant, respectively [10]. The main carotenoids, which are present in winter squash (*Cucurbita maxima*) are  $\alpha$ -carotene and  $\beta$ -carotene. Winter squash fruit contains 0.4-7.5 mg·100 g<sup>-1</sup> of  $\alpha$ -carotene and 1.4-8.4 mg·100 g<sup>-1</sup> of  $\beta$ -carotene, depending on cultivar [11].

*Cucurbita moschata* is used for traditional medicine in several countries to control diabetes as well as for treating worms and parasites [12]. They are consumed in various ways such as fresh or cooked vegetable, as well as being stored frozen or canned [13].

The rowanberries (*Sorbus*) belong to the subfamily *Maloideae* of the family *Rosaceae* and their berries have been promoted as a health-food and can be a source of the health-promoting components. The wild rowanberries in Latvia are picked in autumn and they are eatable, but though they have been traditionally used for jellies and jams, their wider use as food ingredients has been less popular because of their bitter taste [14], [15]. Different cultivars of sweet rowanberries and hybrids with other *Rosaceae* genus and species are sweeter and less astringent than wild rowanberries.

The hybrid cultivars were developed by cross-breeding rowan with *Malus*, *Pyrus*, *Aronia* and *Mespilus* [14], [15]. Sweet rowanberry *S. aucuparia* L. var. *edulis* contains 1600-2420 mg of organic acids per 100 g of edible portion, including in average 10 mg of parasorbic acid and 98 mg of vitamin C per 100 g, and 2.5 mg of total carotenoids per 100 g.

The quality of the product is complex factor that includes different parameters, such as biochemical composition of product, physical, microbiological, sensory parameters and others.

The stability of many bioactive compounds is dependent on pre-treatment of the raw material, processing operations of the product, and storage conditions. In many fruits carotenoids and flavonoids, which are located predominantly in epidermal tissues, are removed by peeling operations, which can greatly reduce concentrations of bioactive compounds in processed products. Removal of seeds can result in losses of phenolics (e.g. ellagitannins) [16]. High temperature also causes the loss of the important nutrients. In the temperature just above 50°C degradation of several phenolic compounds occur [17], but at 60-63°C anthocyanin degradation begins [18], [19] and fruit tissue is damaged [19]. During the thermal processing 50-70%-losses of ascorbic acid can occur. The losses of this vitamin are used as an indicator of food quality. On the other hand, carotenoids after thermal processing are more easily extracted from plant tissues due to tissue softening and destruction of the membrane-protein complex [16].

Colour and texture are very important quality parameters of marmalades. Colour is one of the most important parameters to which consumers are sensitive when selecting foods. Colour stability of fruit and fruit products is influenced by many factors. At least three factors can cause the colour deterioration: the loss of red anthocyanin pigment, formation of brown pigments, and discoloration through various factors such as heavy metal contaminations. The fruit cultivar, temperature, pH, presence of oxygen and time of processing were found to exert a great influence on colour stability of fruit products [20], [21].

A shelf life of a product can be altered by changing its composition and form, the environment to which it is exposed, or the packaging system [22]. Nowadays microbiological parameters are very meaningful due to development of product manufacturing [23]. The transport of moisture into or from food materials is an important factor in controlling food quality, chemical reactions and microbial growth during storage [24].

Packaging has a significant role in the food supply chain [25]. Optimal packaging solutions could prevent or minimize quality changes, resulting in increased shelf life as well as quality and safety maintenance [26]. The main resource used for food packaging, including sweets, is plastic materials [27].

The effective packaging must prevent the transmission of oxygen, light and water vapour, and microbial growth to retard quality deterioration of packaged goods [28]. Nowadays, perspective methods to extend the shelf life of food products are vacuum packaging and packaging in the protective gas mixture or modified atmosphere (MAP) [29].

However, vacuum packaging and MAP do not always remove oxygen completely. Moreover, the O<sub>2</sub> that penetrates through the packaging film cannot be removed by those technologies.

In order to optimize product and packaging compatibility, materials with improved barrier properties should be used. Optimization may include new areas such as active packaging concepts, and nanocomposite technology [26]. As all packaging materials are permeable to moisture to some extent, active packaging can balance moisture and compensate for moisture loss, suggests Roberto Sablo from Multisorb Technologies [30]. Active packaging includes concepts that will absorb oxygen, moisture or remove compounds that may cause taints. Other systems of active packaging release antimicrobial agents, antioxidants, flavours and/or colours [31].

The objective of this investigation was to evaluate the quality changes of pumpkin-rowanberry marmalade candies packed in different packaging materials during the storage of 15 weeks, and to find the most suitable packaging material for prolongation of low sugar marmalade candies shelf-life.

## II. MATERIALS AND METHODS

### A. Experimental design

Experiments were carried out at the Department of Food Technology, Latvia University of Agriculture in 2011. The object of the research was pumpkin-rowanberry marmalade candies from pumpkin (*Cucurbita maxima*) and sweet rowanberry (cultivar 'Nevezhinskaya') purees. Rowanberry puree was prepared from frozen and thawed rowanberries which were scrubbed through sieve, and the pumpkin puree, which was made from cut pieces of pumpkin, boiled (in 100 °C) and scrubbed through sieve. The obtained rowanberry-pumpkin mass was mixed with sugar (sucrose 25% from the total amount of product), heated to 85 °C, and supplemented with 4.5% pectin (Gen pectin LM-104-AS powder mixed with sugar).

After it the mass was filled in polymer forms (65 mm x 45 mm x 20 mm; mass 80±5g), than left for hardening, after which it was took out of forms and stored three days at room temperature (+21±0.5 °C) for ripening and thickening..

### B. Packaging and storage of samples

The study involved packaging of marmalade candy pieces in unhermetical ready made paper bags, biodegradable polymer VC999 BioPack lidding film PLA pouches with barrier properties, and pouches made from Multibarrier 60 polymer film. For shelf life extension the use of both usual MAP conditions as well as MAP with oxygen scavenger commitment in the pouch was investigated.

Modified atmosphere of 100% carbon dioxide (CO<sub>2</sub> (E 290)) was used. For reduced oxygen packaging (ROP) creation (O<sub>2</sub> – 0%) in pouches an iron based oxygen scavenger sachets of 500 cc obtained from Mitsubishi Gas Chemical Europe Ageless® were used. The samples packed in polymer pouches were hermetically sealed by MULTIVAC C300 vacuum chamber machine and stored at room temperature +20.0±2 °C, (controlled by MINILog Gresinger electronic) and about 40% RH for 15 weeks under day and night conditions.

Characteristics of the used packaging materials are shown in the Table I and structure of performed experiments – in Fig. 1. The materials for experiments were selected with different water vapour transmutation rate and various thicknesses in order to assess whether hardening can be ascribed by water loss from marmalade candy samples or from redistribution of the moisture inside the product, or by combination of moisture loss and redistribution.

TABLE I  
CHARACTERISTICS OF USED MATERIALS IN EXPERIMENTS

Nr.	Packaging material	Composition	Thickness, $\mu\text{m}$
1.	Paper bag	Double layer, brown colour	130±2
2.	Multibarrier 60 film	Laminate, APA/TIE/PA/EVOH/PA/TIE/PE/PE, transparent	60±2
3.	VC999 BioPack lidding film	Single layer, transparent PLA coated with SiOx	50±2

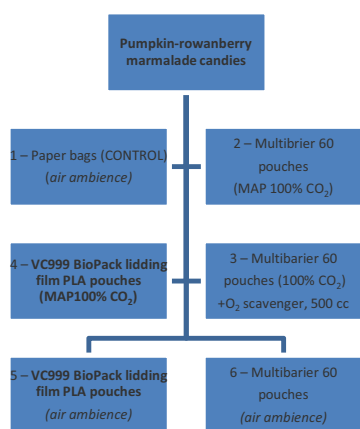


Fig. 1 Structure of performed experiments

Samples (5) and (6) (Fig. 1), which were packed both in VC999 BioPack lidding film PLA (100% CO<sub>2</sub>) and Multibarrier 60 film pouches in air ambience grow mouldy after 5 to 6 days storage, therefore those samples were not used for further experiments.

One piece of the pumpkin-rowanberry marmalade candies was placed in each pouch. Size of 110 mm x 110 mm, the product mass in each package – 80±5.0 g. Two packages for each treatment were randomly selected on sampling days (day 0) and after 1, 3, 5, 8, 11 and 15 storage weeks; several (depending on the analyse) measurement repetitions of each sample were performed. The results were reported as averages of the measurements.

### C. Physical, chemical, and microbial analysis

The following mechanical and physical characteristics were analyzed:

- *The dynamics of gas composition* in a hermetically sealed pouch headspace at the storage time was measured as a percentage of oxygen and carbon dioxide by the gas analyser OXYBABY® V O<sub>2</sub>/CO<sub>2</sub>.

- *Moisture content* was determined using standard method ISO 6496:1999 by verified balance KERN (Germany) with

precision ±0.001g; mass loss calculation (%) were determined by weighing packed samples on the electronic scales, by standard LVS ISO 1442: 1997.

- *Hardness* analyses (cutting force in N) of marmalade candies were determined on the Texture Analyser, “TA.XT.plus Texture Analyser” (Stable Micro Systems Ltd., Surrey, UK) and the measuring probe A/BC (butter cutter, can be used for soft samples, supplied in association with the Texture Analyser). The system was equipped with compression cell of 50 kg and software Texture Exponent 32. Cutting force was determined in six marmalade candy samples; thickness of each sample was 2 cm. Each piece of marmalade candy sample for cutting was placed centrally under the cutter edge. Hardness was measured as the maximum penetration force (N) reached during breakage of the sample. The measuring parameters were: pre-test speed 1 mm s<sup>-1</sup>; test speed 1 mm s<sup>-1</sup>; post-test speed 10 mm s<sup>-1</sup>; cutting distance: 13 mm pressing into the sample. The measurement is triggered automatically at 0.09807 N. The samples were cut partly through, in order to check the differences of structural characteristics. Plotting force (in N) versus storage time (in weeks), and the hardness changes during storage of marmalade candy samples stored in different packaging materials was calculated. The maximum cutting force (N) was used as an index for the cutting test. The maximum force required for sample compression was calculated as an average of 10 measurements.

- *pH* was measured by JENWAY 3510 pH-meter, standard method LVS ISO 5542:2010.

- *Water activity* was determined by standard method ISO 21807:2004, using AquaLab LITE device.

The content of **ascorbic acid** was determined by titration with 0.05-M iodine solution [32]. The marmalade sample (25 g) were poured with 6-% solution of oxalic acid and homogenized. Then the sample was filtered. 2 ml of 1-% solution of starch was added to 10 ml of filtrate and the filtrate was titrated until the colour changed which does not disappeared during a 30 sec interval. For standard solution of ascorbic acid 20 mg of ascorbic acid were dissolved in 100 ml of the oxalic acid solution. Two ml of the starch solution was added to 25 ml of the standard-solution and the mixture was titrated. The content of vitamin C (ascorbic acid) mg per 100g of the product dry matter was calculated with the following equation (2):

$$C = 5000 \cdot \frac{V_{\text{sample}}}{m \cdot V_{\text{standard}}}, \quad (2)$$

where  $V_{\text{sample}}$  – volume of the iodine solution titrated in a sample, ml;

$V_{\text{standard}}$  – volume of the iodine solution titrated in a standard solution, ml.

$m$  – the amount of sample, g

*Carotenoids* were analyzed by spectrophotometric method (used the UV/VIS spectrophotometer Jenway 6705) at 440 nm [33]. A sample of 2g of homogenized marmalade sample was placed in 100 ml conic flask and 20 ml of 96% ethanol was

added. The sample was stirred on a magnetic stirrer for 15 min then 25 ml of petrol ether were added and stirring was continued for one more hour. After 3–4 hours when both layers were completely divided, the top yellow layer was used for further detection of carotenoids at 440 nm. Carotene equivalent (KE) was found, using a graduation curve with  $K_2Cr_2O_7$ . The content of carotenoids ( $mg\ 100g^{-1}$ ) was calculated by equation (3):

$$X = \frac{12.5 \cdot 100 \cdot KE}{36 \cdot a}, \quad (3)$$

where 12.5 and 36 coefficients for relationship between  $K_2Cr_2O_7$  and carotenoids;

KE – carotene equivalent by graduation curve;

a – sample weight, g.

Total phenol content was determined by photometric method with Folin-Ciocalteu reagent [34]. A sample of 4–6 g homogenized berries were placed in 250 ml conic flask and 5 ml distilled water was added. The prepared sample was mechanically shaken for 5 minutes. Then 50 ml 96% ethanol was added and solution was continuously shaken by 2 hours. After shaking, the sample was filtered, and residues were washed three times with 10 ml of 80% ethanol. The filtrate was placed in a 100 ml flask and filled with water. For analyses of phenols the Folin-Ciocalteu reagent and 4 ml 7.5% sodium carbonate was used. After 30 minutes the samples were analyzed using the spectrophotometer Jenway 6705 at wave length 765 nm. As a control, solution 1 ml water with 5 ml Folin-Ciocalteu reagent and 4 ml 7.5% sodium carbonate solution were used. The concentrations of phenols was found out from calibration diagram which was drawn by measuring absorbance of different concentrations of gallic acid and the content of phenols is calculated with formula (4):

$$X = \frac{C}{a \cdot 10}, \quad (4)$$

where X – concentration of phenols,  $mg\ 100\ g^{-1}$ ; C – absorption at 765 nm; a – the amount of analyzed sample, g.

#### E. 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. Two-way analyses of variance ( $p \leq 0.05$ ) were used to determine significance of differences between weight losses, moisture content, hardness,  $a_w$ , pH, colour changes of atmosphere content ( $CO_2$  and  $O_2$ ) in headspace of packs, and microbial conditions by different packed samples.

### III. RESULTS AND DISCUSSION

The carbon dioxide ( $CO_2$ ) content during the storage of 15 weeks differed significantly ( $p < 0.05$ ) among all pumpkin-rownberry marmalade candy samples packed in the different kinds of materials, (Fig. 2), and the results were similar to experiments done with apple- blackcurrant marmalade candies [35].

The carbon dioxide content during the first 8 weeks of storage in Multibarrier 60 pouches without oxygen scavenger (sample 2) were stable (100%), but at the 11<sup>th</sup> week the  $CO_2$  content began to decrease gradually and after 15 weeks the pouches collapsed and a vacuum was formed (Fig.2). In Multibarrier 60 pouches (MAP, 100%  $CO_2$ ) with incorporated  $O_2$  scavenger, 500 cc the  $CO_2$  content began to decrease after 5 weeks of storage and in 8<sup>th</sup> storage week reached 0% and the vacuum remained in pouches until the end of experiment. In VC999 BioPack lidding film PLA pouches (MAP, 100%  $CO_2$ ) the  $CO_2$  began to decrease already after one week of storage, decreasing to 89% and after 5 weeks of storage the decrease was notable, but at the 8<sup>th</sup> storage week vacuum was established in pouches and the packs lose their market appearance. This phenomenon can be explained with carbon dioxide dissolving in the marmalade candies having quite high moisture content (39.73-43.35%). As a result, the pressure decreased in the packages and destroyed them. The solubility of  $CO_2$ , as we can see in the Fig. 3, was related with simultaneous decrease of oxygen content in pouches. The concentration of carbon dioxide in paper bag was not more than  $1.0 \pm 0.1\%$ .

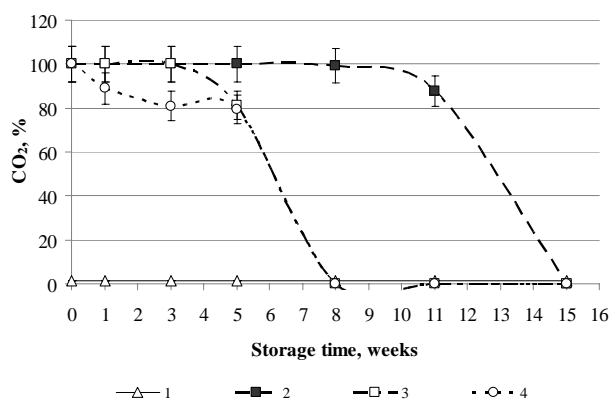


Fig. 2 The dynamics of carbon dioxide ( $CO_2$ ) content in the headspace of package in MAP (100%)

1 – Paper bags; 2 – Multibarrier 60 pouches (MAP 100%  $CO_2$ ); 3 – Multibarrier 60 pouches (100%  $CO_2$ ) +  $O_2$  scavenger, 500 cc; 4 – VC999 BioPack lidding film PLA pouches (MAP100%  $CO_2$ )

There were significant ( $p < 0.05$ ) differences between packaging materials in content of oxygen inside of packages during the storage time. The oxygen content ( $O_2$ ) in Multibarrier 60 packages without  $O_2$  scavenger during 11 storage weeks increased from zero to  $7.3 \pm 0.6\%$  in average, than decrease of  $O_2$  began and during 15 storage weeks reached 0% (Fig. 3). In the Multibarrier 60 packages with oxygen scavenger, the  $O_2$  content increased only in average to  $2.25 \pm 0.18\%$  during 5 storage weeks; after that it started to decrease, and already before 8 storage weeks the vacuum was developed in the packages (Fig. 3).

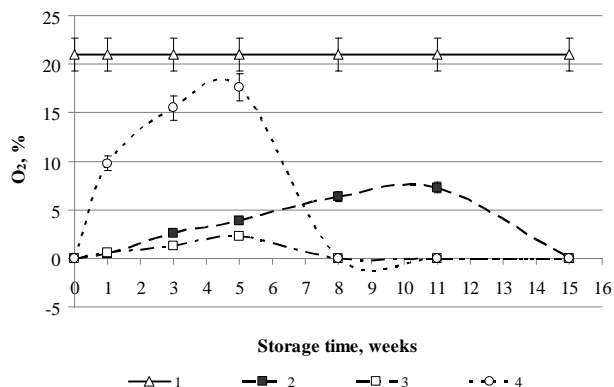


Fig. 3 The dynamics of oxygen (O<sub>2</sub>) content in the headspace of package in MAP (100%)

1 – paper bags; 2 – Multibarrier 60 pouches (MAP 100% CO<sub>2</sub>); 3 – Multibarrier 60 pouches (100% CO<sub>2</sub>) + O<sub>2</sub> scavenger, 500 cc; 4 – VC999 BioPack lidding film PLA pouches (MAP100% CO<sub>2</sub>)

These results prove the influence of oxygen scavenger for minimizing the oxygen content in packages. The O<sub>2</sub> content in packages of VC999 BioPack lidding film PLA rapidly increased to 17.6±1.4% during the first five storage weeks, but at the 8<sup>th</sup> week of storage the vacuum was developed in the packages. Thus we can summarize that after 15 weeks of storage the vacuum was formed in all pouches from investigated polymer materials, consequently, the O<sub>2</sub> content did not differ in these samples. The O<sub>2</sub> contents in the paper bags during all the experiment period were similar as it was in the surrounding environment (about 21%).

Initial moisture content of marmalade candies was 39.73±0.09%. As we can see in the Fig. 4, moisture content increased to 43.08-46.35% in all polymer materials during first three weeks of storage, and in samples packed in Multibarrier 60 with and without oxygen scavengers the moisture content remained between 42.7-47.3% until the end of the experiment. In VC999 BioPack lidding film PLA after 5-weeks storage it decreased again to 39.58±0.08%, and remained almost the same until the 15<sup>th</sup> week of storage. In samples packed in paper bags the moisture content differed (p<0.05) from those packed in polymer packaging materials and was decreased to 18.2% during the first week of experiment and did not change notably during the rest of the experiment time. Interestingly that moisture content in the pumpkin-rowanberry samples packed in paper bags remained much higher than it was in apple-blackcurrant samples [35], which could be explained with differences of samples chemical composition and some structure properties.

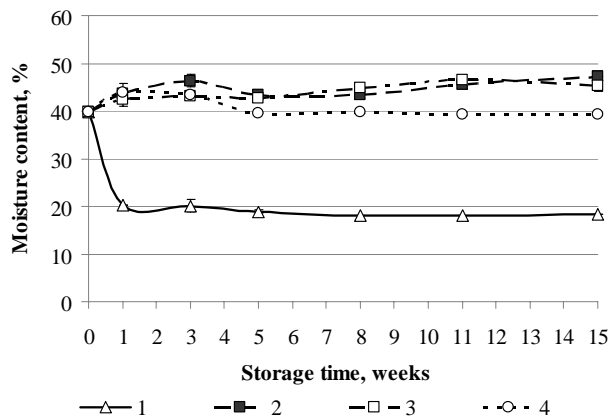


Fig. 4 The dynamics of moisture content in pumpkin-rowanberry marmalade candy samples during storage

1 – paper bags; 2 – Multibarrier 60 pouches (MAP 100% CO<sub>2</sub>); 3 – Multibarrier 60 pouches (100% CO<sub>2</sub>) + O<sub>2</sub> scavenger, 500 cc; 4 – VC999 BioPack lidding film PLA pouches (MAP100% CO<sub>2</sub>)

The ascorbic acid content rapidly decreased in the first week of storage in all samples: from 19.5 mg 100g<sup>-1</sup> of dry weight (similarly to many fresh fruits, like raspberries, apples, etc. [36]) at the beginning of the experiment until in average 7.66 mg 100g<sup>-1</sup> of dry weight after one week of storage (Fig. 5). The lowest ascorbic acid content after 3 weeks of storage was in the samples packed in the paper bags. The ascorbic acid content in the other samples was similar to each other and continued to decrease slowly during the next weeks of storage, reaching 5.48 mg 100g<sup>-1</sup> of dry weight at the 11<sup>th</sup> week of storage. Therefore it could be concluded that none of the evaluated packaging materials can prevent the oxidation process of ascorbic acid.

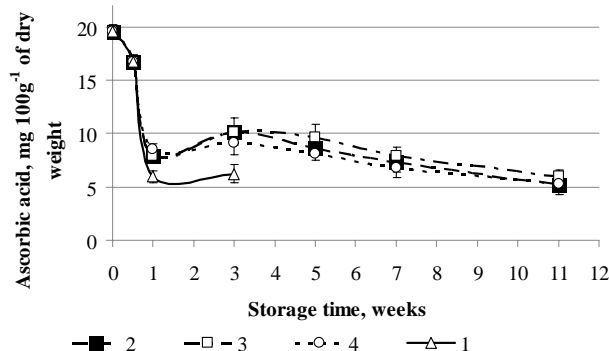


Fig. 5 The ascorbic acid content (in dry weight of the product) in pumpkin-rowanberry marmalade candy samples during storage

1 – paper bags; 2 – Multibarrier 60 pouches (MAP 100% CO<sub>2</sub>); 3 – Multibarrier 60 pouches (100% CO<sub>2</sub>) + O<sub>2</sub> scavenger, 500 cc; 4 – VC999 BioPack lidding film PLA pouches (MAP100% CO<sub>2</sub>)

The total content of carotenoids at the beginning of the experiment in the pumpkin-rowanberry marmalade candies was 3.14 mg 100g<sup>-1</sup> of dry weight. These data are comparable with the content of carotenoids in many fresh fruits and vegetables [36], [37].

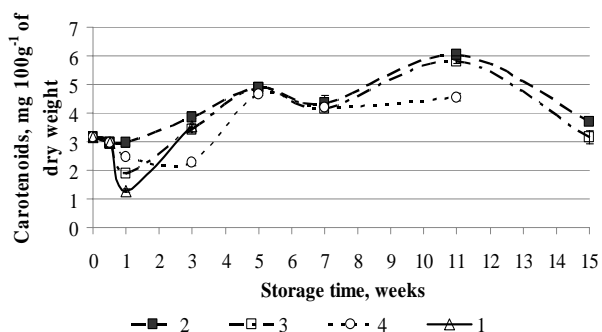


Fig. 6 The total carotenoids (in dry weight of the product) in the pumpkin-rowanberry marmalade candy samples during storage 1 – paper bags; 2 – Multibarrier 60 pouches (MAP 100% CO<sub>2</sub>); 3 – Multibarrier 60 pouches (100% CO<sub>2</sub>) + O<sub>2</sub> scavenger, 500 cc; 4 – VC999 BioPack lidding film PLA pouches (MAP100% CO<sub>2</sub>).

After the 15 weeks of storage the total content of carotenoids in the samples did not decrease below the values at the beginning of the experiment and was between 3.15 and 3.68 mg 100g<sup>-1</sup> of dry weight (Fig. 6). Therefore we can conclude that the storage of the samples in the polymer materials doesn't cause decrease of the content of total carotenoids.

The total content of phenols in the samples at the beginning of the experiment was 110.27 mg 100g<sup>-1</sup> of dry weight, consequently, fresh marmalades is a good source of the phenols, being one the most important bioactive compounds in the fruits and berries [36]. Already after one week of storage there were some significant differences between samples packed in different materials. The highest content of phenols were detected in samples packed in the Multibarrier 60 pouches (MAP 100% CO<sub>2</sub>), but the lowest in paper bags (fig. 7). Fast decrease of the phenols occurred between the 5<sup>th</sup> and 7<sup>th</sup> week of the storage. At the 15<sup>th</sup> week of storage, the phenol content ranged from 79.29 to 90.73 mg 100g<sup>-1</sup> of dry weight. The highest content of phenols was in the samples packed in the Multibarrier 60 pouches (100% CO<sub>2</sub>) + O<sub>2</sub> scavenger.

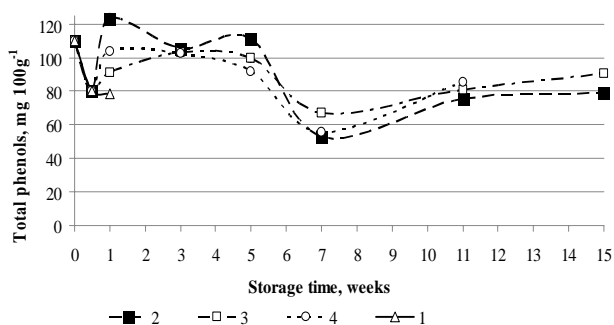


Fig. 7 The total content of phenols (in dry weight of the product) in pumpkin-rowanberry marmalade candy samples during storage 1 – paper bags; 2 – Multibarrier 60 pouches (MAP 100% CO<sub>2</sub>); 3 – Multibarrier 60 pouches (100% CO<sub>2</sub>) + O<sub>2</sub> scavenger, 500 cc; 4 – VC999 BioPack lidding film PLA pouches (MAP100% CO<sub>2</sub>).

The hardness of the marmalade candies was highly dependent of the used packaging material and technology. Presumably, the major hardening reason can be water vapour migration through the packaging material as well as some biochemical and chemical processes during the storage, which induce hardening or de-hardening. The initial cutting force of all samples was 1.06±0.03 N. Mouth feel of all tested samples, excepting sample packed in paper bags, during the storage time was acceptable. Candies packed in the transparent Multibarrier 60 film with and without incorporated oxygen scavenger showed insignificant increase of the cutting force of packed product after 15 storage weeks (1.57±0.05 N). At the same time, samples packed in the VC999 BioPack lidding film PLA increased their hardness at the end of their storage in the 11<sup>th</sup> week higher – 2.31±0.11 N (fig. 8).

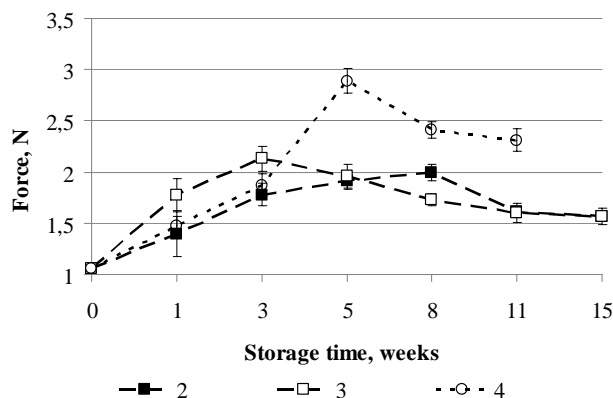


Fig. 8 The dynamics of hardness changes of the pumpkin-rowanberry marmalade candies during the storage in the polymer materials 2 – Multibarrier 60 pouches (MAP 100% CO<sub>2</sub>); 3 – Multibarrier 60 pouches (100% CO<sub>2</sub>) + O<sub>2</sub> scavenger, 500 cc; 4 – VC999 BioPack lidding film PLA pouches (MAP100% CO<sub>2</sub>).

After first three to five weeks of storage, the cutting force of the samples packed in the films continued to increase, but later it again decreased. It could be explained with carbon dioxide dissolving into the product, which could make it softer, because rapid decrease of CO<sub>2</sub> in the headspace of the packages at the same time was detected (fig. 2).

The increase of the cutting force of the marmalade candies packed in the paper bags was very high compared to the samples packed in the other materials – from 1.06±0.03 N at the beginning of the experiment rising up to 129.26±10.44 N at the end (fig. 9), the maximal cutting force substantially differed (p<0.05) from all other tested samples, and the candies after 1 to 2 weeks storage were not suitable for eating.

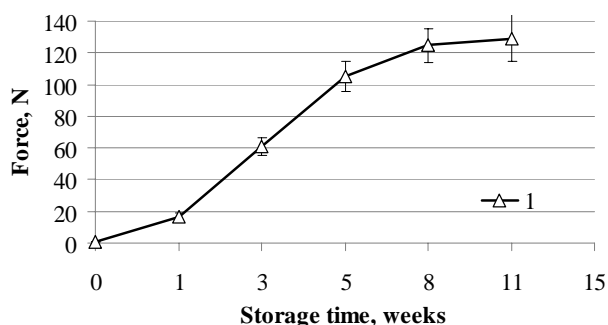


Fig. 9 The dynamics of hardness changes of the pumpkin-rowanberry marmalade candies during storage in the paper bag packaging

The initial pH of the pumpkin-rowanberry marmalade candies was  $3.56 \pm 0.006$ . Slight decrease of pH value before 5<sup>th</sup> week of storage occurred in all samples, while next 10 weeks it remained stable and at the end of storage pH was 3.28-3.44 (Fig. 10).

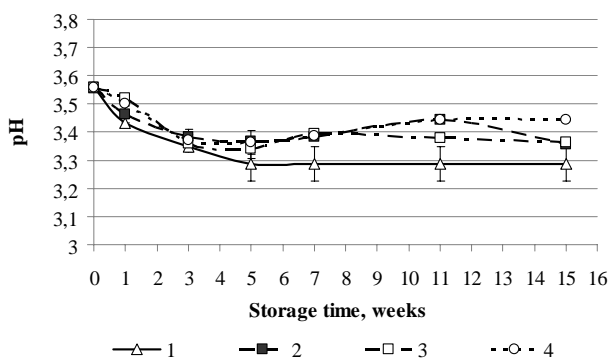


Fig. 10 The dynamics of pH value of the pumpkin-rowanberry marmalade candies during storage

1 – paper bags; 2 – Multibarrier 60 pouches (MAP 100% CO<sub>2</sub>); 3 – Multibarrier 60 pouches (100% CO<sub>2</sub>) + O<sub>2</sub> scavenger, 500 cc; 4 – VC999 BioPack lidding film PLA pouches (MAP100% CO<sub>2</sub>).

The value of water activity ( $a_w$ ) of the freshly prepared pumpkin-rowanberry marmalade candies was 0.871 (Fig. 11). After 15 storage weeks  $a_w$  of marmalade samples packed in the Multibarrier 60 without and with incorporated oxygen scavenger, as well as in the VC999 BioPack lidding film PLA did not change significantly ( $p > 0.05$ ). These marmalade candies had lower  $a_w$  (and also water content) than previously described apple-blackcurrant marmalade candies ( $a_w = 0.927$ ) [35]. The water activity of the candies packed in the paper bags decreased from 0.871 to 0.5 during the same storage time, accordingly with reduction of moisture content from  $39.73 \pm 0.09\%$  to 18.2% (Fig. 4).

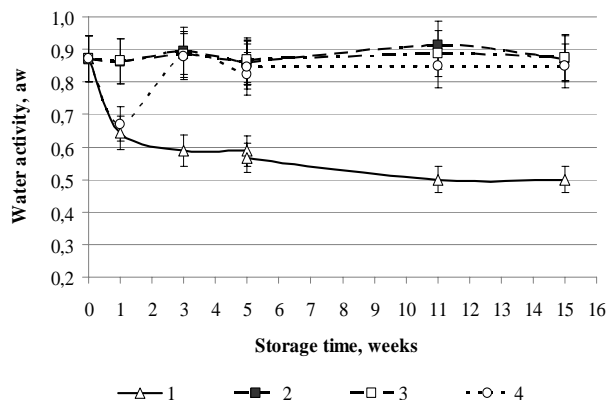


Fig. 11 The dynamics of the pumpkin-rowanberry marmalade candies water activity ( $a_w$ ) during storage time

1 – paper bags; 2 – Multibarrier 60 pouches (MAP 100% CO<sub>2</sub>); 3 – Multibarrier 60 pouches (100% CO<sub>2</sub>) + O<sub>2</sub> scavenger, 500 cc; 4 – VC999 BioPack lidding film PLA pouches (MAP100% CO<sub>2</sub>).

#### IV. CONCLUSION

The investigated conventional Multibarrier 60 film is applicable for pumpkin-rowanberry marmalade candy's long-term storage maintaining quality, and incorporation of the oxygen scavenger in the Multibarrier 60 pouches (MAP, 100% CO<sub>2</sub>) can be recommended as the best variant. Packaging in the biodegradable VC999 BioPack lidding film PLA can maintain quality of pumpkin-rowanberry marmalade candies up to 8 weeks. The paper bags could be used only for candy's short-term packaging on the supermarket shelves.

The bioactive compounds, like carotenoids and phenols are quite stable during the storage of packed marmalade candies at room temperature, while the ascorbic acid content decrease faster - already after one week of storage it decreased more than two times.

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