Influence of Active Packaging on the Shelf Life of Apple-Black Currant Marmalade Candies

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Abstract—The research object was apple-black currant marmalade candies. Experiments were carried out at the Faculty of Food Technology of the Latvia University of Agriculture. An active packaging in combination with modified atmosphere (MAP, CO₂ 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® was tested on the quality during the shelf of marmalade. 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 room temperature +20.0±1.0 °C. The physiochemical characteristics such as physiochemical properties, moisture content, hardness, pH, colour, changes of atmosphere content (CO₂ and O₂) in headspace of packs, and microbial conditions were analysed before packaging and in the 1st, 3rd, 5th, 8th, 11th and 15th weeks of storage.

Keywords—Active packaging, marmalade candies, shelf life

I. INTRODUCTION

FOOD choice is a complex function of sensory characteristics (taste, odour and texture), and non sensory characteristics such as physiochemical properties, familiarity, food-related expectations, attitudes and health claims [1], [2]. Consumers today demand high-quality products in various innovative forms and for competitive prices [3]–[7].

Sweet foods are an important part of birthdays, holidays and family traditions. For most of us, however, indulging in sweets must be done with care. More than half of developed countries adults are overweight or obese [8].

Historically, jams and jellies may have originated as an early effort to preserve fruits for consumption in the off-season. Processing of different fruit kinds into juice, marmalade, jam or dried products is important for insuring of fruits during all year. In the same time, it is important for bioconstituents recovery. The importance of state of ripening at harvest is important for its physico-chemical and sensory characteristics (appearance, smell and flavour). As a result of ripening soluble sugar content grows, acidity decreases, modification of fruit texture and conversion of protpoetin to pectin happens. Native pectin in plant cell walls plays a central role in the ripening, texture, and storage qualities of fruits and vegetables. The technological qualities of the products and jam processed thereof, vary with the variety of fruit and its physico-chemical characteristics [7], [9]–[11].

Jams and jellies are products made principally from fruits, but they can also be made from some vegetable materials [12]. Marmalades are basically jellies with fruit purée and sugar-acid-pectin gel or low-methoxyl pectin-calcium gels [7]. Jams, marmalades are a mixture of fruits – whole, in pieces, or pulped; fresh, concentrated, frozen, or canned – sugar and other minor ingredients that help develop texture because of the formation of a gel between sugars and pectin substances along with fruit and vegetable acidity. Sometimes it is necessary to add the last two substances because not all fruits and vegetable have enough acidity or pectin content for gel formation. Numerous snack products and pastries are prepared with a variety of jams and jellies, and thus their quality is a very important factor in the quality [7], [12].

Confectionery (is what many people think of as „candy” or „sweets”) belong to either of two groups, sugar confectionery or chocolate confectionery, although broader classifications sometimes are used. These products contain sugar, syrups, jams, honey, or other sweeteners [13], [14]. The 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 [15]. Nowadays microbiological parameters are very meaningful due to the development of product manufacturing and variety of preservation [16]. According to Kawo and Abdulmumin, sweets receive most of their contamination from their ingredients, although some contaminants may be added by unwrapped pieces by air, dust and handling. Additional contamination may come from equipment coming in contact with food from packaging materials and from personnel. The diffusion of moisture in food materials is of fundamental importance for processing and storage. The transport of moisture into or from food materials is an important factor in controlling food quality, chemical reactions and microbial growth during storage [17]. One way to slow down moisture transport is to use barrier between the domains of a food material. Barrier packaging films protect the candy from air whereas edible films inhibit moisture migration between different moisture domains within a confection [18].
Relative humidity plays an important role in food product development, storage and packaging. Water activity \( a_w \) is the intrinsic product characteristic that most influences the microbial ecology of candies. High concentrations of sugar, especially those of low molecular weight, afford low \( a_w \). Most bacteria cannot grow at \( a_w \) below 0.85, and growth of spoilage-causing yeast and moulds is unlikely at \( a_w \) levels below 0.61 [13, [19].

The diffusion of moisture in food materials is of fundamental importance for processing and storage. The transport of moisture into or from food materials is an important factor in controlling food quality, chemical reactions and microbial growth during storage [18]. When the difference in water activity is large, moisture migration is rapid, although the rate of moisture migration depends on the nature of resistances to water diffusion. Methods to protect confections against moisture migration are continually being studied to preserve quality and extend the shelf life. This includes approaches to retard migration to the environment through use of improved packaging materials and to retard migration within multi-domain candies through the use of edible films and/or reformulation to balance \( a_w \) of the different domains [17].

Packaging is a medium between product manufacturers and consumers [20], packaging has a significant role in the food supply chain [21]. The main functions of packaging are to extend the shelf life, and maintain the quality and safety of the packed goods. An effective packaging must prevent the transmission of oxygen, light and water vapour, and microbial growth to retard quality deterioration of packaged goods [22]. Mouth feel, texture and eating qualities are adversely affected by loss of moisture. 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 [23]. 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 [24, [25].

The main resource used for food packaging, including sweets, is plastic materials [26]. The shelf life of foods packed in plastics depends on the permeation of gas and water vapour through the packages. This is because a significant amount of food deterioration results from oxidation and changes in the water content. Polyolefin’s (PE, PS), polystyres (PS), polyethylene terephthalate (PET) and polycarbonates (PC) are the principal families of thermoplastics in food packaging [27]. Those materials need to be examined for their suitability for use as various sweet and candy’s packaging materials.

For the time being the application of vacuum and modified atmosphere packaging technologies appeared successful in extending the shelf-life and quality of the food [28–30]. However, these technologies do not always remove oxygen completely. Moreover the \( O_2 \) 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 [31].

The objective of this work was to evaluate the application of traditional Multibarrier 60 film without and with oxygen scavengers incorporated and VC999 BioPack lidding film PLA, coated with a barrier of pure silicon oxide (SiOx) for apple-black currant marmalade candy’s packaging in modified atmosphere (MAP) consisting of 100% carbon dioxide \( CO_2 \) (E 290), as control selecting air packaging of the marmalade candies in paper bags.

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 apple-black currant marmalade candies. The marmalade candies were made from blackcurrant and apple purees. Blackcurrant puree was made from frozen and thawed blackcurrants which were scrubbed through a sieve, and the apple puree was made from boiled till 100 °C apples cut in pieces, which were scrubbed through a sieve, too. The mass, mixed with sugar (sucrose 25% from the total amount of product), was pasteurized till 85 °C, adding 4.5% pectin (Gen pectin LM-104-AS powder mixed with sugar), after than filled in polymer forms and stored for three days at the room temperature (+2.0±1.0 °C) for ripening and thickening. Dimensions of one piece of marmalade candies on average was (65 mm x 45 mm x 20 mm), mass 80±5g.

B. Packaging and storage of samples

The study involved packaging of marmalade candy pieces in unhermetical ready made paper bags, preliminary from roll stock made 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 with oxygen scavenger commitment in the pouch was investigated. Modified atmosphere consisting of carbon dioxide \( CO_2 \) (E 290) 100% was used. For reduced oxygen packaging (ROP) creation (\( O_2 \) – 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 the room temperature of +20.0±1.0 °C, (controlled by MINILOG Gresinger electronic) and about 40% RH for 15 weeks under day and night conditions. A characteristic of packaging materials used in experiments is shown in the Table 1 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 asses 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.

Samples (5) and (6) shown in Fig. 1, packed both in VC999 BioPack lidding film PLA (100% \( CO_2 \)) and Multibarrier 60 film pouches in air ambience grow mouldy already after 5 to
6 days storage, therefore for ulterior experiments those samples were not used.

<table>
<thead>
<tr>
<th>Nr.</th>
<th>Packaging material</th>
<th>Composition</th>
<th>Thickness, μm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Paper bag film</td>
<td>Double layer, brown colour Laminate, APA/TIE/PA/EVOH/PA/T</td>
<td>130±2</td>
</tr>
<tr>
<td>2.</td>
<td>Multibarier 60 film</td>
<td>APE/EPE/PE, transparent</td>
<td>60±2</td>
</tr>
<tr>
<td>3</td>
<td>VC999 BioPack lidding film</td>
<td>Single layer, transparent</td>
<td>50±2</td>
</tr>
</tbody>
</table>

Exponent 32. Hardness was measured as the maximum penetration force (N) reached during breakage of tissue. The measuring parameters were: pre-test speed 1 mm s⁻¹; test speed 1 mm s⁻¹; post-test speed 10 mm s⁻¹; 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), the hardness changes 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. At each time of measurement, two identical packages for each treatment were randomly selected on sampling days (day 0) and after 1, 3, 5, 8, 11 and 15 storage weeks. 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 ISO 21807:2004, AquaLab LITE device.

Colour of marmalade candy samples was measured in CIE L*ab colour system using Tristimulus Colorimeter, measuring Hunter colour parameters by Colour Tec PCM/PSM. Colour values were recorded as L* (brightness), a* (greenness, +a, redness) – the horizontal co-ordinate, that runs from -a* (green) through grey to +a* (red) and b* (b, blueness, +b, yellowness) – another horizontal co-ordinate, that runs from -b* (blue) through grey to +b* (yellow) [32]. The measurements were repeated on different randomly selected locations at the surface of each sample. For evaluation of colour change, the total colour difference (ΔE*), was calculated between measurements before packaging of cheese samples and during the storage time according to equation (1):

$$
\Delta E^* = \sqrt{\left( L^* - L_{0}^* \right)^2 + \left( a^* - a_{0}^* \right)^2 + \left( b^* - b_{0}^* \right)^2},
$$

Where:

- \( L^*, a^*, b^* \) – value of marmalade sample colour components measured before packaging;
- \( L_{0}^*, a_{0}^*, b_{0}^* \) – value of marmalade sample colour components measured after storage time.

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 16.0. Two-way analyses of variance (p≤0.05) were used to determine significance of differences between weight losses, moisture content, hardness, aw, pH, colour
changes of atmosphere content (CO₂ and O₂) in headspace of packs, and microbial conditions by different packed samples.

II. RESULTS AND DISCUSSION

Significant differences in carbon dioxide (CO₂) content during the 15 weeks storage among all marmalade candy samples packed in different kinds of materials were found (p<0.05), (Fig. 2). The changes of carbon dioxide content during the first 12 weeks storage in Multibarrier 60 pouches without oxygen scavenger (sample 2) were not significant (p>0.05). An interesting phenomenon we have observed analysing experimental data of head space composition in modified atmosphere (MAP 100% CO₂). In Multibarrier 60 pouches without oxygen scavenger the CO₂ content at the 11th week began to fall down step by step and after 15 weeks the pouches collapsed and a perfect vacuum established (Fig.2). In Multibarrier 60 pouches (MAP, 100% CO₂) with incorporated O₂ scavenger, 500 cc and as well in VC999 BioPack lidding film PLA pouches (MAP, 100% CO₂) the decrease of CO₂ content began to fall down already after one week of storage, decreasing till 80% and after 5 storage weeks the decrease was notable – as early as at the 8th storage week vacuum established in pouches and the packs lost marketing appearance. This phenomenon can be explained with carbon dioxide dissolving in the marmalade candies having quite high moisture content (44.78±0.09%). As a result, the pressure decreased in the packages and destroyed them. Presumably, the solubility of CO₂, as we can see in the Fig. 3, was mutually connected with simultaneous decrease of oxygen content in pouches. The concentration of carbon dioxide in paper bag was not more than 1.0±0.1%.

The oxygen content (O₂) in Multibarrier 60 packaging during 11 storage weeks increased on average till 4.7±0.5%, from this moment decrease of O₂ began and during 15 storage weeks reached 0% (Fig. 3). In the packages made of Multibarrier 60 with incorporated oxygen scavenger as well as of VC999 BioPack lidding film PLA during 5 storage weeks the O₂ content increased on average till 2.8±0.3% to 4.8±0.5%, after that the decrease started considerably faster, and already during 8 weeks a vacuum was developed in both packages. Though during 15 weeks storage in all pouches made from investigated polymer materials the O₂ content did not disparate (p<0.05), as in all samples vacuum was formed. In return in the paper bags the content of O₂ during all experiment period was disparate from packages in polymer pouches – it was similar like in the surrounding environment.

![Fig. 2 The dynamics of carbon dioxide (CO₂) content in the headspace of package in MAP (100%)](image1)

![Fig. 3 The dynamics of oxygen (O₂) content in the headspace of package in MAP (100%)](image2)

The mass losses formed during the storage time are presented in Fig. 4. Experimentally we have observed that mass losses from the marmalade candy samples packed in VC999 BioPack lidding film PLA film pouches, as well as in paper bags considerably differed (p<0.05) from those packed in Multibarrier 60 film pouches both without and with oxygen scavenger. The mass losses of marmalade candy samples packed in paper bags were very high – during 15 storage weeks more than 30%, as long as in environmental friendly packing – not more than 15%. On the contrary mass losses from the marmalade candies packed in Multibarrieer 60 without and with incorporated oxygen scavenger within 15 storage weeks were considerably smaller – only 3.2% to 3.5%. Following these results, we can come to a conclusion that Multibarrieer 60 film packaging without as well as with incorporated oxygen scavenger could be the best from investigated packaging materials for marmalade candy packaging and long-term storage.

![Fig. 4 The dynamics of mass losses of marmalade candy samples in MAP (100%) packages](image3)
Initial moisture content of marmalade candies was 44.78 ± 0.09%. As we can see in Fig. 5, the moisture content decrease during 15 storage weeks was various, influenced by different water vapour permeation of packaging materials (p<0.05). Significant difference in moisture content values at the end of storage among marmalade candy samples packed in Multibarier 60 without and with incorporated oxygen scavengers was not found (p>0.05), as well as the changes of moisture content during the storage were in little importance. In VC999 BioPack lidding film PLA it decreased till 37.0 ± 0.1%, at the same time in samples packed in paper bags the moisture content differed (p<0.05) from those packed in previously mentioned packaging materials and decreased even till 12.0 ± 0.1%.

Hardness changes in apple-black currant marmalade candy samples stored in various packaging materials are presented in Fig. 6–7. The marmalade candies became harder irrespective of used packaging material type and technology. Presumably the major hardening reason can be water vapour migration through the packaging material as well as some biochemical and chemical processes accruing during the storage, which promote hardening. The initial cutting force of all samples, was 1.38 ± 0.02 N. Mouth feel, texture and eating qualities are adversely affected by the loss of moisture [30]. Mouth feel of all tested samples, excepting sample packed in paper bags, during the investigated storage time was acceptable. Evaluating from the view point of product hardening, as the best variant for marmalade candy packaging we have found transparent Multibarrier 60 film without incorporated oxygen scavenger – after 15 storage weeks showing insignificant increase in the cutting force of packed product only till 1.68 ± 0.03 N. At the same time in Multibarier 60 film with used oxygen scavenger and in VC999 BioPack lidding film PLA packaging the increase of hardness was somewhat higher – till 2.60 ± 0.05 N. On the contrary the increase of cutting force of marmalade candies packed in paper bags was very high – from 1.38 ± 0.02 N to 179.19 ± 21.02 N, the maximal cutting force substantially differed (p<0.05) from all other investigated samples, and the candies after 1 to 2 weeks storage were not useful for eating.

Significant difference of pH values among investigated marmalade candy samples after 15 weeks storage was not found (p>0.05), and the decrease of pH was not notable (Fig. 8).

The value of water activity (a_w) of freshly prepared apple-black currant marmalade candies was 0.927 (Fig. 9). After 15 storage weeks a_w of marmalade samples packed in
Multibarier 60 without and with incorporated oxygen scavenger, and as well in VC999 BioPack lidding film PLA did not change significantly (p>0.05). For its part, water activity of candies packed in paper bags, accordingly with reduction of moisture content from 44.78±0.09% to 12.6±0.1% (Fig. 5), during the same storage time considerably reduced from 0.927 to 0.591.

The results demonstrated preference of active packaging realised with oxygen scavenger incorporation in the pouches as an effective method to prevent microbial growth during storage and enhancing the shelf life of marmalade candies till 15 weeks. The CFU count in packages with oxygen scavenger in Multibarier 60 pouches was 2.1 log cfu g⁻¹, which is considerably less than in the same material packed samples without oxygen scavenger – 3.7 log cfu g⁻¹. Microbial analyses of candy samples packed in VC999 BioPack lidding film PLA pouches (MAP100% CO₂) were accomplished only till 8th storage week, because the samples got spoiled. The samples packed in paper bags was microbiologically analyzed only during 2 weeks storage, because the cutting force of marmalade candies in this packaging turned very high and the candies got not eatable.

Growth of microorganisms in apple-black currant marmalade candies was affected by packaging material, and as well packaging method (Figs 10–11).

Evaluation of the experimentally obtained results is concerned with the fact, that for the time being in Latvia any document recommending colony forming unit (CFU) count, yeast, and mould allowable colony forming units (cfu g⁻¹) in candies made of fruits and berries does not exist. Therefore, in this experiment we have followed the recent regulation of the Cabinet of Ministers of Latvia No. 292 “For food contamination” being in force from 02.08.1999, as well as considered previously accomplished microbiological studies. In this research we have supposed, that CFU count could not be allowed more than 4.0 log cfu g⁻¹, and yeast and mould count – not more than 2.0 log cfu g⁻¹.

The initial mould count of marmalade samples was 0.51±0.01 log cfu g⁻¹ (Fig. 11), which during storage time increased different. The mould count in candy samples packed in Multibarier 60 film pouches during 15 storage weeks did not exceed 2 log cfu g⁻¹, as we had supposed previously, however the mould count was influenced by active packaging – the incorporated oxygen scavenger slowed down the mould growth (p<0.05). The samples packed in VC999 BioPack lidding film PLA pouches got moldy jet after 4 weeks storage, when mould count reached a value 3 log cfu g⁻¹, and the samples turned mouldy and spoiled. Consequently, the environmentally friendly PLA could not be used for long term storage of marmalade candies, the shelf life in this packaging could be to the utmost only till 4 weeks.

The yeast growths in all packaging conditions was equal – after 15 storage weeks in average 9 log cfu g⁻¹ were observed. The presence of enterobacteria *Escherichia coli* (*E. coli*) in the tested marmalade candy samples was not found.

The initial value of marmalade candy colour component L* was 18.2±0.5 units, which in all investigated packaging situations in polymer films during 15 storage weeks step by step decreased till 14.0±0.5 units (Fig 12). It means – the colour became lighter and did not differ among samples.
As we have already described previously, the VC999 BioPack lidding film PLA could be useful for the marmalade candy quality maintenance only along 4 storage weeks, this material most of all influenced the changes of L* value during the storage – along the first 4 storage weeks the L* value increased till 20.0 ± 0.5 units, henceforward till 8 storage weeks – step by step till 22.0 ± 0.5 units, it means, the samples lost moisture and became darker.

The values of a* meaning (-a) – greenness, (+) – redness, are graphically represented in Fig. 13. The initial a* value of marmalade candy samples was +7.8 ± 0.5, which in Multibarrier 60 film packaging both without and with oxygen scavengers gradually decreased on average till +6.4 ± 0.5 units, and after storage of 15 weeks there was not observed substantial influence of oxygen scavengers on the a* value.

The a* value of samples packed in paper bags in air ambiance essentially differed (p<0.05) from previously mentioned samples in Multibarrier 60 packaging, already after one week storage decreasing till 3.0 ± 0.5 units, which as well, similarly like L* value changes, could be explained by moisture loss during storage.

To describe the product overall colour change during the storage time, the influence of packaging materials and oxygen scavenger on the total colour difference (ΔE*) has been calculated by equation (1) (Fig. 15.). We have observed notable total colour difference (ΔE*) of examined samples.

Comparing the control sample in paper bags, and samples packed in Multibarrier 60 film without and with oxygen scavenger, and in VC999 BioPack lidding film PLA packaging, we found disparity in colour difference (p<0.05).

Total colour difference (ΔE*) substantially differed of those samples packed in paper bags during storage 8 weeks, what could be explained by influence of high moisture loss during storage as well as by oxygen presence in non hermetrical packs. The presence of oxygen scavengers in packaging during 15 storage weeks did not substantially influence the colour difference of candy samples packed in Multibarrier 60 film pouches (p>0.05). The colour difference of samples packed in biodegradable VC999 BioPack lidding film PLA pouches did not disparate from all samples packed in Multibarrier 60 film (p>0.05).
Multibarier 60 pouches (MAP, 100% CO₂) with incorporated paper bags; 2 – Multibarier 60 film PLA pouches (MAP, 100% CO₂).

Our obtained results show, that the investigated conventional Multibarrier 60 film is applicable for apple-black currant marmalade candy’s long-term storage in packaging maintaining quality, among them the active packaging in Multibarier 60 pouches (MAP, 100% CO₂) with incorporated oxygen scavenger could be considered as the best variant. Packaging in biodegradable VC999 BioPack lidding film PLA can maintain quality of apple-black currant marmalade candies during storage time only till 8 weeks. The paper bags could be used only for candy’s short-term packaging on the supermarket shelves.

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