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EFFECT OF THE PACKAGING TYPE ON THE QUALITY PARAMETERS OF SEA BUCKTHORN SYRUP

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Sea buckthorn syrup is a product that derives from the sea buckthorn fruits, an alternative crop for the Subcarpathian region of Moldova and Muntenia, Romania. The purpose of this study is to analyze a series of physico-chemical parameters of home-made sea buckthorn syrup stored in three kinds of packaging (glass (symbolized G), PET plastic (symbolized PET) and aluminized cardboard symbolized (AC)), in order to highlight the influence of packaging on product quality and shelf life. The duration of the study was six months, from September 2022 to February 2023, with measurements of the physico-chemical parameters being performed approximately every 30 days. The effect of storage period on the quality parameters, pH, ºBrix, density, titratable acidity, electrical conductivity, antioxidant activity (ascorbic acid content), and sensory attributes was investigated. Significant variations were observed for the content of total soluble solids (°Brix), which reflects the sugar content, and antioxidant activity.

Keywords: *sea buckthorn, quality parameters, antioxidant activity, °Brix, shelf life*

1. Introduction

Sea buckthorn (*Hippophae Rhamnoides Linnaeus*) is a flowering plant (Angiosperm) of the order Rosales and the family *Elaeagnaceae* which is cultivated in orchards in the USA, Canada, and Europe [1]. It is characterized by silvery leafy leaves and small orange fruits with a bitter and sour taste, resistant to harsh conditions, drought, low temperatures, or polluted air. Sea buckthorn fruits are rich in nutrients that are essential for the proper functioning of the human body, such as fatty acids, vitamins C, A, E, K, riboflavin and folic acid, minerals

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(calcium, magnesium, phosphorus, iron), but also bioactive compounds such as carotenoids (lutein, zeaxanthin, β-cryptoxanthin, lycopene, γ-carotene, β-carotene), phenolic compounds (gallic acid, chlorogenic acid, etc.), sterols (ergosterol, stigmasterol, lansterol, amyrin), triterpenols, isoprenols, organic acids (malic, oxalic), serotonin and polysaturated fatty acids [2-4]. Along with vitamin C, polyphenols confer sea buckthorn fruit its high antioxidant activity [5].

Sea buckthorn fruits are usually very acidic and with a low degree of sweetness due to the high acid/sugar ratio in the fruits, which is influenced by weather conditions and water source [6]. Their astringent taste is caused by the malic acid, the major acid in the fruit. Although it has a unique taste (mainly due to its sourness and bitterness), the sea buckthorn juice is a refreshing and healthy drink for many consumers. It has numerous positive physiological, biological, and medicinal effects, being characterized as antioxidative and immunomodulating [7], anti-inflammatory [8], cardioprotective and antiatherogenic [9], antidiabetic [10], antibacterial and antiviral effects [11], antiradiation [12], anticarcinogenic [13], healing influence on acute and chronic wounds [14], hepatoprotective and dermatological effects [15, 16].

There are numerous sea buckthorn crops in Romania, especially in the Subcarpathian region of Moldova and Muntenia. A large percentage of the fruits harvested in the months of August-September are used in food products, in order to avoid waste and to extend the shelf life. The content of nutrients and bioactive compounds varies considerably among varieties.

Sea buckthorn can be consumed in several ways, as a food supplement, juice, syrup, jam or hot tea. In all these forms, it provides health benefits to the consumer. Using syrup is a handy way to profit all year round from the beneficial effects of sea buckthorn. Therefore, processing fruits into syrup is an effective means to improve sensory characteristics and extend the shelf life of the product up to approximately 2 years due to the large amount of sugar that acts as a preservative. During the processing, additives and other ingredients can be added to enhance the taste and shelf life of the syrup.

Food packaging aims to protect the product and preserve it along the supply chain from physical, chemical, microbiological, or other hazards. PET, glass, metals (tinplate, aluminum, foils and laminates, and tin-free steel), cardboard and plastics are all common materials used for food packaging, each having a different impact on the environment during its life cycle and on the product [17]. It is important to choose the packaging material according to the components of the final product and all possible product-packaging interactions regarding food quality and safety. The packaging material must display excellent barrier characteristics against the transfer of diverse permeants like gases, moisture, and lipids across the packaging wall [18]. The factors that should be taken into account when selecting the packaging material are the quality of the product, the cost, and its ability to maintain the freshness of the product [19].

Research on sea buckthorn has increased considerably in the last two decades, but only a few studies are found in the literature on the quality of sea buckthorn syrup [20,21]. The present study aimed to analyze a series of physico-chemical parameters of sea buckthorn syrup stored in three types of packaging (glass, PET plastic and aluminized cardboard) in order to evaluate the influence of the packaging on quality parameters of the syrup. The period of the study was six months, from September 2022 to February 2023, with measurement of physico-chemical parameters and sensory attributes approximately every 30 days.

2. Materials and Methods

2.1. Chemicals

The following reagents were used: ethanol (96%) and sodium hydroxide (0.1 N) from Chemical Company®, Romania, 2,2-diphenyl-1- picrylhydrazyl and potassium chloride (≥99.5%) from Sigma Aldrich, L(+)-ascorbic acid (≥99.7%) from Schar lab. All the chemical substances were used without any further purification.

2.2. Samples

The sea buckthorn fruits were purchased from a local supermarket in Brasov, Romania. They were washed, then crushed using a blender. The mixture obtained was soaked in water (1:1 ratio) for 12 hours in a dark, cool place. It was then filtered through stainless steel sieve (d= 320 mm) to obtain sea buckthorn juice, which was boiled with the gradual addition of sugar (0.9 kg of sugar per 1 L of juice) for 5 hours until a homogenous syrup was obtained. Sugar was added both for preservation and to improve the sensory attributes. The syrup samples were poured in glass, PET plastic and aluminized cardboard, coded as G, PET and AC, respectively, followed by pasteurization at 85°C for 1 minute. The syrup samples were immediately cooled in an ice bath to approximately 6°C (heat shock to kill bacteria). Then they were stored during the study in a dry, dark environment at an average ambient temperature of 12°C. The samples were divided into six batches (corresponding to six months of storage) and analyzed every 30 days (coded T0-T6). T0 represents initial sample, T1 is sample stored for one month, T2 is sample stored for two months, T3 describes sample stored for a period of three months, T4 is sample stored for four months, T5 and T6 are samples stored for five months and for six months, respectively.

2.3. Apparatus

The soluble solids were measured with a digital refractometer (HI 96801 Hanna Instruments) and were expressed as ^{o}Brix. The pH values and electrical conductivity were measured with a pH, EC meter from Hanna Instruments (HI

991301). The acidity was performed using an automatic potentiometric titrator HI 902 (Hanna Instruments). UV-Vis spectrophotometer 1280 Shimadzu was used to determine the antioxidant activity given by the ascorbic acid content. The density was determined using a 100 mL pycnometer and an analytical balance Kern EWJ for weighing. All the experiments were conducted in triplicate.

2.4. Physico-chemical and organoleptic analyses

For the organoleptic examination, four sensory attributes were considered: appearance, consistency, taste and smell. The evaluation of sea buckthorn syrup was carried out according to the standard SR EN 784-3:2009 specific for honey [22], which was chosen as a reference document due to the similarities between sea buckthorn syrup and honey in terms of the level of sweetness and consistency.

The density of sea buckthorn syrup was determined at temperature of 20°C using the pycnometric method. The mass of a known sample volume was determined using a pycnometer of approximately 100 mL volume and an analytical balance Kern EWJ (precision ±0.001 g).

Total soluble solids (°Brix) of the sea buckthorn syrup were determined by the refractometric method according to SR EN 12143:2003 [23]. The sugar content in the sea buckthorn syrup was determined according to equation:

$$\text{Sugar content}\left[\frac{g}{L}\right] = {}^{\circ}\text{Brix} * \rho * \frac{1000}{100} \quad (1)$$

High sugar concentrations in syrup (ºBrix > 55) are beneficial because produce food environments with high osmotic pressure that prevent bacterial proliferation [24].

The pH was determined by the potentiometric method using 5 mL of sample. The pH-meter (pH accuracy ±0.02) was calibrated with acidic and basic solutions of KCl (pH 4.0 and 9.0). Sea buckthorn syrup has an acidic pH due to the organic acids, which give an astringent and sour taste to the product. Foods with a low pH are less likely to spoil than those with a high pH, due to the inhibition of the growth of microorganisms [24].

Electrical conductivity was measured with a pH/EC meter (resolution 1 µS/cm). Electrical conductivity is expressed by the ability of the ions present in the solution to conduct electrons and its determination provides information about the moisture and the salt content of the syrup. Since electrical conductivity varies inversely with viscosity, it provides indirect information of the viscosity of the samples.

The volumetric method of direct titration with NaOH was used to determine the acidity of the syrup. The samples (10 mL) were diluted with 40 mL water and neutralized with a measured volume of 0.1 N NaOH (V_{NaOH}) until the color changed to pink in the presence of phenolphthalein indicator. The acidity of the sea buckthorn syrup was given as citric acid and calculated with:

$$acidity\left[\frac{g\ citric\ acid}{L}\right] = \frac{V_{NaOH}*0{,}1*192{,}13}{Vsample} \quad (2)$$

The antioxidant activity of the syrups was determined spectrophotometrically based on the measurement of DPPH radical scavenging activity as described by El-Sohaimy et al. [25] and expressed as ascorbic acid content. To determine antioxidant activity, an ethanolic solution containing DPPH radicals (9.85 mg/250 mL) was used. The reduction of DPPH free radicals was indicated by a change in color from violet to yellow and was measured after 30 min at 517 nm.

Quantitative determination of ascorbic acid was performed with ascorbic acid standards applying an external calibration curve between 0 and 600 ppm (Fig. 1). The greater the quantity of ascorbic acid, the greater the power of the sea buckthorn syrup sample to neutralize the free radical responsible for oxidative processes in the body.

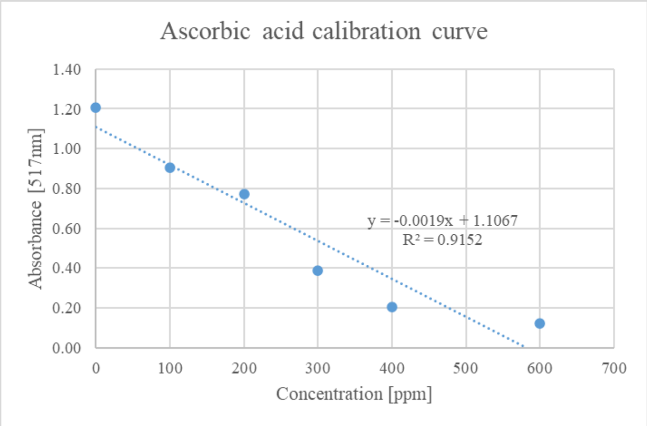


Fig. 1. Ascorbic acid calibration curve

The percentage of DPPH inhibition (%DPPH Inhibition) was determined by measuring the absorbance of the samples (A_S) and of the EtOH/DPPH solution (A_{DPPH}) at 517 nm and was calculated as a percentage of DPPH decolorization using the equation:

$$\%\ \text{DPPH Inhibition} = (A_{DPPH} - A_S)/A_{DPPH} * 100 \quad (3)$$

3. Results and discussion

The organoleptic characteristics of the investigated sea buckthorn syrup are presented in Table 1. The sea buckthorn syrup samples showed no variations in terms of organoleptic characteristics during the study, regardless of the type of packaging used. The samples retained their opaque dark reddish-brown colour.

The consistency remained dense, viscous, and honey-like, leaving a sticky feeling to the touch. The sweet-bitter and slightly sour taste was also maintained. The sweet and fruity scent with citrus notes was also consistent during the study. These results are similar with those from literature, that indicate that glass packaging has almost no impact on the foods organoleptic properties [19]. In the case of PET bottles, it is reported in the literature that they contain acetaldehyde as the main volatile substance with effects on juice odours, especially in cola-type beverages [26].

Table 1

Organoleptic examination results

Organoleptic property	Storage time							
	Sample	T0	T1	T2	T3	T5	T5	T6
Aspect	G	Opaque, dark (reddish-brown)						
	PET							
	AC							
Consistency	G	Viscous, sticky, with fruit sediments						
	PET							
	AC							
Smell	G	Intense, sweet, fruity with slight citrus notes						
	PET							
	AC							
Taste	G	Pleasant, intensely sweet, slightly astringent, bitter and sour						
	PET							
	AC							

These observations show that the mass transfer of colour, aroma, flavour, and nutrients from the syrup to the packaging, with the impact on the organoleptic properties, did not take place [27].

The results of the physico-chemical analyses pH, density, titratable acidity, electrical conductivity, ascorbic acid concentration, % DPPH inhibition, °Brix, and sugar content are presented in Table 2 and illustrated in Fig. 2.

The quality of obtained results was evaluated by standard deviation (s), calculated with the equation:

$$s = \sqrt{\frac{\sum_{i=1}^{N}(x_i - x_m)^2}{N-1}} \quad (4)$$

The high density (1.2538 - 1.2750 g/cm^3) of sea buckthorn syrup is mainly due to the sugar content. These values are higher than those of Sevenich et al. [21] of 1.130 g/cm^3 for varieties *Leikora* and *Botanica*, probably due to the smaller content of added sugar (0.5 and 0.7 kg respectively, of sugar to 1 L of juice).

Table 2.1

Influence of the packaging type (S, P, C) and storage time (T0-T3) on the physico-chemical parameters of the sea buckthorn syrup

Parameter	Sample	**T0**	s	**T1**	s	**T2**	s	**T3**	s
Density (g/cm^3)	G	1.2720	$3\cdot10^{-4}$	1.2720	$3\cdot10^{-4}$	1.2738	$3\cdot10^{-4}$	1.2748	10^{-4}
	PET	1.2682	$2\cdot10^{-4}$	1.2682	$2\cdot10^{-4}$	1.2702	$3\cdot10^{-4}$	1.2728	$2\cdot10^{-4}$
	AC	1.2538	10^{-4}	1.2538	10^{-4}	1.2548	$3\cdot10^{-4}$	1.2554	10^{-4}
pH	G	2.77	10^{-2}	2.77	10^{-2}	2.74	$5.8\cdot10^{-3}$	2.75	0
	PET	2.79	$5.8\cdot10^{-3}$	2.79	$5.8\cdot10^{-3}$	2.82	$5.8\cdot10^{-3}$	2.84	$5.8\cdot10^{-3}$
	AC	2.81	$1.1\cdot10^{-2}$	2.81	$1.1\cdot10^{-2}$	2.8	$5.8\cdot10^{-3}$	2.78	0
Titratable acidity (g citric acid/L)	G	7.99	0.013	7.99	0.013	8.25	0.052	8.36	0.057
	PET	8.02	0.191	8.02	0.191	8.26	0.194	8.31	0.238
	AC	7.54	0.139	7.54	0.139	7.92	0.168	8.04	0.182
Electrical conductivity (μS/cm)	G	397	0.577	397	0.577	375	0.577	371	1.155
	PET	383	2.082	383	2.082	369	0.577	369	1.527
	AC	395	2.309	395	2.309	360	0.577	358	2.082
Ascorbic acid content (mg/100 g)	G	290	5.253	290	5.253	292	0.938	295	5.810
	PET	293	4.641	293	4.641	296	3.963	304	3.975
	AC	308	3.134	308	3.134	310	3.118	309	4.264
% DPPH inhibition	G	68.7	1.552	68.7	1.552	71.5	0.256	72.4	1.585
	PET	69.6	1.371	69.6	1.371	72.7	1.081	74.8	1.084
	AC	73.8	0.926	73.8	0.926	76.6	0.850	76.0	1.163
˚Brix	G	59.0	$5.8\cdot10^{-2}$	59.0	$5.8\cdot10^{-2}$	60.2	$5.8\cdot10^{-2}$	59,8	$5.8\cdot10^{-2}$
	PET	58.9	$5.8\cdot10^{-2}$	58.9	$5.8\cdot10^{-2}$	60.6	0	59.9	$5.8\cdot10^{-2}$
	AC	58.6	0	58.6	0	59.8	$5.8\cdot10^{-2}$	59.4	$5.8\cdot10^{-2}$
Sugar content (g/L)	G	751	0.734	751	0.734	766	0.735	763	0.736
	PET	747	0.732	747	0.732	770	0	763	0.735
	AC	735	0	735	0	751	0.724	746	0.725

Table 2.2

Influence of the packaging type (S, P, C) and storage time (T4-T6) on the physico-chemical parameters of the sea buckthorn syrup

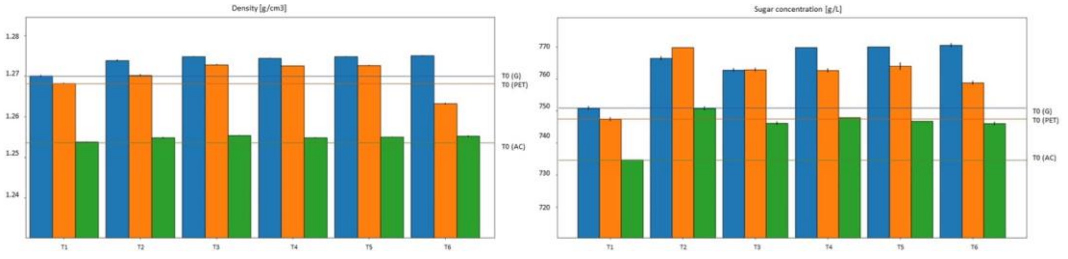
Parameter	Sample	**T4**	s	**T5**	s	**T6**	s
Density (g/cm^3)	G	1.2744	10^{-4}	1.2748	10^{-4}	1.2750	10^{-4}
	PET	1.2725	10^{-4}	1.2726	$2\cdot10^{-4}$	1.2632	$3\cdot10^{-4}$
	AC	1.2548	$2\cdot10^{-4}$	1.2550	10^{-4}	1.2552	$3\cdot10^{-4}$
pH	G	2.77	$2.3\cdot10^{-2}$	2.72	$1.1\cdot10^{-2}$	2.67	$5.8\cdot10^{-3}$
	PET	2.8	$5.8\cdot10^{-3}$	2.73	$2.6\cdot10^{-2}$	2.66	$1.7\cdot10^{-2}$
	AC	2.78	$5.8\cdot10^{-3}$	2.74	$5.8\cdot10^{-3}$	2.7	$5.8\cdot10^{-3}$
Titratable acidity (g citric acid/L)	G	8.49	0.067	8.84	0.156	10.00	0.129
	PET	8.50	0.311	8.91	0.281	10.18	0.197
	AC	8.35	0.135	8.89	0.159	9.60	0.261
Electrical conductivity (μS/cm)	G	367	0	370	1.527	364	2.082
	PET	352	2	360	0.577	347	0.577
	AC	355	0.577	350	0.577	352	2.517
Ascorbic acid content	G	303	5.094	306	0.831	319	4.052
	PET	306	3.012	315	2.709	339	1.176

(mg/100 g)	AC	313	2.273	325	1.318	341	2.837
% DPPH inhibition	G	74.4	1.400	75.2	0.229	72.0	1.473
	PET	75.1	0.828	77.7	0.748	79.2	0.427
	AC	77.1	0.625	80.2	0.364	80.1	1.032
˚Brix	G	60.4	0	60.4	0	60.4	$5.8\cdot10^{-2}$
	PET	59.9	$5.8\cdot10^{-2}$	60.0	$1.2\cdot10^{-1}$	60.1	$5.8\cdot10^{-2}$
	AC	59.6	0	59.5	0	59.4	$5.8\cdot10^{-2}$
Sugar content (g/L)	G	770	0	770	0	771	0.736
	PET	763	0.735	764	1.469	759	0.729
	AC	748	0	747	0	746	0.725

Regarding the influence of the type of packaging on the density, it can be observed (Tables 2.1 and 2.2) that sample G T6 has the highest value (1.2750 g/cm^3) and sample AC T0, the lowest (1.2538 g/ cm^3). Fig. 2 shows that the packaging type influences the density values at all moments, the variation being: glass > plastic > cardboard.

The pH values of the sea buckthorn syrup varied in the range 2.66 – 2.84, the most acidic being the samples packed in glass (G). The lower pH values are given by organic acids, including citric acid, that contribute to the sour taste of sea buckthorn. Unlike previous parameters, no regular variation of pH values with the packaging type is observed. The highest values are recorded for the plastic packaging (PET) at T0-T4, compared with G and AC samples. The pH values for PET samples increase to a maximum at T3, followed by a decrease (a difference of 4.77% was observed between the values at T0 - T6). The samples packed in G and AC, have no regular variation during the study. These results agreed well with the literature, where are indicated pH values between 2.5 - 3.0 [5, 21].

From Fig. 2 it can be observed that acidity slowly increases during the storage regardless of packaging type. This is consistent with existing literature. [28]. Acidity varies inversely with pH, as expected. A higher growth in acidity was observed for the syrup stored in plastic (from 8.02 to 10.18 g citric acid/L), and a smaller one, for the sample stored in glass container (from 7.99 to 10.0 citric acid/L). This could indicate a better syrup preservation in glass packaging. The results expressed as citric acid content are higher than the values found in the literature (0.42 - 2.34 g/L) for sea buckthorn juice [29]. This is possible because the acidity strongly depends on the species variety and growing conditions.



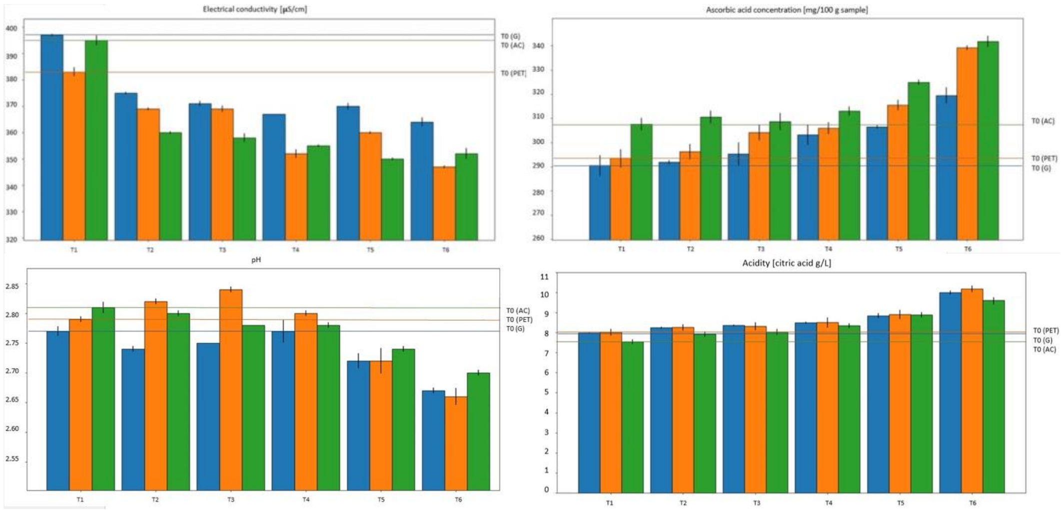


Fig. 2. Physico-chemical analyses results for the sea buckthorn syrup stored in glass packaging (blue), plastic packaging (orange), cardboard packaging (green) during storage period (T1-T6) (T0 (PET); T0 (G) and T0 (AC) symbolize the initial values of the analyzed parameters

The electrical conductivity values ranged from 350 to 390 µS/cm, showing a decreasing trend during storage for all samples, regardless of packaging. This decrease in electrical conductivity could indicate an increase in viscosity of samples during storage or separation of some layers over time. The highest values are recorded for samples stored in G. The largest decrease from 395 to 352 µS/cm (of 11%) was observed in samples stored in AC. The obtained values are according with literature values (297-539 µS/cm) for sea buckthorn juice [29] and below the maximum value allowed by legislation for honey (800 µS/cm) [30].

The concentration of ascorbic acid presented an upward trend during the study period, varying from 308 (T0) to 341 mg/100 g (T6) for the samples packaged in AC and from 290 (T0) to 319 mg/100 g (T6) for the samples packaged in G. This difference may be due to the better protection from light provided by the AC; the antioxidants can be damaged if they are exposed to UV light [30]. However, the samples stored in G have a smaller difference between T0 and T6, of around 10%, compared to the other two types of packaging. We can say that the syrup is more stable in glass packaging (G). It is known from literature that sea buckthorn does not contain ascorbate oxidase, so there is no loss of vitamin C during processing and storage [20, 21]. In our syrup samples the content of vitamin C did not change, but slightly increased during storage time. The vitamin C concentration varies between 50 mg and 2500 mg/100, being strongly influenced by sea buckthorn variety and growing conditions [5, 25].

Soluble solids of syrup ranged between 58.6 and 60.4 °Brix, values in the upper ranges reported in the literature [20, 21]. The sugar content of the sea buckthorn syrup varies between 735-771 g/L and reflects the high content of added sugar. Literature studies indicate much smaller values of 93-173 g sugar/L for sea buckthorn juice [29, 31]. Like the concentration of ascorbic acid, the sugar content slightly increased during the study. This can be due to the moisture loss of sea buckthorn syrup during storage through mass transfer through the package walls [19]. The most significant increase (of 2.61%) and the highest sugar concentration was observed for the syrup packed in glass and the lowest, in the samples stored in cardboard.

4. Conclusions

The sea buckthorn syrup samples were stable over 6 months of storage at 12°C, regardless of packaging. Storage in different packaging did not influence the organoleptic properties, the sensory attributes of sea buckthorn syrup remained constant for six months. An increase in syrup density, acidity, soluble solids, and sugar content and a decrease of pH and electrical conductivity during storage period were observed. The variation of the physico-chemical properties with packaging type indicates a better syrup preservation in glass.

The most important and interesting quality parameters of sea buckthorn syrup are the content of total soluble solids (°Brix), which reflects the sugar content, and the antioxidant activity. Important variation for the sugar content was found for the G container, less for the PET one. At the end of the study, the highest concentration of ascorbic acid was found in the samples packed in AC containers. This difference may be due to better protection from light provided by the AC packaging. Sea buckthorn syrup samples packaged in G containers had the lowest ascorbic acid concentration and the lowest DPPH radical inhibition power. The increase in ascorbic acid and sugar content during storage is possible due to the loss of moisture of sea buckthorn syrup during storage. The decrease of electrical conductivity over time may be due to the increase of viscosity samples.

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