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ORIGINAL RESEARCH PAPER

PHYSICO-CHEMICAL CHARACTERISTICS AND ANTIOXIDANT ACTIVITY OF GOJI FRUITS JAM AND JELLY DURING STORAGE

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Since the 1990s, when the news about antioxidants and their benefits to health has begun to spread to the general public, statements about the benefits of antioxidants ranged from preventing colds to cancer treatment. Fruits and vegetables are excellent sources of antioxidants. Since the beginning of the 21st century, goji berries have become increasingly popular in Europe and North America and have been promoted in advertisements and in the media as an anti-aging remedy. Goji is a relatively new name given to *Lycium Barbarum* and *Lycium chinense,* two nearby species, with a long history of use as medicinal and food plants in East Asia, particularly in China. In the present paper are presented analysis results of Goji fruits and food products made from goji fruits (jam and jelly). Storage conditions are important factors for jams and jelly quality. The objective of this study was to monitor the physicochemical stability, antioxidant activity and sensorial profile of goji fruits jam and jelly. Special attention was paid to total phenolic and flavonoid content, antioxidant activity, total soluble solids, titratable acidity, pH and sensorial characteristics. Our results showed the antioxidant activity of the goji fruit, values which correlate well with the results obtained for total phenolic (351±7.25 mg GAE/100g) and flavonoid content (53.06±1.23 mg QE/100g). The antioxidant activity of the goji fruits was maintained also in the finished products obtained in the present study jam (60.98 %) and jelly (41.96 %). Both goji fruits jam and goji fruits jelly showed no significant variations of physico-chemical characteristics and sensorial parameter scores after storage at refrigeration temperature for 10 days.

Keywords: *Lycium barbarum*, antioxidant activity, goji fruits jam, goji fruits jelly, sensorial characteristics.

Introduction

Lycium barbarum L. is one of the important traditional Chinese medicinal plant species. It has been cultivated in Northwest China and used as daily functional food in China, Southeast Asia and many European countries. (Amagase and Nance, 2008; Chang and So, 2008).

Lycium barbarum (goji) fruit has become more popular over the last few years due to its public acceptance as a “super food” with highly advantageous nutritive and antioxidant properties. Concentrated extracts and infusions prepared from the berries have a history of use as ingredients in various soft or alcoholic drinks marketed for their perceived anti-aging, vision, kidney and liver function benefits (UK Food Standard Agency).

More functions were recently reported as immunity improvement (Lin *et al*., 2008), anti-oxidation (Lu *et al*., 2008), anti-radiation (Qian *et al*., 2004), anticancer (Chao et al., 2006), enhancing hemopoiesis (Hsu *et al*., 1999), anti-aging (Yu *et al*., 2005).

The extracts also exhibit neuroprotection, promotion of endurance, increased metabolism, improved control of glucose and other diabetic symptoms, antiglaucoma effects, immunomodulation, antitumor activity, and cytoprotection (Amagase and Nance, 2008; Chang and So, 2008).

Various chemical constituents are found in *Lycium barbarum* fruit. The most important active compounds are polyphenols, polysaccharides and carotenoids. Its reddish orange color is derived from a group of carotenoids, which make up only 0.03% to 0.5% of the dried fruit (Peng *et al*., 2005). The predominant carotenoid is zeaxanthin, which exists mainly as dipalmitate. Among these chemical constituents of *Lycium barbarum* fruit, the most valuable and well-researched components are a group of unique, water-soluble glycoconjugates - collectively termed *Lycium barbarum* polysaccharides (LBP)—that are estimated to comprise 5% to 8% of the dried fruit (Wang, 1991).

Lycium barbarum polysaccharides have been focused on as the active compounds responsible for the various effects of the *Lycium barbarum*, as mentioned above (Amagase and Nance, 2008; Chang and So, 2008).

Carotenoids have been demonstrated to be effective in preventing chronic diseases such as cardiovascular disease and skin cancer (Fraser and Bramley, 2004; Kohlmeier and Hastings, 1995). Polysaccharides possess antitumor activity (Sheng *et al*., 2007), and they can enhance immunity through production of interleukin and antibody (Yang *et al*., 2008).

In recent years, phenolic acids have received considerable attention because of their protective role against cancer and heart diseases. This may be attributed to their antioxidant activity which was reported to be higher than the vitamin antioxidants (Tanaka *et al*., 1993; Han *et al*., 2001; Tsao *et al*., 2004). On the other hand, flavonoids belong to a family of C6–C3–C6 polyphenol compounds and the major flavonoid subclasses in the diet include flavonol, flavone, flavanone, flavanol, anthocyanin and isoflavone. Of the various flavonols, quercetin is the most frequently studied and has been shown to possess anti-inflammatory and anticancer activities (Deschner, 1991; Ferry *et al*., 1996; Whitaker *et al*., 1996).

Historically, jams and jellies were originated as an early effort to preserve fruit for consumption in the off-season (Baker *et al*. 2005). In traditional jam and jelly manufacture, all the ingredients are mixed in adequate proportions, and the mix concentrated by applying a thermal treatment to reach the required final soluble solids content. Nevertheless, this process also implies an undesirable impact in colour, nutritional value and flavor properties due to the high temperature reached in the cooking process. The objective of the present study was to investigate the physico-chemical properties and antioxidant activity of goji fruits and the effect of storage time on physico-chemical characteristics, antioxidant activity and sensorial characteristics of the obtained jam and jelly.

Materials and methods

Material

The most important raw materials utilized in this research study were represented by: dehydrated goji fruits (*Lycium barbarum* L.), gelatin and sugar purchased from a local supermarket Galati, Romania.

Sample preparation

In this study we have prepared two products: goji fruits jelly and jam.

Goji fruit jam was made according to the following steps:

- quality and quantity reception of raw materials;
- fruits hydration with water at 30°C (ratio 1:3) for 1 h;
- mixture of raw materials with auxiliary materials (sugar and water);
- boiling and mixing at the same time up to bonding the composition;
- storage at refrigeration temperature of 4ºC.

Goji fruits jelly was made according to the following steps:

- quality and quantity reception of raw materials;
- fruits hydration with water at 30°C (ratio 1:3) for 1 h;
- mixture of raw materials with auxiliary materials (sugar, water and gelatin);
- boiling, cooling and casting mold on;
- storage at refrigeration temperature of 4ºC.

The jam and jelly prepared stored at refrigeration temperature were analyzed at 0, 5 and 10 days.

Chemical analysis

Initially it was determined the general composition of the Goji fruits including: water content according to STAS 2213/4-86, the total nitrogen content according to the SR ISO 9037:2007 method, fat content according to the AOAC, 1984 method, carbohydrates using Schoorl method according to STAS 3750-66, ash was achieved by calcination of the sample under defined conditions, slow method at 550-650 °C (reference method) and quick method at 900-920 ° C.

The pH of the samples was measured at 25°C using a Metrohm Instruments pH-meter according to the AOAC method (1984).

The determination of acidity of the samples was achieved using titrimetric method according to STAS 3750-66 for jam and STAS 2213/8 – 86 for jelly.

Soluble substances of the samples were achieved using the refractometric method according to STAS 3750 – 66.

Extracts preparation

The samples for the analysis of flavonoids, polyphenols and antioxidant activity were prepared by extraction with 80% methanol using ultrasounds for 2h, at room temperature. An ultrasound bath Transsonic T310, Elma, Singen, Germany was used as the ultrasound source (frequency 35 kHz). After the extraction, the extracts were collected and filtered. Methanolic phases obtained after extraction were used for flavonoids, polyphenols and antioxidant activity determination by specific spectophotometric methods. The UV VIS Double Beam PC & Scanning Auto Cell Spectrophotometer, model UVD – 3200 (Labomed, Inc., U.S.A) was used for all absorbance measurements.

Analysis of total phenolic content

The total polyphenol content (TPC) of the extracts was determined by spectrophotometry, using gallic acid as standard, according to the method described by the International Organization for Standardization (ISO) 14502-1. Briefly, 1.0 mL of the diluted sample extract was transferred in duplicate to separate tubes containing 5.0 mL of a 1/10 dilution of Folin-Ciocalteu's reagent in water. Then, 4.0 mL of a sodium carbonate solution (7.5% w/v) was added. The tubes were then allowed to stand at room temperature for 60 min before the absorbance at 765 nm was measured against the control sample. The TPC was expressed as gallic acid equivalents (GAE) in mg100 g^{-1} material and tannic acid equivalents in mg100 g^{-1} material. The concentration of polyphenols in the samples was derived from gallic acid standard curve ranging from 10 to 50 mg/mL (Pearson correlation coefficient: R^2: 0.9988).

Estimation of total flavonoid content

The total flavonoid content in the investigated samples was spectrophotometrically measured by using a method based on formation of complex flavonoid-aluminium having a maximum absorbability of 430 nm. A quantity of 1 mL of samples was separately mixed with 1 ml solution of 2% $AlCl_3$; the absorbance was measured after 30 min incubation at room temperature. The flavonoid content was expressed as quercetin equivalents (QE) in mg100 g^{-1} material using the standard curve of quercetin ranging from 10 and 50 mg/mL (Pearson correlation coefficient: R^2: 0.9956)

Antioxidant activity

The DPPH assay was performed as previously described (Mimica-Dukic *et al.*, 2004) with some modifications. The samples (ranging from 0.1 to 1 ml of stock solution) were mixed with 1 ml DPPH solution and made up with 80% MeOH to a final volume of 4 ml. The absorbance of the resulting solutions and the blank (with the same chemicals, except for the sample) was recorded after 1 h at room temperature. For each sample, three replicates were recorded. The disappearance of DPPH was measured spectrophotometrically at 515 nm. RSC, expressed as a percentage, was calculated by the following equation:

$$RSC (\%) = 100 \times (A_{blank} - A_{sample}/A_{blank})$$

where A_{blank} is the absorbance of the control reaction (methanol with DPPH) and A_{sample} is the absorbance of the examined extracts.

Sensorial analysis

Sensory analysis of Goji fruits jam and jelly was made by a team of seven panelists who tested eight characteristics using the scoring scale for: general appearance, taste, color, flavor, texture, aftertaste, astringency (only for Goji fruits jam) and transparency (only for Goji fruits jelly).

Statistical analysis

Statistical analysis was performed using Statistica 5.1. Programme for Windows-Microsoft excel statistics. Means and standard deviations were calculated among samples.

Results and discussion

Physico-chemical properties of goji fruits

Before using them for chemical analysis, dry goji fruits (*Lycium barbarum* L.) were hydrated with water at 30°C (ratio 1:3) for 1 h. Table 1 shows the chemical composition of hydrated goji fruits.

Table 1. Physico-chemical composition of goji fruits

Sample	Proteins g/100g	Carbs g/100g	Lipids g/100g	Dry matter g/100g	Ash g/100g	pH	Total acidity mg malic acid/100 g
Goji fruits	7.88 ± 0.35	42.56 ± 1.50	4.5 ± 0.26	59.16 ± 1.20	0.063 ± 0.45	6.05 ± 0.15	154 ± 4.13

The data are displayed as having a mean ± standard deviation of two replications.

From the data shown in Table 1, the chemical composition of hydrated goji fruits is different from the chemical composition provided by the manufacturer (proteins 12 g/100g, carbohydrates 57.82 g/100g and lipids 6.2 g/100g) and from the chemical compositions shown in the scientific literature. This was due to the operation of hydration which dried goji fruits have been subjected to for more efficient use in the manufacture of jam and jelly. Given that the goji fruits have an astringent taste, the pH and total acidity values highlighted the acid property thereby falling within the limits of weakly acidic pH (Table 1).

Total phenolics, flavonoid contents and antioxidant activity of goji fruits

Phenolics are naturally occurring compounds widely distributed in the plant kingdom and beneficial components of human daily diet. They are important constituents of plants with multiple functions and as dietary phytochemicals for human they display a broad range of functional and biological activities (Lee *et al*., 2007). Phenolic compounds present in goji fruits are mainly phenolic acid and flavonoids. The results regarding total phenolic and flavonoid contents and antioxidant activity of goji fruits are reported in table 2.

Table 2. Total phenolics, flavonoid contents and DPPH free radical scavenging activity of goji fruits

Sample	Total phenolics	Total flavonoids	DPPH free radical scavenging activity (RCS %)
	mg GAE/100g	mg QE/100g	
Goji fruits	351±7.25	53.06±1.23	69.45±2.35

The data are displayed as having a mean ± standard deviation of two replications.

Phenolic compounds exhibit antioxidant activity by inactivating lipid free radicals or preventing decomposition of hydro peroxides into free radicals. The Folin-Ciocalteu method is a rapid and widely-used assay, to investigate the total phenolic content but it is known that different phenolic compounds have different responses in the Folin-Ciocalteu method (Kahkonen *et al.,* 1999). Therefore, in this study, we calculated the total phenolic contents in units of mg gallic acid equivalent (GAE) of phenolic compounds as shown in table 2. The total phenolic content of goji fruits obtained in this study is in good agreement with the results obtained by Kosar *et al*., (2003) and Ionica *et al*., (2012) but significant lower than the data reported by Medina (2011) and Kosar *et al*., (2003). Le *et al*., 2007 used 95% ethanol to extract flavonoids from *L. barbarum*. This extract contained 1.56 mg quercetin equivalents/g of total flavonoid which is much higher than the result obtained in the present study. The data demonstrate the influence of the solvent used for the extraction of flavonoids on the values obtained for total flavonoid content.

Data presented in table 2 showed the antioxidant activity of goji fruits, values which correlate well with the results obtained for total phenolic and flavonoid content. The antioxidant activity of phenolic compounds was mainly due to their redox properties, hydrogen donators and single oxygen quenchers (Rice-Evans *et al.,* 1995).

Effect of storage time on the physico-chemical characteristics and antioxidant activity of the obtained goji fruits jam and jelly

Since the organoleptic and nutritional qualities are affected during thermal treatment, the conditions during this treatment should not be too aggressive. Although spoilage and pathogenic microorganisms are destroyed and endogenous enzymes are inactivated during thermal treatment, the sensorial and nutritional properties are also affected (Smout *et al*., 2003). Processing of fruits and vegetables requires pre-treatments such as peeling, rehydration and thermal processing. While these treatments confer some nutritional benefits (Deosthale, 1982), they are reported to alter the content and physico-chemical properties of components (Siljestrom *et al*., 1986).

Table 3 shows the physico-chemical parameters and antioxidant activity of goji fruits jam and jelly at different periods of storage.

Table 3. Physico-chemical characteristics and antioxidant activity of goji fruits jam and jelly

Chemical analysis	Goji fruits jam			Goji fruits jelly		
	Storage time, days			Storage time, days		
	0	5	10	0	5	10
Dry matter (g/100g)	78.35±1.75	79.66±2.43	78.41±1.86	39.5±1.52	38.9±0.97	38.01±1.08
Reducing sugar (Glucose %)	1.90±0.03	2.11±0.07	2.16±0.06	3.46±0.09	3.62±0.16	3.80±0.18
Soluble substances (Sucrose %)	70.92±2.78	70.25±2.08	69.55±1.94	41.65±1.72	40.38±1.98	39.77±1.46
pH	4.32±0.06	4.29±0.18	4.25±0.14	5.92±0.34	5.87±0.25	5.81±0.09
Total acidity (mg malic acid/100 g)	615±9.82	638±10.43	642±9..24	-	-	-
Total acidity (mg citric acid/100 g)	-	-	-	239±4.56	241±8.31	255±5.72
Titratable acidity (mg malic acid/100 g)	608±7.68	625±6.08	634±6.63	-	-	-
Titratable acidity (mg citric acid/100 g)	-	-	-	218±5.47	226±5.76	238±7.03
DPPH free radical scavenging activity (RCS %)	60.98±2.13	60.31±1.67	59.56±1.45	41.96±0.95	41.01±1.16	40.38±1.24

The data are displayed as having a mean ± standard deviation of two replications.

From the experimental data presented in Table 3 it can be observed that the storage in refrigerated conditions of the analyzed samples did not lead to significant changes of physico-chemical properties and antioxidant capacity. The total acidity is one of a number of physico-chemical parameters which affect product quality; to a large extent, acidity protects against the development of microorganisms. The storage time of the analyzed samples was not enough to cause significant decreases in pH and therefore increase of total acidity. Total soluble solids are primarily represented by sugars, with acids and minerals contributions. According to the Codex standard for jams, jellies and marmalades (CODEX STAN 296-2009), the soluble solids content for jam shall be between 60 to 65% or greater and in the case of jelly the soluble solids content shall be 40 - 65% or less. The changes in the total soluble solid values found in this study at different storage time were within the limits imposed by the standard and in agreement with the values reported by Ferreira *et al.* (2004) for quince jams and Touati *et al.* (2014) for apricot jams.

Experimental data indicated good antioxidant activity of goji fruits jam and jelly. The highest values of antioxidant activity were recorded at goji fruits jam as compared to the goji fruits jelly. This difference was due to different quantities of goji berries used to produce finished products. Large quantities of fruits goji resulted in obtaining products which recorded higher values of antioxidant activity.

Sensorial analysis of goji fruits jam and jelly

The sensory profile of the goji fruits jam and jelly was evaluated in terms of color, appearance, consistency, flavor, aftertaste, sweet taste and sour taste. Table 4 shows the sum of scores of goji fruits jam and jelly for each evaluated attribute.

Table 4. Sensory scores of goji fruits jam and jelly

Time of storage (days)	Parameter	Goji fruits jam	Goji fruits jelly
0	Color	4.28±0.25	3.71±0.12
	Appearance	3.50±0.03	4.00±0.15
	Consistency	4.42±0.32	6.14±0.24
	Flavor	3.14±0.11	2.85±0.08
	Sweet taste	2.28±0.06	2.57±0.04
	Sour taste	1.28±0.02	1.42±0.01
	Aftertaste	6.57±0.33	6.28±0.19
5	Color	4.32±0.19	3.83±0.09
	Appearance	3.56±0.18	4.00±0.19
	Consistency	4.52±0.16	6.22±0.27
	Flavor	3.32±0.06	2.79±0.14
	Sweet taste	2.35±0.04	2.63±0.23
	Sour taste	1.15±0.03	1.52±0.04
	Aftertaste	6.68±0.28	6.41±0.22
10	Color	4.22±0.24	3.84±0.15
	Appearance	3.71±0.17	3.96±0.25
	Consistency	4.34±0.14	6.48±0.38
	Flavor	3.52±0.06	2.91±0.05
	Sweet taste	2.41±0.07	2.63±0.12
	Sour taste	1.25±0.05	1.54±0.04
	Aftertaste	6.75±0.26	6.46±0.27

The data are displayed as having a mean ± standard deviation of two replications.

Both goji fruits jam and goji fruits jelly recorded scores closely to the maximum for the attributes color, appearance, consistency, aroma and sweet taste and for the attributes sour taste and aftertaste lower scores. Considering the scores recorded, goji fruits jelly showed a maximum sensory acceptability as compared to the goji fruits jam.

In the present study both goji fruits jam and goji fruits jelly showed no significant variations of sensorial parameter scores after storage at refrigeration temperature at 4° C for 10 days. Touati *et al.* (2014) reported that apricot jam showed no significant variations of sensorial parameter scores after storage, except for both spread ability and overall acceptability at 5°C and taste and overall acceptability at 37°C. The overall acceptability was less affected by storage temperature of 5°C and 25°C than 37 °C. Also, Chauhan *et al.* (2012) reported that the sensory attributes for color, appearance, flavor and overall acceptability of the coconut jam samples showed a decreasing trend, while the spread ability remained almost constant throughout the storage period of 6 months.

Conclusion

Based on the total phenolics and flavonoid contents one can consider that goji fruit (*Lycium barbarum*) has high antioxidant activity. The results of our study provide detailed information regarding the antioxidant activity, physico-chemical and sensorial stability of Goji fruits jam and jelly. Experimental data have indicated high antioxidant activity of jam and jelly, the highest values recorded at goji fruits jam. Storage at 4°C did not induce changes in the physico-chemical parameters of goji fruits jam and jelly. Sensorial quality was well preserved under the investigated storage conditions. The information obtained in the present study finds its practical application because it showed the behavior of the goji fruits jam and jelly during storage in refrigeration conditions.

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