Study regarding the production and characterization of rose petal jam enriched with Saint John`s wort (*Hypericum Perforatum*) essential oil

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Abstract

The purpose of this study was to extract the volatile oil from St. John's wort and use it to obtain a new product with improved qualities and real health benefits. In order to characterize the innovative product, several physicochemical analyses were conducted (dry matter, ash content, total sugar, total acidity, vitamin C, flavonoid content and antioxidant capacity). Also the consumer perception was followed by conducting a sensory analysis. The study revealed that the adition of *H. perforatum* essential oil in the rose petal jam imporved the vitamin C and flavoinoid content and also the antioxidant capacity of the product, meanwhile the dry matter, ash content and total sugar determinations were normal for the free pectin jam category.

Keywords: *St. John's wort, essential oil, rose petal jam, hydrodistillation, flavonoids.*

INTRODUCTION

From the plant species living on Earth (250.000 to 500.000) only 1 to 10% are used as sources of nutrients in animal and human diets (Rao *et al.*, 2002). The use of medicinal and aromatic plants was reported since ancient times, as evidenced by archaeological records from Chinese, Egyptian, Mesopotamian, Greek and Roman origins. Nowadays, the interest regarding plants' curative properties is re-emerging, motivated by the cost of complex pharmaceuticals synthesis and the consumers´ concern regarding the impact of synthetic chemicals on health and environment (Marasco *et al.*, 2007).

As a consequence, about 60% of anti-tumour and anti-infectious drugs, currently on the market or yet under clinical trials, are from natural origin. Furthermore, 11% of the 252 drugs considered as basic and essential by the World Health Organisation (WHO) are exclusively of plant origin. In the past decade, the market for herbal remedies rose at a rate of about 4-10% a year in North America and Europe (Saxena *et al.*, 2007).

Essential oils (EOs) are complex mixtures of volatile compounds with strong odor that are synthesized in several plant organs, including buds, flowers, leaves, stems, twigs, seeds, fruits, roots, wood or bark, and stored in secretory cells, cavities, canals, epidermic cells or glandular trichomes (Franz and Novak, 2010; Bakkali *et al.*,2008).The essential oils contain a large variety of volatile secondary metabolites such as terpens, terpenoids, phenolic and aliphatic derivatives. In general, EOs were previously well known as important medicinal remedies (Burt 2004) and they were also used for their fragrance and in the preservation of food. Nowadays, these characteristics have been confirmed and much more is known about their biological mechanisms, e.g. as antimicrobial ar potential anticancer agents.

Hypericum perforatum L. (*Hypericaceae*) is a perennial herb that is commonly known as St. John's Wort. The plant has been valued for its important biological and chemical perspectives and its use in the treatment of infectious diseases has been documented in ethnobotanical reports.

Most recent interest in *H. perforatum* focused on its antidepressant effects, and recently its antimicrobial activity has been evaluated against a number of bacterial and fungal strains. Various types of preparations, ointments, creams and extracts prepared with and compounds isolated from this species have been found to possess a broad spectrum of biological and pharmacological effects such as antidepressant effects, wound-healing, antiviral and antimicrobial activity.

Rosa damascena, is a rose hybrid, derived from *Rosa gallica* and *Rosa moschata*. According to ayurvedic wisdom, the rose has a cooling effect on our mind, body and emotions. The flowers are renowned for their fine fragrance, and are commercially harvested for rose oil used in perfumery and to make rose water. The flower petals are also edible. They may be used to flavor food, as a garnish, as a tisane, and preserved in sugar as rose petal jam (Harkness, P. 2003). Rose-petal jam is a Persian treat called „ gulkand", which simply means flower and sugar. The National Institute of Ayurvedic Medicine provides a list of the benefits obtained from eating gulkand.

This includes reduction of pitta and heat in the body, a reduction in eye inflammation and redness, strengthening of the teeth and gums, and the treatment of acidity.Gulkand has cooling properties, thus it is beneficial in alleviating all heat related problems like tiredness, lethargy, itching, aches and pains. It also helps in reducing burning sensations in the soles and palms (Gulkand: Nature's Coolant, published on the website of the National Institute of Ayurvedic Medicine. Retrieved February 2, 2008).

The rose petal jam is a powerful antioxidant and a very good rejuvenator and also it has a calming effect on the nervous system, thus helping in reducing stress (http://healthmeup.com/news-healthy-living/ayurvedic-home-remedies-top-11-health-benefits-of-gulkand-rose-petal-jam/21735).

This paper is a plea for the active principles of plants from the spontaneous flora (*H. perforatum* essential oil), passing by the food refinement (rose petal jam), to innovation (product made exclusively from plant essence- rose petal jam with St. John's wort essential oil).

MATERIALS AND METHODS

Essential oil extraction

For the present study St. John`s Wort was harvested in June 2012 from Ţara Lăpuşului, Maramureş County, and then naturally dried. For the extraction, ***hydrodistillation*** method was used as follows: 50 g of plant material along with 750 ml of distilled water were introduced into the distillation flask. For homogenization, a few glass beads were also added. The plant material was subjected to distillation for 3 hours. At the end of the distillation, the volatile oil obtained was collected and the volume was measured. Essential oil sample was stored in a refrigerator in tightly sealed vials until use. In order to remove any traces of water, in the storage vials, anhydrous sodium sulfate was added.

The production of the rose petal jam

Acording to CE 1200/2009 Regulation, *rosa damascena*, the rose used for the study, falls within the aromatic, medicinal and culinary herb section.

For the production of the rose petal jam an original recipe was used:200 g of rose petals after their cleaning and sorting (removal of sepals and receptacles), 25 g lemon salt (citric acid), 1 L of water and 1 kg of sugar. As a preliminary treatment, before boiling, the rose petals were mixt with the citric acid and left to soak for 2 hours after their friction. The water and sugar were put in a large pan and were slowly brought to boil, stirring constantly. Then the macerated rose petals were added and boiled until the mass looked like a gel when tested on a saucer (aprox.30-45min). After cooling, 1% of St. John`s wort essential oil was added and mixt in, then the jam was transfer into hot sterilized jars, coverd with a lid, labeled, and stored in a cool place.

In order to characterize the enriched rose petal jam the following analyzes were performed:

Moisture determination

For the determination of the moisture content was used the standard drying oven method, in wich a test portion of rose petal jam enriched with St. John`s wort, is dried at 103 ±2°C until constant mass.

In the weighing capsule, previously weighed, 2 g of properly mixed sample (jam) was added

and dried in the oven for two hours at 103 ° C. After cooling at room temperature, the vial with the sample is weighed with 0,001 g precision. The drying, cooling and weighing operations will be repeated until constant mass (the distinction between two successive weighings do not overcome 0.0006 g). All of the samples were analized in duplicates.

The moisture content was calculated as follows:

$$SU = \frac{m_2 - m}{m_1 - m} x100 \ [\%] \qquad U = 100 - SU \ [\%]$$

where:
m = mass of the empty capsule (g);
m1 = mass of the capsule with the sample before drying (g);
m2 = mass of the capsule with the sample after drying (g);
SU = solids
U = moisture

(Tofană and Mureşan, 2012)

Ash content determination

Mineral substances were determined by calcination at 550-600°C until a white ash, carbon-free was obtained.

In a porcelain crucible, previously ashed and tared, about 5 g of the sample was weighed, with a 0.002 g accuracy. The crucible with the sample was placed on a porcelain triangle over a small gas burner flame. The sample lights up without any intervention, and the combustion must not be too fast. After the flame is extinguished the sample is introduced into the furnace preheated to 550-600 ° C, until a white residue with no blackheads is obtained.

The ash content was calculated as follows:

$$Ash = \frac{m_1 - m_2}{m - m_2} x100 \ [\%]$$

where:
m1 = mass of the capsule with the ash, (g)
m2 = mass of the empty capsule, (g)
m = mass of the capsule with the sample , (g)

(Tofană and Mureşan, 2012)

Total acidity determination

The titration method was used in which the total acidity of the sample is neutralized with NaOH in the presence of an indicator.

Twenty grams of the jam, weighed on the analytical balance, was placed in a volumetric flask of 250 ml, with distilled water (up to ¾ of the volume of the flask). Then the flask was heated in a water bath at 80 ° C for 10-15 minutes, then left for 30 minutes at room temperature, stirring from time to timp. After the flask was cooled to 20 ° C its content was diluted to the mark with distilled water. After a good mixing, the content from the flask was filtered through a dry filter. Fifty ml of the filtrate was placed in an Erlenmeyer flask and titrate with 0.1 N NaOH in the presence of phenolphthalein until pink coloration appeared and lasts 1 minute.

Total acidity, expresed in malic acid was calculated as follows:

$$\text{Acidity \% (malic acid)} = \frac{Vx0,0067}{m} x100$$

where:
V = volume of NaOH 0.1N used [ml]
m = sample mass (20g jam) [g]

(Tofană and Mureşan, 2012)

Total sugars determination

From the test sample 2 g of jam were weighed and disolved in distilled water, then transfered quantitatively to a 200 cm3 volumetric flask. The content of the flask was stirred and brought to mark with distilled water, and then after a few minutes, filtered into a dry recipient. From the filtrate obtained, 100cm3 were taken and transferred into a 200cm3 volumetric flask with 2 drops of methyl orange. For hydrolysis,7 cm3 HCl (d = 1.19g/ml) were added. Then the flask was immersed in a water bath and heated to 70-75 ° C by monitoring the temperature inside the flask every 2 or 3 minutes with a thermometer. When the content reached 67-70 °C, the flask was maintained at that tempreture for 5 minutes. Then the content was cooled immediately to room temperature and neutralized with 25% NaOH solution in the presence of methyl orange until orange-yellow color and after that the content was diluted to the mark. 10 ml of thus inverted solution were transfered in a 200 cm^3 Erlenmeyer flask on which were added 25 cm^3 of cupric solution, 15 cm3 of distilled water and a few glass beads. The flask was placed on a sieve of asbest and brought to boiling point for 2-3 minutes and then maintained at moderate boiling for exactly 10 minutes. After that, the content was immediately cooled to room temperature. 10 cm3 of KI solution and 25 cm3 of H2SO4 were added. The solution obtained was titrated with sodium thiosulphate solution, using 2-3 cm^3 starch solution as indicator . The titration was considered finished when the purple color had disappeared. Also a blank is needed with the same reagents, replacing the 10 cm3 test solution with distilled water.

Total sugars content was calculated as follows:

$$n = n_1 - n_2$$

$$\%\ \text{total sugars (Zt)} = \frac{cxVxV_i x100}{V_1 x100x1000xm}$$

where:

n1 = quantity of $Na_2S_2O_3$ 0,1n, used to titrate the blank
n2 = quantity of $Na_2S_2O_3$ 0,1n, used to titrate the test sample
c = cantitatea de zahăr invertit corespunzătoare volumului de $Na_2S_2O_3$ folosit la titrare (mg)
V = volume of the volumetric flask in wich the test sample was disolved (cm^3)
Vi = volume of the flask in wich the inversion took place, (cm^3)
V1 = volume of the titration solution , (cm^3)
m = sample`s mass, (g)

(Tofană and Mureşan, 2012)

Determination of ascorbic acid

The method consist in the extraction of ascorbic acid from the test sample with a solution of hydrochloric acid (HCl) and titration with a solution of potassium iodate (KIO3) to a blue color which must persist for 30 seconds.

Ten grams of jam were weighted, with a precision of 0.01 g, and grind in a mortar together with 10 ml of 2% hydrochloric acid and 2.5 g of quartz sand or glass powder, for 10 minutes. After that, the content is passed quantitatively into a 50 ml volumetric flask and diluted to mark with a solution of HCl 2%. A part of the HCl added to bring the sample to the mark is used for washing the mortar after which added to the flask. Then the content of the flask was filetred into a clean and dry recipient. From the filtrate 10 ml were transfered in a 100 ml Erlenmeyer flask, with the addition of 5 ml of KI and 1.5 ml of 0.2% starch solution. Then the content of the flask is titrated with a 0.004 N solution of KIO3, until a blue-purple color, which must persist for 30 seconds.

The ascorbic acid was calculated as follows:

$$V_c = \frac{nxV_1x0{,}352}{GxV_2} x100 \quad [\text{mg}/100\text{g}]$$

where:

n = volume of KIO_3 used
G = test sample`s mass
V1= total volume of the extract
V2 = the volume of the extract used for the titration

(Tofană and Socaci, 2011)

Total flavonoids determination

The most common procedure used to evaluate the total flavonoid content is a spectrophotometric assay, based on the formation of a complex between the aluminium ion and the carbonyl and hydroxyl groups of the flavonoids.

Ten grams of jam, were grind with 20 ml of methanol. The homogenate was then centrifuged at 2000 rpm for a period of 15 minute at a temperature of 4oC. The supernatant was transferred into a vial, then wrapped in an aluminium foil and stored at 0-4 0C. 1 ml of the ontained extract was added to a flask containing 4 ml double-distilled water. Then, 0.3 ml 5% NaNO2 solution and 0.3 ml 10% AlCl3 solution were added. After mixing, the sample was left on repose for 5 minutes in the dark, after which 2 ml 1M NaOH solution and 6.4 ml of double-distilled water were added. The solution was mixed and the absorption was measured at 510 nm.

Antioxidant capacity determination

In order to determine the antioxidant capacity, a simple colorimetric method was used.

In a Berzelius flask were introduced 10 ml of KMnO4 0.01N, 8 ml of distilled water, 1 ml of H2SO4 20% and 1 g of jam. Right after the addition of the tested sample, a timer is turned on and the time elapsed until total fade is noted.

Sensory analysis

For the sensory analysis a simple hedonic scale with a small number of points (from 1 to 4 with 1- i don`t like it and 4 - i like it very much) was used in order to evaluate the first impression, the aspect, the flavour, the texture, the taste and the smell of the samples of jam with different amoungs of St. John`s wort essential oil added.

The analysis was conducted on 60 students of the "Petru Rares" High School from Targu Lapus. Three samples were analyzed, numbered as follows: 111 – simple rose petal jam; 222 – rose petal jam with 0.5% St. Johns Wort essential oil and 333 – rose petal jam with 1% of St. Johns Wort essential oil.

RESULTS AND DISCUSSIONS

From 50 g of dried *H. perforatum* were obtained 0,1 ml of essential oil.

Regarding the determinations made on the final product the results are presented in *Table 1.*

Table 1. Results of the physico-chemical analyzes performed on the 1% essential oil enriched rose petal jam

Determination	Results	Reference
Moisture	69,85 % Solids	68-72 % in strawberry jam **
Ash	4,86 %	Normal for free pectin products **
Total acidity	0,2 % malic acid	0,2 % malic acid in pumpkin jam **
Total sugars	53,31 %	Max 68% for jam category **
Ascorbic acid	54,18 mg/100 g	14-29 mg/100 g in roselle jam *
Total flavonoids	16,65 mg/100 g	9,42 mg/100 g in simple rose petal jam
Antioxidant capacity	18 min	> 18 min for the simple rose petal jam

Note: All the determinations were made in dublicate, data are reported as mean.
Sources: * Ashaye, O.A. and Adeleke, T.O – 2009. Quality attributes of stored Roselle jam. International Food Research Journal 16: 363-371
** http://maia.gov.md - Monitorul Oficial nr. 49-50/311 din 11.03.2008.
Reglementarea tehnică "Gemuri, jeleuri, dulceţuri, pireuri şi alte produse similare"

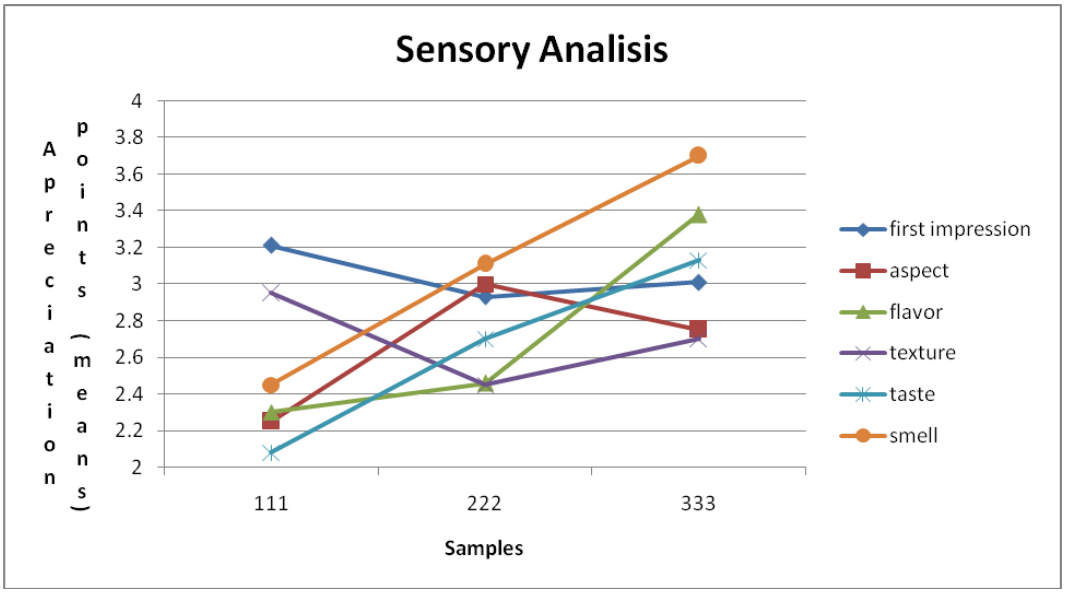

Fig. 1. Sensory analysis results

According to the sensory analysis results, most of the student have appreciated the taste, the flavor and the smell by scoring higher the enriched product (*Fig. 1*).

CONCLUSION

The yeld of essential oil extraction was only of 0,2%.

The dry matter, ash content and total sugar determinations place the new product in the free pectin jam category.

According to the results obtained for the simple rose petal jam and for the rose petal jam enriched with St. John's wort, an increase in vitamin C content is noticed for the jam with added essential oil. Also, the addition of *H. perforatum* essential oil improved the flavonoid content and the antioxidant capacity of the product.

Moreover, the sensory analysis showed that the essential oil improved the smell, taste and flavor of the jam and the rose petal jam with added essential oil was preferred by the subjects.

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