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Preliminary Results on Attributes of Distillation Products of the Rose *Rosa damascene* as a Dynamic and Friendly to the Environment Rural Crop

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Abstract

The present study aims to enrich the field of friendly to the environment rural crops, by introducing the distillation products of *Rosa damascena*, rose oil and water, and their benefits. The chemical composition of these extracts is presented. According to the results, the rose oil processes a significant number of organic compounds that render it as excellent essence oil competitive with all existing oils in the market. It is worth mentioning that the presence of 59 detectable compounds on that oil may contribute to the aromatic character of the produced oil and particularly to the forecasted centesimal proportion of the final composition. However, the exact way that they participate in the attributes of the final product should be studied further. Moreover, the analysis of the rose water indicated that its physicochemical properties are similar to those of potable water lacking microbial presence. Our results support rose cultivation and exploitation in the region of Western Macedonia in Greece, since it is friendly to the environment, and its products (rosewater, rose oil and roses) have significant qualitative characteristics, which make them highly competitive in different products and applications.

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Keywords: Rosa damascena; distillation; rose oil; rose water

1. Introduction

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Rosa damascena belongs to the family of *Rosaceae*, it is generally considered to be a hybrid and it has been cultivated for centuries. The name "Rosa" comes from the greek word "rodo" which means red, as the rose plant of the ancient world was red and according to the mythology took its colour from Goddess Aphrodite's blood [1, 2]. *Rosa damascena* is a deciduous bush. A mature plant can exceed 2.5 m in height. Its shoot is well shaped, with many brunches and dense thorns, 5-7 complex leaves, and pink or pink-red flowers with thirty petals and perfect perfume. It flourishes in semi-mountain regions and in medium constitution fields, with many rainfalls and well drained. It blooms once per year (May-June). Each plant develops several shoots from the ground which give many flowers from the second year of cultivation and afterwards. A developed plant (4 years and above) can give 500 or 600 flowers at blooming period (May-June). *Rosa damascena* is mainly cultivated in the region of Kozani, while *Rosa alba* is also cultivated but to a smaller extent.

Essential oils are volatile aromatic oily liquids isolated from plants mostly by steam distillation. Aromatic oils can be found in various parts of the plant, including flowers, leaves, seeds, fruits, wood, bark, root, balsam, and resin. The principal use of essential oils is in pharmaceutical, perfumes, cosmetics and foods [3-7]. The essential oil of *Rosa damascena* is one of the most valuable and important base material in flavor and fragrance industry [8]. It is also called "fluid gold", because of its high price [9]. The rose oil is three times more expensive than gold and almost 4.000 kg of roses are required for the production of approximately 1 kg of rose oil.

Several pharmacological effects such as therapeutic activity on premenstrual breast tenderness and inflammation reduction were reported for these species distillates. In addition, it is a valuable natural drug agent possessing bacteriostatic, gall curative, antispasmodic action; and it is also used on aroma-therapy for the treatment of cardiac diseases, and relaxation [10, 11], as well as in cosmetics and bread-candy industry [12-15]. The most common aroma concentrates of rose are rose oil and rose water, derived by a hydrodistillation method For quality control of this product, a suitable and effective analytical extraction method is required [16, 17]. Furthermore, in recent years, numerous studies have been published on the antioxidant, antibacterial activities and chemical composition of rose essential oil [18-23].

Rose oil production is mainly performed in four countries which are Bulgaria, Turkey, Morocco and Iran. It should be mentioned that in 2004 in the promotion frame of aromatic and pharmaceutical plants' culture in prefecture of Kozani-Greece, the first effort to cultivate roses began by a farm producer's team in the area of Voio, Kozani (Fig. 1a). First plants that were planted in 2004 were imported by Bulgaria. The first harvest took place in 2006 and in 2009 distillation was executed in commercial scale for the first time in Greece, using a high technology distiller. It is considered as a profitable culture compared to other common cultivates as wheat, barley, maize e.t.c. [24]. It is also significant in plant breeding for the selection of superior cultivars as well as for reproductive isolation among closely related species [25].

Its medicinal quality and commercial values range, depending on its aroma composition, are highly important and therefore, the present study aims at a detailed analysis of Kozani's area product. A series of data is presented which includes, the particular type of culture, the choice of cultivating parameters (relative land analysis of the area proposed for the particular crop), the distillation process of rose oil and rosewater. In the case of the rose oil, an analysis of its chemical composition and aromatic substances is presented, while in the case of the rosewater, the authors present an analysis of its natural-chemical and microbial characteristics. Finally, the possible use of roses in pastry industry as a product for direct consumption is studied.

2. Experimental

2.1. Sampling site and harvest

The plantation took place from November until March 2004 and at days without frost in rows, without support. The spacing was 0,80-1 m within rows and 2,5-3 m between the rows (Fig. 1b). The plants reached their higher production of flowers between the fourth and fifth year of cultivation. They were pruned in January or February of each year prior to shoot development. Fertilization took place each summer after blooming with digested manure (1kg/plant) in order to achieve best growth of blooming eyes for the next year.

Soil analysis in a representative sample of one field was performed (Table 1). A representative soil sample was chosen from one field of the whole culture area, as soil in all fields is similar. Therefore, the soil quality was analyzed in order to correlate it with the quality of the rose oil produced in this specific area. The harvest of roses took place from middle May up to the middle of June and lasted roughly 20-25 days. The harvest hours were between 5:30 - 9:00 in the morning, the period with the highest and best oil concentration. All open bloomed roses were gathered. Rose petals were kept in specific sachets and were transported as fast as possible in the distillation area (not later than an hour).

Table 1. Soil analysis

a/a	Parameter		Method
1	pH	7,26	ISO 10390:2005
2	Conductivity (μS/cm)	145,3	ISO 11265:1994
3	Mechanic constitution of soil	Characterization: ammopilodes of sand 50-70%. Sandy loam (SL)	Qualitive method modified from Thein, 1979
4	Nitrogen (%w/w)	0,074	ISO 11261:1995
5	Phosphate(mg/kg)	22,03	ISO 11263:1994
6	Potassium (mg/kg)	88.67	Practical Handbook N.T. Faithfull 2002, CABI Publishing
7	Cuprate(mg/kg)	1,02	ISO 14870:2001
8	Zinc (mg/kg)	3,18	ISO 14870:2001
9	Maganese (mg/kg)	7,09	ISO 14870:2001
10	Ferrate (mg/kg)	13,61	ISO 14870:2001
11	Boron (mg/kg)	0,18	METHODS OF ANALYSIS FOR SOILS, FAO, 2007

2.2. Distillation for the production of rose oil

A sample of 200 kg of fresh roses was inserted in a big cauldron and water was added to the top. The mixture was boiled for approximately one hour (Fig. 1c). When boiling was finished, the steam was transported with a special pipe from the cauldron to the refrigerator where it was liquefied. Thus the distillation product was collected (flower water). This distillation mixture moved from the 1st Floridian container, where a segregation of the oil from the flower water took place (very small quantity of oil ~ 15%), and leaded to a big container. This process lasted for about two and a half hours while the distilled product that is isolated was roughly 400 lt. When the big container was filled, (this happened when the above process took place twice) the process of the second boiling began, in order to end the distillation process. At this stage the distillation made a circle of transportation from the big container to an alternator and from there to another cauldron where it was boiled again. The steam was transported to a second refrigerator and was liquefied. There, it passed from a 2nd Floridian container, where it was separated

from the remainder oil (quantity 85%). The process of distillation was completed when the whole distillated from the big container finished (it took approximately half an hour).

a  b  c 

Fig. 1. (a) The rose *Rosa damascene*; (**b**) Picture of the plantation with spacing 0,80-1 m within rows and 2,5-3 m between the rows; (**c**) Presentation of the distillation process of roses for the production of rose oil

When the distillation was fulfilled, the rose oil was gathered from the two Floridian containers, (the rose distillates were gathered separately since the second one had better quality than the first one). Then it passed through a clean filter, so that any microbial load was avoided, and was placed in special glass bottles stored in cool and shady area. From the above process 50-60 gr of essential oils were isolated. The colour of rose oil was light yellow- crystal clean with perfect and pleasant and long lasting perfume.

For the determination of its chemical composition and the existing aromatic substances, a sample of approximately 10 ml was dissolved in 10% dichloromethane w/v and was analyzed in Gas Chromatograph – Mass Spectrograph (GC-MS), according to the methodology proposed by Crews, 1998 [26], with the use of "Agilent 7890A Gas Chromatograph/5975C Mass Selective Detector System", "Carrier Gas: He, Flow 0.8 mL/min, Mode Split, Split Ratio 100:1", "Heater 240 ^{o}C, Column: HP-5MS 5% Phenyl Methyl Siloxane max Temp. 325 ^{o}C: 30 m x 250 μm x 0.25 μm", and "Oven Program: 40 ^{o}C for 20 min, then 4 ^{o}C/min to 240 ^{o}C for 20 min Thermal Aux 2{MSD Transfer Line} 280 ^{o}C".

2.3. Distillation for the production of rose water

For the distillation for the rose water, 100 kg of roses were inserted in the cauldron and the necessary quantity of water to fulfill the cauldron was added. The process was the same as the rose oil distillation with the difference that the flower water was produced after 4 hours and about 650 lt of distillation product were isolated in the big container. At the second boiling stage, the difference with the previous procedure was that the distillate, which was obtained by the 2nd Floridian container, was not subjected to another circle in the big container, but on the contrary it was produced straight as rose water. A quantity of about 100 lt of rose water was gathered. An analysis for the determination of the chemical characteristics and for its microbial charge was performed by using 300 ml of the final rose water. The first 100 ml of distillate were analyzed for the presence of its microbial content. A second 100 ml sample was analyzed for its physicochemical attributes in order to be compared with those of potable water. Finally, analysis was made at the rose water in order to detect the melting point and any remains of the distillate.

3. Results and Discussion

Results on the analysis for the chemical components of the oil product are presented in Table 2. The results of rose oil analysis refer to rose oil produced by roses from all fields of the culture area. Moreover, spectrums of all detectable compounds of the previously mentioned table are quoted as these were

obtained according to the method Agilent 7890A Gas Chromatograph/5975C Mass Selective Detector System (Fig. 2). Among the 59 constituents identified by GC-MS analysis of rose essential oil, citronellol was found to be the major compound (52.44%), followed by geraniol (22.65%), nonadecane (4.43%) and ethyl-eugenol (2.59%).

Table 2. The major four compounds of rose oil as analyzed with Gas Chromatograph/5975C Mass Selective Detector.

No	RT min	Area Pct %	IUPAG name	Molecular Type	Empirical Name
1	20.79	52.44	3,7-Dimethyloct-6-en-1-ol	$C_{10}H_{20}O$	citronellol
2	21.68	22.65	3,7-Dimethylocta-2,6-dien-1-ol	$C_{10}H_{18}O$	Geraniol
3	40.27	4.43	Nonadecane	$C_{19}H_{40}$	
4	26.55	2.59	1-ethoxy-2-methoxy-4-prop-2-enylbenzene	$C_{12}H_{16}O_2$	Ethyl Eugenol

Trace amounts of other chemical compounds were also identified. These results are in agreement with previous studies on rose oil composition [18, 21, 27, 28, 29, 11]. Recently, Gochev et al. [22] reported citronellol, geraniol and nonadecane as the major constituents in the Turkish, Bulgarian and Chinese rose oil. Moreover, volatile oil samples of *R. damascena* from India [30], France [31], and Iran [32] showed an aromatic profile dominated by alcohols mainly 2-phenylethyl alcohol, citronellol, nerol, and geraniol, respectively [33]. Another work from India reported that the essential oil obtained by the distillation of fresh flowers of *R. damascene* was dominated by alcohols mainly by 2-phenethyl alcohol [34], while in the Iranian *R. damascena*, the main constituent of the essential oil was *β*-citronellol [35]. The analysis of qualitative and quantitative contistution of the rose oil, revealed the presence of 59 compounds. It could be assumed that the presence of these 59 compounds may contribute to the aromatic character of produced oil and particularly in the forecasted centesimal proportion of its final composition. This renders obviously in supremacy the qualitative content of the produced rose oil as a distillation product of the plant *Rosa damascena*. Nevertheless, the total contribution of these 59 detectable compounds in the attributes of the rose oil should be further studied.

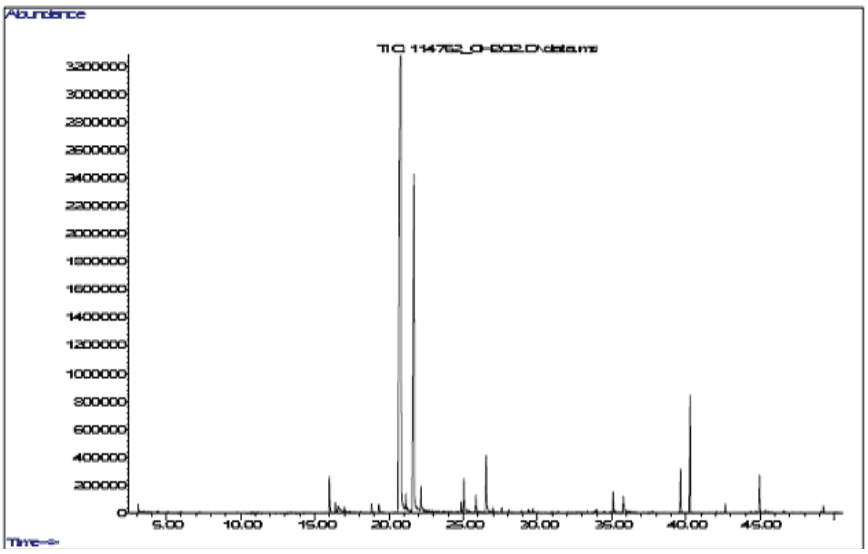

Fig. 2. Agilent 7890A Gas Chromatograph/5975C Mass Selective Detector System sample of rose oil (product of roses distillation

of plant *Rosa damascena*). Each top corresponds in an organic compound that is found in the sample and determines the constitution of sample as essence oil. It is credited that the contribution of organic compounds in the final sample is different.

In the first sample of 100 ml of rose water, the results of the microbial content analysis are showed in Table 3 and declare that there is no concern as for these parameters. It should be stressed out that analyses were also repeated after one and a half year period in the same sample with precisely the same results. These results suggest that the probability of growth of microbiological charge doesn't exist even during storage of the rose oil in the special plastic small bottle of 100 ml. In a second 100 ml sample, the results of its physicochemical attributes analysis appear in Table 4 and suggest that physicochemical attributes of rose water are similar with those of potable water.

Table 3. Results of microbiological analyses in a sample of rose water

Microbiological Analysis	**Result**	**Units**	**Report Values**	**Method**
Total Microbiological Charge 30^0 C	<10	Cfu/ml	1.000	ISO 6222:1999
Coliforms	<10	Cfu/ml	1.000	Laboratorial
E.Coli	<10	Cfu/ml	100	Laboratorial
Staphylococcus aureus	<10	Cfu/ml	100	ISO 6888
Enterococcus	<10	Cfu/ml	100	ISO 7954
Salmonella	Absence	-	-	ISO 6785
Listeria	Absence	-	-	ISO 11290-1

Table 4. Results of physicochemical attributes of analyses in a sample of rose water.

PHYSICOCHEMICAL ATTRIBUTES	**ROSE WATER**	**POTABLE WATER**
Density	0,9916 g/ml	0,9982 g/ml
Viscosity (25^OC)	1,04 Stokes	1,01 Stokes
Viscometer 75,constant 0,008		
Viscosity (25^OC)	1,02 Stokes	
Viscometer 50,constant 0,004		
pH (25^OC)	7,2	7
Total Hardness	0,2 d^0	0,3 d^0
Conductivity (25^OC)	49 μS/cm	48 μS/cm

Finally, as it is reported in Table 5 the melting point of rose water is 93 ^OC, which is close to that of potable water, while the remains after distillation is inconsequential, a fact that indicate its degree of purity. The remains of distillation are of a minimal value of 0.073g. It can be concluded that the rose water is completely safe, due to the fact that its physicochemical properties are similar to those of the potable water.

Table 5. Results of distillation in a sample of rose water

% of Distillate	5	10	20	30	40	50	60	70	80	90	95	100
mL	2.5	5.0	10.0	15.0	20.0	25.0	30.0	35.0	40.0	45.0	47.0	49.0
^{o}C	94	95	96	97	97	97	97	97	97	97	98	98

According to the analyses made in the samples of the distillate products of the plant *Rosa damascena*, it is concluded that the rose oil contains numerous organic compounds that render it as excellent essence oil, competitive to all existing market oils. All our results indicate that the rose distillate products are of high quality can be used in different sectors and further enhance the exploitation of the rose cultivation in the region of Western Macedonia in Greece. In conclusion, the results of the present study indicate an integrated system of management of production process of *Rosa damascena* in agricultural economy domain. It is worth mentioning that this process is greatly friendly to the environment and can serve as a successful choice of agricultural cultivation in a relatively difficult financial environment.

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