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The Effect of Seawater Used for Hydrodistillation on Essential Oil Yield and Composition of Oil-Bearing Rose (*Rosa damascena* Mill.)

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Abstract: Oil-bearing rose (*Rosa damascena* Mill.) is the most important rose species having a high-value volatile oil, used in the fragrance and cosmetic industries. Epidermal cells of the flower petals are the main essential oil source. During the boiling process of hydrodistillation, the essential oil in the cells diffuses through the cell walls by means of osmosis. The purpose of this research was to find out what happens when seawater or salt water used instead of distilled water for hydrodistillation. Fresh rose flowers collected at full blooming stage in the early hours of morning were distilled with pure water (control) and Mediterranean seawater using Clevenger hydrodistillation apparatus. Constituents of essential oils obtained by hydro distillation were identified with GC-FID/MS apparatus. Essential oil yield were not significantly affected by the distillation practices. However, the hydrodistillation with seawater gave a little higher yield as 0.045% than the hydrodistillation with pure water as 0.042%. A total of 23 essential oil constituents were detected by GC-FID/MS analyses. The main compounds in both rose oils distilled by tap water and seawater were citronellol, geraniol, nerol, and nonadecane. As results, hydrodistillation of oil-bearing rose with seawater provided a statistically insignificant increase in the essential oil yiled from 0.040 to 0.045%, but caused a significant decrease in citronellol rate from 41.49 to 33.56 %, and significant inceraeses in geraniol rate from 17.58 to 27.44 % and nerol rate from 6.45 to 12.21 %. The results obtained from this research should be examined in more detail at industrial scales.

Keywords: Oil-bearing-rose, essential oil, distillation, seawater treatment

1. INTRODUCTION

The oil-bearing rose (*Rosa damascena* Mill) has a pink flower with 30 petals with heavy rosy scent. Even with its high price in the world markets (over 10.000 €/kg with a value of 2017), rose oil is the most widely used essential oil in perfumery and cosmetics. The main industrial product from oil-bearing rose is rose oil, rose water, rose concrete and rose absolute, which are produced by water distillation and solvent extraction processes [1, 2, 3, 4]. Turkey and Bulgaria are the leading countries that they meet over 90 percent of the world rose oil, concrete and absolute production. Hydrodistillation with cohobation is widely used method for producing volatile oils from oil-bearing rose in the industrial process of hydrodistillation with retorts or stills [5].

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Epidermal cells of the flower petals are the main source for fragrance compounds which are complex of organic volatile molecules, e.g. monoterpenes, sesquiterpenes, aromatic alcohols, oxides, ethers, esters and aldehydes [6]. The amounts and relative contents of the scent constituents are the most important parameters which determine the quality of the rose products [7]. Monoterpene alcohols such as linalool, citronellol, nerol and geraniol, hydrocarbons such as nonadecane, nonadecene, heneicosane, heptadecane, octadecane and tricosane, sesquiterpene hydrocarbons such as α-guaiene, humulene, λ-muurolene and δ-guaiene, oxides and ethers such as methyl eugenol, esters and aldehydes such as geranyl acetate and geranial, phenols such as eugenol are among of the most important rosaceous characters found in the Turkish rose oil [1, 2, 8].

Essential oil yield of rose flowers is very poor (0.03-0.04%). About 3.5 tons or 1.250.000 fresh rose flowers are hand-picked in the early hours during the flowering season to produce only 1 kg rose oil after hydrodistillation [9]. Rose flowers produce and immediately emit the volatiles by the epidermal layers of their petals [10]. Therefore, the yield is even in intensive smelling flowers very low, and besides distillation special techniques. Although there are different kinds of traditional and modern devices for distillation technique [11], the most common and economical rose oil extraction method in the world is hydrodistillation [12].

However, the processes of the hydrodistillation importantly influence the final yield and quality of the rose oil. For example, the quality of the water (such as purity, salinity, turbidity, heavy metals, electrical conductivity, pH, etc.) used in the distillatin retorts or stillscan directly affect rose oil productivity and quality by influencing the boling temperature and steam pressure, and also releasing of the essential oil present in the oil glands or cells. Surfaceactive substances like Tween 20 alter the outer wall of some oil glands, and reduce surface tension between the water and the glands, which subsequently may result in greater oil extraction efficiency. These results confirm previous researches [13, 14, 15] on the potentially beneficial effect of Tween 20 on essential oil yield of oil-bearing rose. During the hydrodistillation, the plant material soaks up water during the boiling process and the oil contained in the oil cells diffuses through the cell walls by means of osmosis. Once the oil has diffused out of the oil cells, it is vaporized and carried away by the stream of steam [11]. The purpose of this research is to find out what happens when seawater or salt water is used instead of distilled tap water in the hydrodistillation of oil-bearing flowers.

2. MATERIAL and METHODS

The fresh flowers of *R. damascena* Mill. were hand-picked in the early morning hours of the flowering season (May and June, 2016) from Rose and Rose Products Research and Implementation Center (GULAR) at Suleyman Demirel University in Isparta province of Turkey (latitude 37^{o}45’ N, longitude 30^{o}33’E, altitude 997 m). In the research, seawater was taken from the Mediterranean Sea in Antalya/Turkey. The salinity is about 3.8% (38 g/L, predominantly sodium and chloride ions) and pH is between 7.8-7.9 at 25 ^{o}Cin the Mediterranean seawater [16]. Distilled water was produced by boiling the tap water and then condensing the vapor into a clean container, leaving solid contaminants behind. Fresh rose flowers (1 kg) and distilled or sea water (3 L) were placed in a flask (6 L) connected to the condenser of a Clevenger type hydrodistillation apparatus according to standard procedure as described in the European Pharmacopoeia [17]. After 3 hours of distillation was completed, the essentail oil and the water mixture were finally separated by decantation. The rose oil yield was measured as a percentage (v/w) on average in triplicate analyses and evaluated for statistical significanceapplying ANOVA statistical test. The essential oils distilled from both water treatments were dried with anhydrous sodium sulphate, and stored at 4^{o}C until used for analysis to determine the fragrance compounds by GC-FID/GC-MS analysis [9].

Gas Chromatography/Mass Spectrometry (GC-MS) analysis of the rose oil (50 µL of the oil was solubilized in 5 mL of n-hexane and injected in to the split mode 1/100) was performed on Shimadzu 2010 Plus GC-MS equipped with a Quadrapole (QP-5050) detector. The analysis was employed under the following conditions: capillary column, CP-Wax 52 CB (50 m x 0.32 mm, film thickness 0.25 µm); injector and detector heats, 240 ^{o}C; stove heat program, from 60 ^{o}C (10 min. hold) to 90 ^{o}C rising at 4 ^{o}C/min., and increasing to 240 ^{o}C (11.5 min. hold) rising at 15 ^{o}C/min.; flow speed, 1 psi; detector: 70 eV; ionization type, EI; carrier gas, helium (20 ml/min.); sample injected 1 µl. Identification of constituents was carried out with the help of retention times of standard substances by composition of mass spectra with the data given in the Wiley, Nist, Tutor library. The quantative analysis was conducted using Gas Chromatography/Flame Ionization Detector (GC-FID), Shimadzu Model Thermo Ultra Trace, operating at the same conditions of GC-MS. Average values were calculated and the results are expressed as the mean ± SD.

3. RESULTS and DISCUSSIONS

The essential oil yield and composition of the oil-bearing rose flowers distilled with purewater and seawater are presented in Table 1. Essential oil yield were not significantly affected by the distillation practices. However, the hydrodistillation with seawater gave a little higher yield as 0.045 % than the hydrodistillation with pure water as 0.042 % (Table1).

A total of 23 essential oil constituents were detected by GC-FID/MS analyses. The main compounds in both rose oils distilled by püre water and seawater were citronellol, geraniol, nerol, and nonadecane (Table 1). Some of the essential oil constituents which are naturally not present or very little quantities in the rose flowers can be produced either enzymic splitting or chemical degradation, occurring during the distillation from the high molecular weight and thus nonvolatile compounds present in the plants. The main constituents of rose oil, citronellol, geraniol, and nerol are products of a fermentation that takes place during the water distillation process [11]. The percentage of phenylethyl alcohol was higher in the rose oil distilled with seawater (1.84 %) than the rose distilled with pure water (1.16 %) (Table 1). Due to the solubility in water, rose water and residue water, by-products of the hydrodistillation, contained very high amounts of phenylethyl alcohol than rose oil. Therefore, probably rose water better represent the natural fragrance of the oil-bearing rose due to its very high phenylethyl alcohol content [9].

Methyl eugenol is a high value aroma chemical used in perfume and cosmetic products. However, it is not desired above a certain concentration in the essential oils due to negative side and allergic effects on human health [18]. Rose oil is one of the essential oils containing methyl eugenol that its percentage can increase up to 5.0 %, especially in the rose oils distilled from long-term fermented flowers [19] and also late harvest of full-blown flowers [20]. The percentage of methyl eugenol was lower in the rose oil distilled with seawater (1.15 %) than the rose oil distilled with pure water (1.98 %) (Table 1).

As results, hydro-distillation of oil-bearing rose with sea water provided a statistically insignificant increase in the essential oil yiled from 0.042 to 0.045%, but caused a significant decrease in citronellol rate from 41.49 to 33.56 %, and significant inceraeses in geraniol rate from 17.58 to 27.44 % and nerol rate from 6.45 to 12.21 % (Table 1). According to the interrelationships among the essential oil constituents in oil-bearing rose, citronellol content showed significant negative correlations with contents of the geraniol and nerol ($r = -0.91$ and -0.83, respectively) [14]. Similarly, in our study, the ratio of geraniol and nerol increased while citronellol ratio decreased. The volatility of the oil constituents is not influenced by the rate of vaporization but does depend on the degree of their solubility in water. As a result, the more water-soluble essential components will distil over before the more volatile but less water-soluble ones [11].

Table 1. Comparison of distilled water and seawater practices for rose oil yield and composition

Essentil oil constituents with GC-FID/MS	Rt (min.)	Distillation with	
		Distilled water (%)	Seawater (%)
Tetradecane	35.1	0.32 ± 0.03	t
Linalool	38.6	0.42 ± 0.05	0.92 ± 0.18
α-Guaiene (azulene)	42.1	1.31 ± 0.11	0.98 ± 0.15
t–caryophyllene	42.8	1.03 ± 0.08	1.37 ± 0.49
Citrenellol acetate	46.1	1.94 ± 0.39	0.39 ± 0.05
α–Humulene	47.4	0.67 ± 0.13	0.47 ± 0.21
Hexadecane	47.8	1.65 ± 0.66	1.56 ± 0.71
Z-Citral	48.0	t	0.38 ± 0.12
Germacrene-D	49.8	1.96± 0.45	1.83± 0.24
Germacrene-B	50.0	0.90± 0.10	0.70± 0.17
E-Citral	51.1	0.35± 0.05	0.85± 0.19
Geranyl acetate	51.8	4.27± 0.35	2.08± 0.23
Citronellol	52.3	41.49± 3.97	33.56± 2.81
Nerol	54.4	6.45± 0.12	12.21± 0.77
Phenylethyl acetate	55.8	0.53± 0.04	t
Geraniol	57.0	17.58± 1.34	27.44± 2.25
Nonadecane	59.5	9.68± 0.66	7.05± 0.58
9-Nonadecene	60.6	2.46± 1.18	1.62± 0.65
Phenylethyl alcohol	61.1	1.16± 0.52	1.84± 0.71
Eicosane	65.0	0.55± 0.09	t
Methyl eugenol	66.4	1.98± 0.17	1.15± 0.09
Heneicosane	70.3	2.18± 0.41	1.67± 0.17
Eugenol	74.6	1.12± 0.26	1.93± 0.33
Essential oil yield (%)*		0.042	0.045

NOTE: Rt: Retention time (min), t: trace, <0.05%, *Means vary non-significantly between treatments (P>0.05)

During the hydrodistillation of the rose flowers, essential oils are also released from the epidermal rose cells or glands in the petals by diffusion based on the principle of osmosis. By this way, the distillation water density can affect the recovery of essential oil through osmosis and diffusion processes. This research was conducted with the aim to determine the effect of salty seawater used as distillation water on essential oil content and composition of oil-beraring rose. Osmosis is the transport of water molecules flow from low-salinity side of a semi-permeable to the high-salinity side to equalize the concentration of the dissolved salts. This causes an increase of pressure on the higher-salinity side, called osmatic pressure. The volatile oil molecules in the epidermal layers of their petals glands or cells can go out faster with the water molecules by diffusion. In this research, hydro-distillation of oil-bearing rose with sea water provided a statistically insignificant increase in the essential oil yiled from 0.042 to 0.045%. Shamspur et al. [21] investigated the effects of sodium chloride concentrations in the distillation water on essential oil content and composition of oil-bearing rose. They reported that the salt treatments had no considerable effect on the quality but an increase the oil yield in the flowers treated with 22.0 g of the salt per 220 g of rose flower. On the other hand, seawater and sodium chloride added to distillation water affected the essential oil composition without affecting the essential oil content in lavander and lavandin [22].

4. CONCLUSIONS

According to previous research, it can be concluded that increasing the essential oil content and modifying the essential oil composition of *Rosa damscena*, is possible by using some additives [21] and surfaceactive substances [13-15]. This research was conducted with the aim to determine the effect of seawater used as distillation water on essential oil content and composition of oil-beraring rose. According to the results obtained, while using seawater instead of distilled pure water did not show a significant increase in oil yield, it had a certain effect on the essential oil constituents which were within normal limits of variation for the international standard ISO 9842 [23]. However, distillation equipment must be protected against possible corrosion problems caused by salt. In addition, the results obtained from this research should be examined in more detail at industrial scales.

Conflict of Interests

Authors declare that there is no conflict of interests.

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