THE EFFECTS OF STORAGE TEMPERATURE AND DURATION ON ESSENTIAL OIL CONTENT AND COMPOSITION OIL ROSE (*Rosa damascena* Mill.)

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ABSTRACT

This study was conducted in order to determine the effect of different storage temperatures (0°C and 3°C) and durations (7, 14, 21 and 28 days) on oil yield and essential oil components of oil rose (*Rosa damascena* Mill.). In this study, the rose oils were obtained by hydro-distillation in Clevenger-type apparatus and the components in the rose oil were analyzed by GC-MS. It was determined that the effect of storage temperatures on oil content wasn't significant whereas the effect of storage duration was significant ($p \leq 0.01$). The highest essential oil content was obtained at 0.043% from the petals distilled immediately after the harvest while the lowest oil content was obtained at 0.022% from the petals stored at both temperatures for 28 days, respectively. The rate of citronellol, one of the main components of rose oil, was 25.34% in the petals distilled immediately whereas it varied from 41.07 to 72.52% in the petals stored at 0°C and 3°C for 28 days. The rates of nerol and geraniol in the petals distilled immediately were 14.30% and 33.02%, respectively whereas they are 2.68%, the trace amount, and range between 0.43 and 6.74% in the stored petals, respectively. The rates of hexadecane, nonadecane, eicosane and methyl eugenol in the petals distilled immediately were determined to be lower than those of the stored petals. The optimal results in terms of its oil content and components were obtained from the rose petals distilled immediately after the harvest as well as from the petals stored at 0°C for 7 days.

Key Word: *Rosa damascena*, cold storage, essential oil content and composition

INTRODUCTION

Rosa damascena Mill. is a plant from the *Rosaceae* family in the form of an erect shrub of 1-2 m in height (Boskabady et al. 2006) and is primarily cultivated in Turkey, Bulgaria, Iran, India, Morocco, South France, China, South Italy, Libya, South Russia and the Ukraine in the world (Staikov and Kalaijiev, 1980; Weiss, 1997; Büttner, 2001). In addition, the most important production centers are Turkey (Isparta) and Bulgaria (Kazanlik). The most important products obtained from oil rose are rose oil, rose water, rose concrete and rose absolute (Lawrence, 1991). These products obtained by distillation and extraction are used in food, medicine and perfume industries as well as make-up and health products (Kürkçüoğlu and Başer, 2003; Göktürk Baydar et al. 2004; Jabbarzadeh and Khosh-Khui, 2005).

The plant of oil rose is flowered only once annually and its flowering period in Turkey lasts for almost 35 to 45 days (from the second half of May to the end of June).

The flowers, handpicked daily in the early morning ($05.^{00}$-$10.^{00}$ am) within the above-mentioned period, were brought to rose oil factories for distillation. Some of the rose flowers brought to factories were distilled immediately whereas a considerable amount of them was left in sacks for a long time due to excessive amount of flowers. Fermentation starts in the flowers left for a long time due to excessive temperature and considerable losses of oil yield and quality occur in the fermented petals (Kazaz and Kelen, 1999, Baydar and Göktürk Baydar, 2001, 2005; Baydar et al. 2008a). For instance, it was reported that the oil content in the petals distilled immediately after the harvest was 0.035%, that it became 0.030% 12 hours later, 0.027% 24 hours later and 0.025% 36 hours later in common practice, only fresh rose petals are preferred for oil production (Baydar and Göktürk Baydar, 2005). Due to the above-mentioned reasons, the cold storage of oil rose petals until distillation gains great importance in order to both prevent fermentation and decrease losses of oil yield and quality.

The most important factor in maintaining postharvest quality and extending shelf life in many products is temperature (Kader, 1987; Tano et al. 2007). In general, as storage temperatures increase, respiration rate, cell membrane permeability and symptoms of senescence increase and losses of quality occur more rapidly (Ding et al. 1998; Maalekuu et al. 2006; Jacxsens et al. 2002). Biological reactions generally increase two to three fold for every 10^{o}C rise in temperature (Beaudry et al. 1992; Exama et al. 1993, Sandhya, 2009). So far, numerous studies on the storage of many products at different temperatures and durations (Corbo et al. 2004; Tano et al. 2007; Li et al. 2008) have been conducted; however, there is no report on the cold storage of oil rose flowers. Therefore, this study aimed to determine the effects of different storage temperatures (0^{o}C and 3^{o}C) and durations (7, 14, 21 and 28 days) on the changes in oil yield and quality of oil rose flowers.

MATERIALS AND METHODS

This study was conducted in the Rose and Rose Products Research and Implementation Center (GULAR) and the Postharvest Physiology Laboratory of the Department of Horticulture in the Faculty of Agriculture at Süleyman Demirel University (Isparta, Turkey) in 2008. The rose petals of *Rosa damascena* Mill. were used as plant material in the study. Rose petals were hand-picked from the oil rose farm (latitude 37^{o} 47' N, longitude 30^{o} 30' E, altitude 1122 m) located in Isparta (Turkey) in the early morning ($06.^{00}$ am) on June 06, 2008 and brought to the laboratory in a short period of time. Some of the rose petals brought to the laboratory were immediately subjected to distillation (control) while the rest of them were reserved for cold storage experiments.

Cold storage of rose petals: After the rose petals had been placed in polyethylene bags as 1000 g each application and the bags had been closed tightly, they were stored in cold storage chambers containing a relative humidity of 90%±5 at temperatures of 0ºC and 3^{o}C for 28 days. The essential oil contents and essential oil components were determined in the petals taken out from the cold storage at 7-day intervals during storage.

Determining the essential oil content: The essential oils of rose petals were obtained in the Clevenger-type hydro-distillation apparatus. With this purpose, 1000 g of rose petals were placed in a 6 L Clevenger apparatus and subjected to distillation for 3 h after 3 L of pure water had been added on it. The quantities of rose oils obtained at the

end of distillation were measured as ml and % ratios (v/w) were determined according to standard procedure described in European Pharmacopoeia (1975).

Determining the essential oil components: The components of the oil samples were analyzed by Gas Chromatography-Mass Spectrometry (GC-MS). GC-MS analysis was performed on QP5050 GC-MS equipped with a Quadrapole detector. GC-MS analysis was carried out as follows: capillary column, CP-Wax 52 CB (50 m x 0.32 mm i.d., film thickness, 0.25 μm), oven temperature was kept at 60°C for 10 min and programmed to 220°C at a rate of 10°C/min, and then kept constant at 220°C for 10 min., total run time 60 min., injector temperature, 240°C; detector (70 eV) temperature, 250°C; flow rate for helium, 20 mL/min. 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 NIST library (Stein, 1990) and our created library.

Experimental design and data analysis: This experiment was designed in completely randomized plot design with 3 replications. Oil content means were objected to analysis of variance (ANOVA) using SAS (1998) program and differences among treatments were tested with LSD test (0.05 levels).

RESULTS AND DISCUSSION

The mean weight loss in the rose petals stored at both 0°C and 3°C during storage ranged between 0.5 and 1%, respectively. Furthermore, changes of color occurred in petals stored at 0°C for 28 days and at 3°C for 21 and 28 days; however, these changes were not measured with a color apparatus.

Oil content: The oil contents obtained from rose flowers are presented in Table 1 and Figure 1. No statistically significant differences were determined between storage temperatures in terms of essential oil content whereas significant differences were determined between storage durations. The highest oil content values were obtained from the rose petals distilled immediately after the harvest (0.043%); and oil contents of the petals stored for 7 days (0.039%) at both storage temperatures.

Table 1. Effects of different storage temperatures and durations on essential oil content of *R. damascena* Mill.

Time of Storage (Days)	Oil Content (%)		
	0°C	3°C	Mean
0	0.043	0.043	0,043 a*
7	0.040	0.037	0,039 a
14	0.030	0.029	0,030 b
21	0.021	0.024	0,023 c
28	0.021	0.022	0,022 c
Mean	0,031	0,031	

* Values within a column followed by the same letter or letters are not significantly different at the 1% level (Least Significant Difference)

It was determined that the essential oil contents of flowers decreased significantly as storage duration was extended. The mean oil contents of the petals stored for 7, 14, 21 and 28 days were determined as 0.039%, 0.030%, 0.023% and 0.022%, respectively.

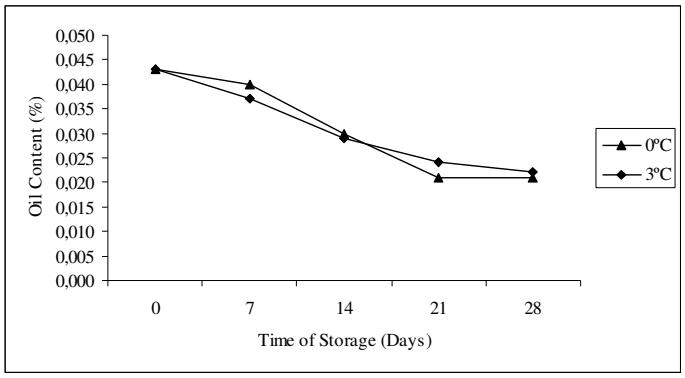

Figure 1. Effects of different storage temperatures and durations on oil content of *R. damascena* flowers

There is no any report about effects of storage on oil content and composition of oil rose flowers up to now; however, it was reported in previous studies that the oil content of the petals distilled immediately after the harvest was high and that oil content decreased significantly as the waiting duration for distillation increased (Kazaz and Kelen, 1999; Baydar and Göktürk Baydar, 2005).

Essential oil composition: The effects of different storage temperatures and durations on the chemical composition of oil rose are given in Table 2.

Table 2. The percentage composition of essential oils at different storage temperatures and durations of *R. damascena* flowers

Components	Control (0 day)	0ºC				3ºC			
		Storage Duration (Days)							
		7	14	21	28	7	14	21	28
Hexadecane	0.53	1.48	1.63	2.10	2.87	1.25	1.74	1.96	2.14
Citronellol	25.34	53.07	54.88	45.04	41.07	62.29	59.50	51.94	72.52
Nerol	14.30	2.68	0.79	0.10	0.78	1.35	0.13	0.21	t
Geraniol	33.02	6.74	2.64	1.75	2.90	3.41	1.14	1.52	0.43
Nonadecane	10.27	17.78	21.94	27.58	28.47	15.37	19.82	23.07	22.67
9-Nonadecene	2.53	3.62	4.24	4.96	4.39	0.42	2.84	3.76	4.89
Phenylethyl alcohol	0.54	0.35	0.14	t*	t	3.26	0.09	t	t
Eicosane	0.33	1.34	1.27	1.74	2.31	1.36	1.34	1.74	3.07
Methyl eugenol	0.84	1.56	1.56	2.62	3.00	1.91	2.54	2.65	1.60
Eugenol	4.16	8.03	9.00	11.09	11.96	6.78	8.19	10.09	16.60
Heneicosane	0.81	0.40	0.29	0.14	0.32	0.31	0.22	0.52	0.42
Citronellyl acetate	0.74	1.02	0.89	0.25	0.59	0.86	0.48	0.45	0.23
Geranyl acetate	4.53	0.58	0.83	0.94	t	0.28	0.41	0.74	t
Citronellol/Geraniol	0.77	7.87	20.79	25.74	14.16	18.27	52.19	34.17	168.65

* trace

In the study, it was found out that the rates of hexadecane, citronellol, nonadecane, eicosane, methyl eugenol and eugenol quite increased but the rates of nerol, geraniol and heneicosane decreased in the oils obtained from the stored petals in comparison to the oils obtained from the petals distilled immediately after the harvest (control, 0 day). It was determined that 9-nonadecane was higher than the control group except for those stored at 3°C for 7 days and that phenylethyl alcohol had irregular changes.

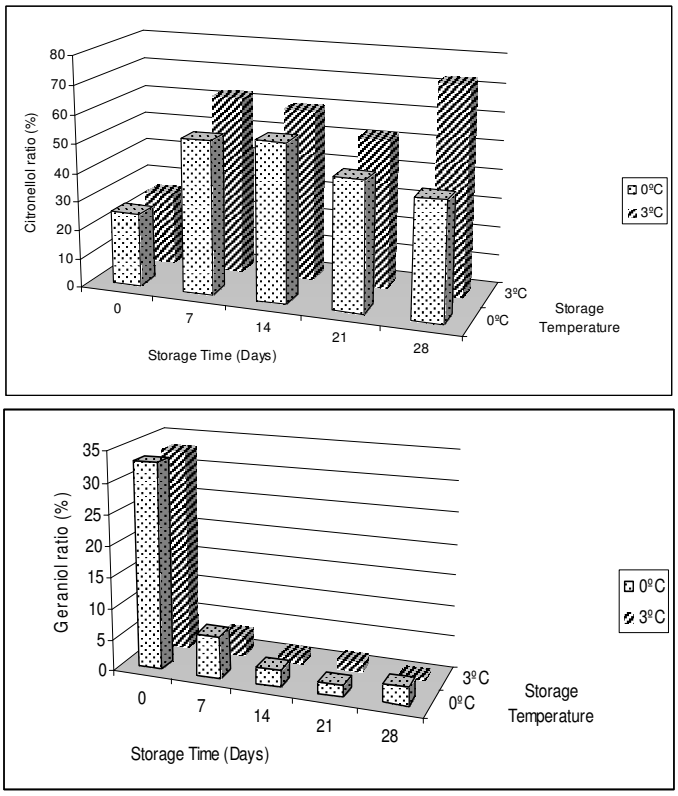

Figure 2. The percentages citronellol and geraniol of essential oils at different storage temperatures and durations of *R. damascena* flowers

It was determined that the rate of citronellol in the oil obtained from the petals distilled immediately after the harvest was 25.34% whereas it varied from 41.07 to 72.52% in those stored at 0°C and 3°C for various durations. The rate of nerol was 14.30% and the rate of geraniol was 33.02% in the petals distilled immediately while the rate of nerol was 2.68, the trace amount, and the rate of geraniol ranged between 0.43 and 6.74% in the stored petals. Methyl eugenol was 0.84% in the petals distilled immediately while it ranged between 1.56 and 3.00% in the stored petals. Being an important criterion due to its facilities in the identification of rose oil quality, the ratio of citronellol/geraniol (C/G) was determined to be quite high in the stored petals in proportion to the oil obtained from the petals distilled immediately.

Monoterpene alcohols are the basic components of rose oil and that these components provided the characteristic odour of the oil (Garnero, 1982; Anaç, 1984; Kovats, 1987; Lawrence, 1991; Başer, 1992; Bayrak and Akgül, 1994). It was reported that citronellol, geraniol and nerol, among monoterpene alcohols, ranged between 25 and 50%, 8 and 16% and 3 and 12% in Turkish rose oils, respectively (Anaç, 1984; Başer, 1992; Bayrak and Akgül, 1994) while they ranged between 22 and 55%, 14 and 18% and 5 and 10% in Bulgarian rose oils, respectively (Garnero, 1982; Kovats, 1987; Kürkçüoğlu, 1988). In the study, it was determined that the rate of citronellol, a monoterpene alcohol, increased in the stored petals in comparison to the petals distilled immediately (Figure 2) whereas the rates of geraniol and nerol decreased in the stored petals in comparison to the petals distilled immediately. Similar results were reported by Mihailova et al. (1997),

Başer (1992), Baydar and Göktürk Baydar (2005) and Baydar et al. (2008b). Hydrocarbons were the second highest components in rose oil following monoterpene alcohols (Bayrak and Akgül, 1994). It was determined that nonadecane and eicosane among hydrocarbons and methyl eugenol in the ester group increased in the stored petals in comparison to the petals distilled immediately. Similar results were reported by Kazaz (1997) and Baydar et al. (2008b). Citronellol/geraniol (C/G) ratio was used for evaluating the odor quality of rose oil (Kovats, 1987). The best odor of rose oil is produced when the ratio is between 1.25 and 1.30 (Başer, 1992). Our results showed that C/G ratio was 0.77 in the petals distilled immediately and 7.87 in the petals stored at 0^{o}C for 7 days. It was found out that this ratio increased further at other storage temperatures and durations. Baydar and Göktürk Baydar (2005) reported that C/G ratio was 0.56 in the petals distilled immediately while it was 10.30 in those distilled 36 h after the harvest. Başer (1992) and Kürkçüoğlu (1995) reported that the ratio of citronellol/geraniol in rose oils increased depending on waiting durations.

Variations in the oil contents of rose flowers and the chemical compositions of the resulting rose oils may be due to factors such as location, ecological conditions, soil, harvesting conditions, postharvest waiting times until distillation, transportation, preliminary processes, distillation technique and storage (Garnero et al., 1976; Tucker and Maciarello, 1988; Başer, 1992).

CONCLUSION

The results of this study showed that the oil contents and oil quality of the petals distilled immediately after the harvest were higher than those of the petals stored at different temperatures and durations. It was found out that the oil content did not vary depending on storage temperatures but the oil content considerably decreased as storage duration increased (particularly after storage for 7 days). In the study, it was determined that the rate of citronellol in the oils obtained from the petals distilled immediately increased during storage while the rates of geraniol and nerol decreased. The optimal results in terms of essential oil components in the study were detected in the oils obtained from the petals distilled after the harvest. On the other hand, the closest results to the control group in the petals stored at different temperatures and durations were obtained from the petals stored at 0^{o}C for 7 days.

In this study, the best results in terms of rose oil content and quality were obtained from the oils from the petals distilled immediately. Nevertheless, the petals are prevented from being distilled immediately since the flowering period of oil rose is short, excessive

amount of petals are brought to factories in a short time and these petals are left for a long time without being processed. This study indicated that the negative cases mentioned may be prevented with the storage of petals at 0^{o}C up to 7 days.

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