Bulgarian Journal of Agricultural Science, 15 (No 4) 2009, 318-322
Agricultural Academy

BULGARIAN ROSE OIL OF WHITE OIL-BEARING ROSE

N. NEDKOV[1], A. DOBREVA[1], N. KOVACHEVA[1], V. BARDAROV[2] and A. VELCHEVA[3]
[1] *Institute of Rose and Aromatic Plants, BG – 6100 Kazanlak, Bulgaria*
[2] *Toxicochemical Laboratory of the Military Medical Academy, BG – 1453 Sofia, Bulgaria*
[3] *Department of Hygiene, Medical Ecology and Nutrition of the Medical University, BG -1453 Sofia, Bulgaria*

Abstract

NEDKOV, N., A. DOBREVA, N. KOVACHEVA, V. BARDAROV and A. VELCHEVA, 2009. Bulgarian Rose Oil of White Oil-bearing Rose. *Bulg. J. Agric. Sci.,* 15: 318-322

The white oil-bearing rose (*R. alba* L.) is the second most important plant for Bulgarian rose production. Regardless of the growing interest to this plant worldwide, its essential oil has not been researched well enough. The present article provides data on physico-chemical properties of the commercial type of oil of *R. alba* L. population, 2007 harvest. Up to 214 components were found by GC and GC-MS. The major ones were heneicosane (17.45%), nonadecan (15.06%), citronellol (13.38%), geraniol (9.07%) and nerol (4.30%).

Key words: *Rosa alba* L., essential oil, GC-FID, GC/MS

Introduction

Even though there is scarce information on origin, biology and processing of white oil-bearing rose (*Rosa alba* L.), it has always been accounted for along with that on *R. damascena* Mill. The white rose is one of the old roses, parent to the Damask rose and included up to 40% in the old oil-bearing rose fields, especially in the highlands of Sredna Gora (Klisura, Svezhen village) (Topalov, 1978). The breeders strongly relied on this powerful shrub with good pedigree and tolerance to diseases in years with unfavorable climatic conditions. For a number of reasons, basically – low oil content of the flower, the fields were strongly reduced and the population almost extinct. It is present only as part of the collection of oil-bearing roses, as a material for breeding purposes, parallel to the studies on roses with different coloring (Decheva, 1977; Staikov and Kalaidjiev, 1980).

Presently, there is a revival in the interest in white rose, induced by the changing rose production profile. Farmland owners keep planting new fields. Scientific research in breeding and processing technology has come out with some prospective results (Kovacheva, 2007; Dobreva, 2008; Dobreva and Kovacheva, 2007, 2008). In line with the worldwide tendencies for searching and launching aromatic alternatives, the white rose comes forward as a typically Bulgarian economically important plant and the essential oil of *Rosa alba* L. – as Bulgarian rose oil of white rose (Anonymous, 2007; Degraf, 2003).

There are scarce and contradictory data on the composition of rose oil of white rose in literature. According to Guenther (1959), it has a high compo-

e-mail: anadobreva@abv.bg

sition of stearoptene. At the same time, the freezing point is said to be 22єC, which is within the standards for *R. damascena* Mill. and, moreover, is a function of the quantity of paraffins. The first studies of Bulgarian researchers show that the difference is only in the contents of oil in the flower (Stefanova, 1936). A number of different investigations have shown large amplitudes in the contents of citronellol (12.5ч19.7%), nerol (5.9ч13.3%), geraniol (11.6ч22.9%) and stearoptenes (22.6ч56.7%). Citronellol/geraniol ratio is in favor of either one or the other component and this is an important parameter of the oil type (Astadjov, 1989; Zolotovich and Kalaidjiev, 1967).

Therefore, obtaining accurate data on quantitative and qualitative composition of white rose oil has become an issue of immediate importance and the present investigations are devoted to this subject.

We used modern gas chromatography techniques for separation, identification and quantitative analysis: capillary gas chromatography with high separation efficiency, columns and detectors with different selection parameters – mass spectrometric for component identification (MS) and flame ionization detector (FID) for quantitative analysis. The data obtained were compared to those of *R. damascena* oil (Nicolov et al., 1977; Kovats, 1987; Jurovetz et al., 2002; Velcheva and Burdarov, 2007). A complete primary characteristic of oil was presented with physico-chemical constant values and analytical data.

Material and Methods

The material for the research was rose oil of a white oil-bearing rose population (*Rosa alba* L.), 2007 harvest. The field was started in 2003 at the Experimental Facility of the Institute of Rose and Aromatic Plants, Kazanlak, based on the usual technology, used at the institute, i.e. research was carried out at the phase of full blooming. Flowers were harvested early in the morning during the most suitable phases of development (IV-V) – semi- to full open.

Distillation was made on a semi-industrial equipment with 100 l distillation apparatuses, using the classic technology of rose flower processing. The analyzed sample was a compound of oils, extracted during the entire campaign and presented the commercial product that could be compared to rose oil batches for sale.

The physico-chemical parameters were evaluated in accordance with Bulgarian State Standard BDS ISO 9842.

The qualitative composition of the sample was analyzed by means of capillary gas chromatography with mass spectrometric detector using chemically bonded phase column HP-35,30 m X 0.25 mm and 0.4 μm film with temperature control within 45°C – 310°C.

Gas chromatograph analyses of essential oil

Quantitative analysis was done by the method of area percentage by gas chromatography (Picture 1) with flame ionization detector using chemically bonded phase capillary column Elite-WAX, 30 m X 0.25 mm and 0.4 μm film at a temperature range within 45°C – 260°C as well as chemically bonded phase column HP-35, 30 m X 0.25 mm and 0.4 μm film with temperature control within 45°C – 310°C.

The sample was introduced in a quantity of 0.30 μl via split injection (1:40) into a continuous stream of carrier gas helium with flow capacity of 0.9 ml/min.

Results and Discussion

Data from essential oil physico-chemical analysis are presented in Table 1. There is no valid standard for the essential oil of *Rosa alba* L., therefore, we

Table 1
Organoleptic and physico-chemical characteristics of essential oil of Rosa alba L. population

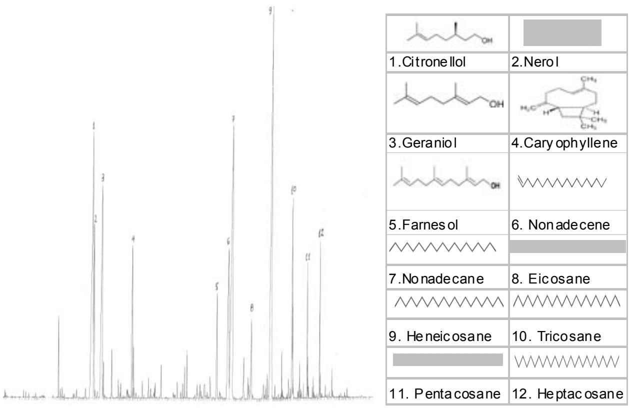
№	Index	Analysis results	Index values as per BDS ISO 9842
1	Appearance	Crystallized mass	Liquid or more or less crystallized mass
2	Color	Pale yellow	Pale yellow
3	Odor	Specific, fresh, rose	Floral, rose
4	Relative density, d20	0.8419	0.848 - 0.880
5	Refraction coefficient, nD	1.4551	1.452 - 1.470
6	Ester number	13.52	from 7 to 24

used BDS ISO 9842 for *R. damascena* Mill. essential oil as a basis for comparison.

It is obvious that appearance and physical indexes of the oil comply with the requirements of the standard for pink rose with a slight deviation in relative density. This is an indication that it can be treated as Damask rose oil if used in perfumery and cosmetic industry (e.g., in evaluation of product solubility in different other products).

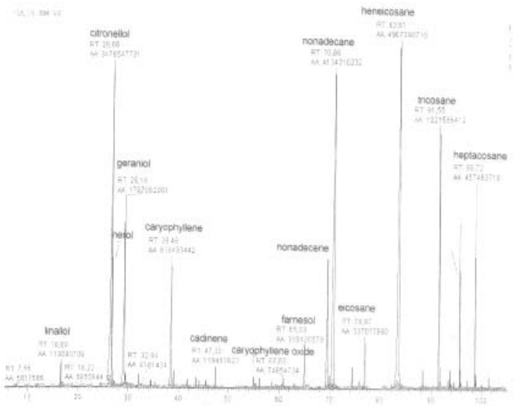
The physico-chemical constant values are a sign of high quality and stability of essential oil of white rose. As such, they are a necessary but insufficient condition for an accurate and complete characteristics. Therefore, they have to be combined with modern instrumental methods of analysis. The use of a combination of gas chromatography and gas chromatography with mass spectrum detector guarantees accurate results. This is especially important for the identification of microcomponents (linalool, rose oxides and ketones, etc.), present in minor quantities but having a very low odor detection threshold and a high contribution to the aromatic profile of the product (Ohloff, 1977). The specific profile of oil is presented with GC-FID and GC/MS chromatograms (Figures 1 and 2).

The information on oil composition, rich in components, proved that it was commensurable to that of *R. damascena* Mill. The quality of both oils was the same. The only differences were in the quantities of separate components.



Retention time, min →

Fig. 1. GC-FID chromatogram of essential oil from *Rosa alba* L.



Retention time, min →

Fig. 2. GC-MS chromatogram of essential oil from *Rosa alba* L.

Table 2
Macrocomponents of essential oil of Rosa alba L population, identified by GC-FID

Peak №	Retention time, min	Component	%
1	24.03	Citronellol	13.36
2	24.33	Nerol	4.3
3	26.33	Geraniol	9.07
4	33.88	Caryophyllene	3.07
5	55.23	Farnesol	2.35
6	58.13	Nonadecene	6.76
7	59.12	Nonadecane	15.06
8	63.9	Eicosane	1.87
9	69.03	Heneicosane	17.45
10	74.32	Tricosane	4.67
11	78.13	Pentacosane	1.48
12	81.32	Heptacosane	1.67

Mass spectral analysis found 164 components, 140 of which were identified. Flame ionization detection using the percentage method showed 214 peaks, 59 of which were identified.

Oil composition includes macro and micro-components, depending on whether their quantity is above or below 1% of the total composition. In white oil-bearing rose, the quantities of 12 components were above 1%. We focused on them. As a whole, they presented 81.14% of the total number of essential oil elements (Table 2).

The complete profile of chromatograms showed that the quantity of easily volatile components eluted from the column prior to linalool was insignificant.

Two groups of compounds were found in rose oil composition – odor carriers and odor fixators. The former are liquid, the so-called eleopten, and the latter are odorless and solid at room temperature. Terpene alcohols are the key ingredients of eleopten that comprised 26.73% of the total number of components in our case (citronellol, nerol and geraniol). Citronellol prevailed, i.e. the oil was of the citronellol type. Compared to pink rose oil, however, its quantity was much lower. The other two terpene alcohols – nerol and geraniol were present in values that were close to the standards of the Turkish and Moroccan oil of *R. damascena* Mill. (BDS ISO 9842:2004).

Of the other components, caryophyllene is an interesting one (3.07%). This is a bicyclic sesquiterpene and according to information in literature, is one of odor fixators. Unlike the other components of stearoptene, it is a liquid with high boiling temperature. This component was present in quantities of up to 0.78% in white rose oil (Baby et al., 2002; Bahaffi, 2005; Kovats, 1987).

The chromatographic profile revealed a significant presence of stearoptene elements – the solid part of rose oil that accounts for odor stability. Saturated aliphatic hydrocarbons with odd number of carbon atoms – C_{17}, C_{19}, C_{21}, C_{23}, C_{25} and C_{27} were its key ingredients. Their overall contents in rose oil amounted to 40%. They presented the major part of stearoptene – 80.8%. Of olephines, the contents of nonadecene (C_{17}) was the largest – 6.76%.

Conclusion

A complete physico-chemical analysis of essential oil of white oil-bearing rose (*Rosa alba* L.) population, 2007 harvest, was made.

GC and GC-MS detected up to 214 components. The major ones were heneicosane (17.45%), nonadecan (15.06%), citronellol (13.38%), geraniol (9.07%) and nerol (4.30%).

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Received April, 29, 2009; accepted for printing June, 5, 2009.