Chemical analysis of essential oil of *Rosa gruss-anteplitz*

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Abstract

This manuscript reports essential oil extraction from the flower petals of *Rosa gruss-an-teplitz* by distillation and solvent extraction methods, and its physico-chemical analysis. Yield of concrete oil was 0.202 and absolute oil 0.199 %, on the basis of fresh flower weight, respectively, whereas absolute oil was 9.827% on concrete oil basis. The extracted oil was yellowish in colour with refractive index 1.43 at 25 °C, congealing point 15 °C, optical rotation -32.50 + 45.25, specific gravity 0.9 at 20 °C, acid number 12 and ester number 25.22. The chemical components of the essential oil of *R. gruss-an-teplitz* were determined by Gas Liquid Chromatography (GLC). Fifteen compounds were identified in essential oil. Major constituents were phenyl ethyl alcohol (49.31%), geranyle acetate (14.78%), citronellol (12.09%), benzyl acetate (0.27%) and citronellyle acetate (12.18%).

Keywords: Chemical composition, essential oil, gas liquid chromatographic (GLC) analysis, Rosa grussan-teplitz.

Introduction

Apart from the extensive use of roses in home and garden beautification or for cut flowers some species contain valuable essential oil. The essential oil is used in wide range of perfumery, cosmetics and medicinal purposes (Younis *et al.*, 2008). The quality and quantity may vary from species to species has a great significance to develop a new local industry in Pakistan which will provide a good number of jobs to the people as well as minimize the burden of foreign exchange spent on the import of essential oil every year.

The essential oil of the roses comprises of a number of different types of constituents (Nenov et al., 1995). These constituents can be analyzed by adopting the traditional methods of quantitative and qualitative analysis (Yaseen and Oureshi, 1995). These methods, however, are time consuming and do not give accurate results. Moreover, they require a large amount of essential oil which makes them uneconomical particularly in case of plants producing very low essential oil contents such as roses. The emphasis therefore, is on the use of more modern techniques of analysis especially Gas Liquid Chromatography (Kapetanovic and Ramie, 1992). The present work was carried out to identify the different components present in the essential oil of R. gruss-an-teplitz by Gas-Liquid-Chromatography.

Materials and Methods

The project was conducted at the Department of Horticulture, University of Agriculture Faisalabad, Pakistan in collaboration with Pakistan Council of Science and Industrial Research (PCSIR) Laboratories, Lahore, Pakistan.

Collection and preparation of flower material

Healthy flowers of *R. gruss-an-teplitz* were collected from the Floricultural Research area of the Institute of Horticultural Sciences, University of Agriculture Faisalabad, Pakistan. Collection was made early in the morning to avoid the possible loss of essential oil. Flowers were placed in the paper boxes and brought to laboratory for further studies. After removing the unwanted materials like sepals, pollens and anthers, extra moisture contents of flower petals were removed by spreading in trays under shade for two days and then weighed.

Extraction and distillation method

Twenty (20) kg of flower petals (1 kg each time) were placed in the Soxhlet extraction apparatus for the recovery of rose oil using 95% pure *n*-hexane as an organic solvent. Volatile oil was sucked up by siphon in the thimble along

with the hexane through the condensers. The process was repeated 3-4 times. After whole aroma taken out from the solvent, rotary evaporator was used for the distillation of recovered solvent.

Recovery of concrete oil and its percentage

Anhydrous sodium sulphate was added in the flask to collect dissolved organic residue in the hexane. Finally, nitrogen gas was bubbled through the oil to remove last traces of hexane. Percentage yield of oil was calculated by using the following formula:

Concrete oil recovery (%) = $\frac{\text{Weight of concrete oil}}{\text{Weight of petals}} \times 100$

Absolute oil recovery and percentage

In order to remove natural waxes from concrete oil, it was dissolved in absolute alcohol and filtered through filter paper. Distillation was carried out to remove alcohol. Finally traces of alcohol were removed by passing nitrogen gas through the oil. Percentage yield of absolute oil was calculated by using following formula:

Absolute oil recovery (%) = $\frac{\text{Weight of absolute oil}}{\text{Weight of petals}} \times 100$

Physico-chemical analysis

Among the physical properties of absolute oil, first of all the colour was noted. By the use of Abbe's refractometer at 20 °C, refractive index of the oil was determined. A capillary tube containing a small amount of oil was suspended inside a large tube. A standard thermometer was also suspended in the tube along with the capillary tube. Both the tubes were cooled together in water to 5 °C. Liquid was stirred gently until it started to solidify. The congealing point was the highest temperature which was recorded during solidification. Pre-weighed specific gravity bottle was filled with 10 mL essential oil with no bubbles in the liquid weighed the bottle again and the density of the oil and then the specific gravity was calculated by using the specific formula (Hayat 1990).

Polari meter tube (10-mL volume) containing oil was placed in between analyzer and polarizer. Slowly turned the analyzer was until both halves of the field were viewed through the telescope and rotation direction was determined (Knap and Winterhalter, 2000). The acid number and ester number contents were calculated by their specific formulas (Hayat, 1990).

Absolute oil (1.5 g) was taken in a 100 mL saponification flask followed by addition of 15

mL of 95% neutral alcohol and three drops of 1% phenolphthalein solution. Aqueous solution of sodium hydroxide (0.1 N) was used for titration of free acid the alkali was added drop wise with constant rate. Red coloured first appearance was regarded as end point. The Procedure was thrice repeated and the mean value recorded. Calculation of acid number was made by using the given formula as under:

Acid No. = $\frac{5.61(\text{mL of } 0.1\text{N NaOH})}{\text{Weight of sample in grams}}$

By following the above mentioned procedure 1.5 g of absolute oil sample was taken again and 10 mL of alcoholic solution of sodium hydroxide (0.5 N) was added. An air cooled glass condenser was attached to the flask and contents were refluxed for 1 hour on a water bath. After completion of the process, apparatus was detached and cooled down for fifteen minutes at room temperature. Titration of excess alkali was conducted against the standardized aqueous HCl (0.5 N). The ester number was calculated by using the formula as under:

Ester No. =
$$\frac{28.05 \text{ a}}{\text{Weight of sample in grams}}$$

Whereas a = Milliliters of 0.5 N NaOH used in the saponification.

Chromatographic and chemical composition analysis

Gas-Liquid-chromatography (GLC) was used for analysis of various constituents of essential oil *R. gruss-an-teplitz*. To obtain the chemical composition, sample of the solvent extracted oil was injected in the chromatograph with a syringe and was run as per set conditions. The chromatogram obtained was then compared with the chromatogram of the standard compounds.

Results and Discussion

Yield of concrete oil

The oil obtained from 20 kg of *R. gruss-anteplitz* showed a yield of 40 g that was 0.202% on the basis of flower weight. The yield obtained was in harmony with that of *Rosa centifolia* and *R. damascena* (0.21-0.24%) (Khan and Rehman, 2003). The oil yield was somewhat lower than *Rosa gallica* (0.29%) as reported by Nofal *et al.* (1982) and significantly greater than *Rosa centifolia* (0.107%) as was observed by Hayat (1990).

Yield of absolute oil

Absolute oil yield recovered from concrete oil for the *R. gruss-an-teplitz* was 3.98 g, which was 9.83% on the concrete oil basis, while 0.0199 % on the petal weight basis. These findings are in line with Khan and Rehman (2005) who reported the same yield from the concrete oil of *R. centifolia* on petal weight basis. The yield (0.34%) is lower than *R. gruss-an-teplitz* as reported by Mumtaz *et al.* (2007).

Physical properties

R. gruss-an-teplitz showed yellowish oil colour somehow different to the yellowish brown colour of *R. centifolia* and *R. damascena* (Mumtaz *et al.*, 2007; Khan and Rehman, 2005). However, Shabbir *et al.* (2009) reported the yellowish brown oil colour of *R. gruss-an-teplitz*. Refractive index of *R. gruss-an-teplitz* was 1.43 at 25 °C (Poucher 1974), it was less as reported in previous findings with different rose species at different temperatures (Khan and Rehman, 2005; Shabbir *et al.*, 2006; Mumtaz *et al.*, 2007).

The congealing point of *R. gruss-an-teplitz* oil was 15 °C (Shabbir *at al.*, 2009); optical rotation was -32.50 + 45.25 (Mumtaz *et al.*, 2007) and specific gravity was 0.9 at 20 °C (Khan and Rehman, 2005).

Chemical properties

Acid number of *R. gruss-an-teplitz* was 12 (Khan and Rehman, 2005; Mumtaz *et al.*, 2007) and ester number was 25.17 (Shabbir *et al.*, 2009).

Chemical constituents

Quantity of geraniol was 2.98%, lower than that of 12.7, 3.42, 3.60 and 2.97% as recorded by Hayat (1990); Mumtaz et al. (2007); Younis et al. (2008) and same as was documented for R. centifolia by Khan and Rehman (2005). Eugenol with 3.29% was greater than 2.62% and 1.68% as reported by Havat (1990) and Khan and Rehman (2005). Rhodinol with 4.05% was less than 23.98% of Rosa bourboniana (Khan and Rehman, 2005). Citronellol was 12% was more than 1.89% (Hayat, 1990). Linalool was 12.68% was greater than previously recorded quantity in many studies (Khan and Rehman, 2005; Mumtaz et al., 2007; Younis et al., 2008). Benzyl phenyl acetate 4.0941% a new component and had not been reported in the cited works of this article. Other important constituents were phenyl ethyl alcohol, benzaldehyde, benzyl acetate, citronellyl acetate, benzyl alcohol, rhodinyl acetate, geranyl acetate, phenyl ethyl acetate and phenyl ethyl formate (Table 1).

Conclusion

R. gruss-an-teplitz gave abundant yield of essential oil containing several significant chemical ingredients and henceforth is very appropriate for the essential oil extraction on lucratively commercial scale.

	Table 1: Chemical	components of essential	oil from Rosa	gruss-an-teplitz.
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	Components	(%)
1.	Geraniol	2.99
2.	Eugenol	3.29
3.	Rhodinol	4.05
4.	Citronellol	12.1
5.	Linalool	1.69
6.	Benzyl Phenyl Acetate	4.1
7.	Phenyl Ethyl Alcohol	49.31
8.	Benzaldehyde	1.94
9.	Benzyl Acetate	0.27
10.	Citronellyl Acetate	0.42
11.	Benzyl Alcohol	2.7
12.	Rohdinyl Acetate	3.7
13.	Geranyl Acetate	14.7
14.	Phenyl Ethyl Acetate	1.02
15.	Phenyl Ethyl Formate	2.5

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