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# Physiochemical Revelation of Essential Oil of Two *Jasminum* Species '*grandiflorum*' and '*officinale*'

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## Abstract

Quantitative and qualitative analysis of essential oil of two Jasminum species '*grandiflorum*' and 'officinale' was carried out. Essential oil was extracted by n-hexane solvent extraction and hydro-distillation methods. Concrete oil recovery on fresh petal weight basis from *J. grandiflorum* and *J. officinale* was 0.26% and 0.30%, absolute oil recovery was 0.14% and 0.16% and essential oil yield from hydro-distillation was 0.011% and 0.14% respectively. The color of oil extracted from both the species was reddish brown. The specific gravity of *J. grandiflorum* was 0.9120 and *J. officinale*, was 0.9100 at 30°C and congealing point of the concrete oil of both species was 55°C. In *J. grandiflorum,* a total of 40 compounds were identified. The major components were 'acetic acid, phenyl methyl ester' (12.20%), 'linalool' (11.04%), '3-hexenyl benzoate' (10.50%) and 'alpha cadinol' (6.70%). In *J. officinale* total 45 compounds were identified. The major components were 'benzyl benzoate' (10.10%), 'cis-jasmone' (9.64%), '3-hexenyl benzoate' (8.84%), 'acetic acid, phenyl methyl ester' (9.0%), 'linalool' (7.11%), 'isophytol' (6.39%) and 'alpha-farnesene' (5.39%). Minor compounds of *J. grandiflorum* were '3-hexenyl acetate', 'benz acetaldehyde', 'alpha farnesene', 'farnesal', 'tau-muurolol', 'benzyl benzoate', 'nerolidol acetate', 'palmitic acid', 'geranyl linalool', 'methyl linolenate', '9-tricosene', 'methyl aracrisate' and 'heptacosane'. Minor compounds of *J. officinale* were 'geraniol', 'cis-3-hexanyl benzoate', 'alpha cadinol', 'phenyl ethyl benzoate', 'linolenic acid', 'phytol', 'tricosane' and 'heptacosane'. Commercial cultivation of these species for essential oil production can provide successfully raw material for industrial purpose.

## INTRODUCTION

An increasing popularity of essential oils and extracts of numerous flowers like rose, jasmine, tuberose etc. in high grade perfumes and aroma therapy is observed (Sahoo, 2001), because essential oils obtained from aromatic plants serve the humanity extensively in cosmetics, pharmaceuticals, aerated water, confectionary, perfumery, disinfectants, ice-creams, sweets, candies, snacks, chewing gums, tobacco and various related products (Skaria *et al.*, 2007). They also provide raw materials for the manufacturing of many important industrial products. Since the middle ages, these oils

had been widely used for bactericidal, virucidal, fungicidal, anti-parasitical, insecticidal, medicinal and cosmetic applications (Bakkali *et al.*, 2008). The sheer number of flowers needed to produce the essential oil makes it one of the most expensive oil (Jones, 2010). Among many odorants, *Jasmine* is a principal perfume with a beautiful scent containing more than 200 components ranging from pleasant jasmine lactone to extremely unpleasant indole (Mitsuri, 1997).

In the present study, two methods of distillation viz. solvent extraction and hydro-distillation were used for the extraction. Both the methods have their own advantages and disadvantages. Handa *et al.* (2008) proposed that solvent extraction is suitable for expansive, delicate and thermally unstable materials like jasmine, tuberose and hyacinth. Solvent extraction involves the use of costly apparatus like rotary vacuum evaporator and centrifuge equipment with high running expenses (Gunkel *et al.*, 2010). This method initially yields concrete oil from which absolute is obtained. In many industrial products, this concrete is used as such. Hydro-distillation is still being used as the principal method for the preparation of essential oils. This is a cheap method and does not involve costly apparatus. For the evaluation of quality and determination of the constituents of essential oil, certain physical and chemical properties were analyzed.

In Pakistan, the total cost of imports of essential oils, perfumes and flavor materials was Rs. 10,158 million in 2003-04. Data about the export of such materials are silent. The climate of Pakistan is favorable for the cultivation of *Jasminum* species. Some species of *jasminum* are already under cultivation in various parts of the country, but only for their ornamental value. Therefore, present study was initiated to extract the essential oil from *J. grandiflorum* and *J. officinale* and their physico-chemical analysis for making further recommendations for growers and manufacturers.

## MATERIALS AND METHODS

Fully open flowers of both species were harvested from 'Rawalpindi' area before sunrise and were shifted immediately to the laboratory for extraction of essential oil. Sepals, leaves and other undesirable plant parts were separated from the flowers to avoid contamination in the final product. Flowers were weighed after cleaning. The petals were put in distillation flasks directly for further working. Essential oil extraction was carried out by solvent extraction and hydro-distillation.

Calibrated glassware and chemicals of analytical grade were used. All the apparatus was thoroughly washed and dried every time before conducting the extraction and distillation. The extraction of oil in solvent extraction method was carried out using n-Hexane as organic solvent (hydrocarbon solvent) with Soxhlet apparatus. Essential oil was extracted in batches of 1 kg each time. Thimbles of Whatmann filter paper No. 40 were filled tightly with flowers and kept in jacket of Soxhlet apparatus, n-Hexane was added in the flask and boiled at a temperature of 55 to 60$^{o}$C. On boiling vaporized n-Hexane passed through flower-containing thimbles in the jacket and condensed by the condenser. The whole material came back in the flask after condensation. This process was repeated 8 to 10 times to get maximum essential oil recovery. When approximately the entire aroma of flowers was taken out by the solvent, the process of distillation was carried out in rotary evaporator leaving a resionid or concrete. The result was concrete oil which included non-aromatic waxes, pigments and highly volatile aromatic molecules. This concrete was further processed to make it pure. In hydrodistillation the plant material was immersed in water in the still and heated to boiling point using external

heat source. The boiling hot water drew out the oil. The vapors were allowed to condense and the oil was then separated from the aqueous phase (Houghton and Raman, 1998). This method produces a finer, more complete product, and shocks the plant material less (Ackerman, 2001). The left-over concrete and absolute oil, kept in tightly packed Falcon tubes and HPLC vials, were stored in refrigerator at low temperature of 2-4°C.

For the evaluation of quality and determination of the constituents of essential oil, certain physical and chemical properties were analyzed. Color of absolute oil was determined using spectrophotometer. The instrument recorded transmittance measurement between 400 and 700nm. For the determination of specific gravity pre-weighed 10 ml specific gravity bottles were filled with absolute oil leaving no air bubbles, and then weighed. The density of oil was computed with the following formula.

Density = Weight / Volume

SG = Density of liquid at 20°C / density of water at same temp.

In case of mixtures, such as essential oils, congealing point was determined instead of melting point. The oil was super cooled so that upon congealation liberation of heat occurs with immediate crystallization (Gunkel *et al.*, 2010). About 10 ml of absolute oil was placed in a dry test tube of 20 mm diameter. This was cooled in water the temperature of which was about 5°C lower than the supposed congealing point of the essential oil. The inner walls of the tube were rubbed with a thermometer quickly up and down in the oil. The temperature was noted constantly. There was a rise in temperature which soon approached a constant value. That value was taken as congealing point of the oil. The process was repeated many times for accuracy. The refractive index of the oil was determined at room temperature of 25°C using Abbe's Refractometer.

Gas chromatography-mass spectrometry (GC-MS) analysis was performed by Agilent Technologies 6890N-5975B system, with data acquisition parameters as follows. Carrier gas was Helium with a flow rate 1.0 mL/min, constant flow mode; injection volume 0.1 µL, inlet temperature 250°C; Agilent Technologies HP-5MS 30 m 0.25 µm column. Temperature program: 50°C for 1 min, 5°C/min to 100°C, 9°C /min to 200°C, hold 7.89 min; transfer line temperature 280°C; electron ionization, electron energy 70 eV, scan mode, mass range 35-400 Da, quadrupole temperature 150°C, source temperature 230°C. Acquired data were analyzed by Agilent Technologies MSD Chem Station software in conjunction with AMDIS (Automated Mass Spectral Deconvolution and Identification System) and NIST MS Search software. Two different mass spectra libraries were used for mass spectra identification: Wiley Registry of Mass Spectral Data 7th Edition (338000 spectra, 289000 unique compounds), NIST/EPA/NIH Mass Spectral Library 05 with 190825 spectra, 163198 unique compounds. The compound identification was finally confirmed by comparison of their relative retention indices with literature values (Adam, 2001; Mimica-Dukic *et al.*, 2003; Rout *et al.*, 2007; Vagionas *et al.*, 2007; Grbovic, *et al.*, 2010; NIST Standard Reference Data No. 69, 2011). All the experiments were conducted in triplicate (except GC-MS analysis) and the data were reported as means ± standard deviation (SD) (Steel *et al.*, 1997).

## RESULTS AND DISCUSSION

The oil obtained from hydro-distillation was almost pure and was directly subjected to physical and chemical analyses. However, the quantity obtained was less as compared to solvent extraction (Table 1). Color of essential oil was reddish brown for both the species (Table 2). Gilbert *et al.* (1999) also found jasmine oil to be reddish brown

in color. Guenther (1952) reported the colour of jasminum absolute as a clear yellow-brown liquid. It may be due to the reason that factors responsible for affecting yield also affect the physical and chemical qualities of essential oil. Therefore, minor variation in results is not uncommon in the available literature. Refractive index for *J. grandiflorum* and *J. officinale* was 1.371 and 1.372 respectively (Table 2). Guenther (1952) observed refractive index of jasmine to be 1.4822 at 20°C. Weiss (1997) reported refractive index of concrete of *J. grandiflorum* to be 1.4750. The varying results may be seen in the context of different climatic and geographical conditions.

Congealing point for both the species was 55°C (Table 2). The highest temperature at which an essential oil is solidified is called its congealing point. Other researchers have reported their results of congealing point which vary from one another to some extent, and vary to large extent for concrete and absolute. The congealing point of concrete is always higher because it contains waxes and other odourless substances.

Determination of specific gravity is also an important physical parameter of essential oils. In the present study, specific gravity of *J. grandiflorum* and *J. officinale* was 0.9250 and 0.9100 at 20°C respectively. In comparison Guether (1992) reported specific gravity at 15°C (without giving the name of variety) to be 0.931 to 0.970; Weiss (1997) reported specific gravity of concrete of *J. grandiflorum* to be 0.9000 at 30°C. The physical properties of essential oils are strongly influenced by variety, degree of maturity, seasonal variations, seasonal rainfall, and method of extraction and yield of oil (Richard *et al.*, 1971).

Total number of compounds identified by GC-MS for *J. grandiflorum* and *J. officinale* having less than 1 percent were 13 and 24; total number of compounds having a percentage of 1 to 5 was 23 and 14, and total number of compounds having a percentage of more than five was 4 and 7 respectively (Table 3 &4). Concrete oil recovery from *J. grandiflorum* and *J. officinale* was 0.26% and 0.30%, while absolute oil recovery from this concrete was 0.14% and 0.16% respectively (Table 1). Yield of essential oil obtained from hydrodistillation was 0.01% and 0.04% respectively. Essential oil yield was 0.14% to 0.16% in n-hexane solvent extraction (Table 1). It means that absolute oil recovery from concrete was about 52% to 53%, i.e., almost half of concrete. Our findings support the results of Younis *et al.* (2011) who observed about half absolute oil recovery from the concrete of Arabian jasmine. The results are also in confirmatory with those of Weiss (1997) who reported the concrete yield of 0.33% for *J. grandiflorum*. Hydrodistillation is a widely used method for the extraction of essential oils, particularly for delicate flowers like those of jasmine and roses. In the present study, this method gave oil yield of 0.01% and 0.14% for *J. grandiflorum* and *J. officinale* respectively. Essential oil yield obtained by hydrodistillation is usually low as compared to solvent extraction. Waheed-ur-rehman (2006) also recorded low essential oil recovery (0.207%) by hydrodistillation as compared to solvent extraction. Researchers have gained quite variable results with respect to oil production. This is so because many factors may affect the oil yield from flowers. These factors may include addition of fertilizer to the soil, time between picking and processing of flowers, method of handling the picked flowers till processing, equipment usage and agronomic practices (Weiss, 1997), cultivar chosen, time of flower picking, skill of labor engaged in picking, stage of flowers at picking, method of extraction, weather, month of harvest (Gunkel, 2010).

In *J. grandiflorum,* a total of 40 compounds were identified. The major components were acetic acid, phenyl methyl ester (12.20%), linalool (11.04%), 3-hexenyl

benzoate (10.50%) and alpha-cadinol (6.70%). In *J. officinale* a total of 45 compounds were identified. The major components were: benzyl benzoate (10.10%), cis-jasmone (9.64%), 3-hexenyl benzoate (8.84%), acetic acid, phenyl methyl ester (9.0%), linalool (7.11%), isophytol (6.39%) and alpha-farnesene (5.39%). It was observed that *J. grandiflorum* and *J. officinale* had 4 and 7 major compounds, respectively. Weiss (1997) also found the major constituents of isophytol (7.41 to 8.37), phytol (9.17 to 24.76%) and linalool (5.74 to 6.54%). Other major compounds reported by him as benzyl acetate and phytol acetate could not be found in our results. Skaria *et al.* (2007) reported linalool and jasmine as major component of *J. officinale* essential oil. Minor compounds of *J. grandiflorum* were: 3-hexenyl acetate (2.17%), benz acetaldehyde (1.81%), alpha farnesene (1.76%), farnesal (1.77%), tau-muurolol (3.59%), benzyl benzoate (1.28%), nerolidol acetate (1.24%), palmitic acid (2.67%), geranyl; linalool (4.23%) methyl linolenate (2.68%), 9-tricosene (3.08%), methyl aracrisate (1.12%) and heptacosane (1.35%). Five compounds could not be identified by their common names. Minor compounds of *J. officinale* were geraniol (1.93%), cis-3-hexanyl benzoate (1.31%), alpha cadinol (1.43%), phenyl ethyl benzoate (1.04%), linolenic acid (3.13%), phytol (2.37%-3.87%) tricosane (1.41%) and heptacosane (1.47%). It can be viewed that some minor compounds are major compounds in other species. For ease of understanding a list of common compounds which were present in two varieties under study has also been prepared. A perusal of this list will make it easy to compare the percentages of different components present in both the varieties. Overall, 15 components were found common in both the species.

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**Table 1:** Oil yield of *J. grandiflorum* and *J. officinale* obtained from two methods of extraction.

| **Type of oil extracted** | ***J. grandiflorum*** | ***J. officinale*** |
|---|---|---|
| Concrete oil yield (g) from solvent extraction | 2.62 (0.26%)<br>SD value: 0.108 | 3.01 (0.301%)<br>SD value: 0.095 |
| Absolute oil yield (g) from solvent extraction | 1.406 (0.14%)<br>SD value: 0.097 | 1.61 (0.16%)<br>SD value: 0.096 |
| Essential oil yield (g) from hydrodistillation | 0.11 (0.01%)<br>SD value: 0.01 | 0.141 (0.014%)<br>SD value: 0.0096 |

**Table 2:** Physical characteristics of essential oil of *j. grandiflorum* and *j. officinale.*

| **Physical characteristic** | ***J. grandiflorum*** | ***J. officinale*** |
|---|---|---|
| Specific gravity at 30°C | 0.9120 at 30°C ± 0.0001 | 0.9100 at 30°C ± 0.0001 |
| Congealing point of concrete | 55°C ± 1.732 | 55°C ± 1.732 |
| Refractive index | 1.371 ± 0.001 | 1.370 ± 0.003 |
| Color | Reddish brown | Reddish brown |

**Table 3:** Chemical constituents of essential oil of *J. grandiflorum* identified by GC-MS.

| **Sr. no.** | **Retention time (min)** | **Name of compound** | **%** |
|---|---|---|---|
| 1. | 4.283 | Hexanal, 2-ethyl | 0.894 |
| 2. | 6.648 | Benz aldehyde | 0.883 |
| 3. | 7.405 | Methyl heptenone | 0.835 |
| 4. | 8.080 | 3- Hexenyl acetate | 2.718 |
| 5. | 9.081 | Benz acetaldehyde | 1.815 |
| 6. | 9.869 | Alpha,-methyl-alpha,-[4-methyl-3-pentenyl] oxiranemethanol | 1.114 |
| 7. | 11.264 | Linalool | 11.044 |
| 8. | 13.141 | Acetic acid, phenyl methyl ester | 12.200 |
| 9. | 13.597 | 3-cyclohexene-1-methanol,alpha,alpha,4-trimethyl(S) | 0.724 |
| 10. | 15.436 | Cyclopropanecarboxylicacid,2-phenylethyl ester | 4.317 |
| 11. | 16.550 | Oxalic acid, di(2-phenylethyl)ester | 1.532 |
| 12. | 18.577 | Geranyl acetate | 0.670 |
| 13. | 19.283 | Anthranilic acid | 0.687 |
| 14. | 21.736 | Alpha.farnesene | 1.760 |
| 15. | 22.042 | Germacrene D-4-ol | 0.895 |
| 16. | 22.105 | Cadina-1(10),4- diene | 0.681 |
| 17. | 22.361 | 2,6,10-dodecatrienal, 3,7,11-trimethyl-(EE) | 1.774 |
| 18. | 22.836 | Farnesal | 2.574 |
| 19. | 23.600 | 3-hexenyl benzoate | 10.501 |
| 20. | 23.969 | E-2- hexenyl benzoate | 1.930 |
| 21. | 24.732 | 4aH-cycloprop(e)azulen-4a-ol,deca hydro-1 1,4,7 tetra methyl-[1aR-(1a.alpha,4 beta, 4a. beta,7 alpha 7a beta, 7b alpha)] | 1.661 |
| 22. | 25.151 | Tau-muurolol | 3.596 |
| 23. | 25.620 | Alpha.cadinol | 6.709 |

| | | | |
|---|---|---|---|
| 24. | 26.402 | Leden oxide | 0.991 |
| 25. | 27.666 | Benzyl benzoate | 1.286 |
| 26. | 27.834 | Isoaromadendrene epoxide | 0.931 |
| 27. | 29.129 | Nerolidol acetate | 1.248 |
| 28. | 29.498 | 2-propanamine, N(phenyl methylene | 1.252 |
| 29. | 30.887 | Palmitic acid | 2.670 |
| 30. | 32.939 | Geranyl linalool | 4.232 |
| 31. | 33.814 | Benzene propionic acid, 10-oxoptricyclo[4.2.1 1(2,5) deca-3,7-dienyl ester | 1.471 |
| 32. | 33.996 | 7,10-octadecadienoic acid, methyl ester | 1.592 |
| 33. | 34.177 | Methyl linolenate | 2.684 |
| 34. | 34.603 | Octadecanoic acid, methyl ester | 2.012 |
| 35. | 35.697 | 1-heptatriacotanol | 0.841 |
| 36. | 37.149 | 9-tricosene | 3.088 |
| 37. | 37.468 | Heptacosane | 0.909 |
| 38. | 37.949 | Methyl aracrisate | 1.121 |
| 39. | 40.658 | Heptacosane | 1.355 |
| 40. | 44.136 | Heptacosane | 0.803 |

**Table 4:** Chemical constituents of essential oil of J. *officinale* identified by GC-MS.

| **Sr. no.** | **Retention time (min)** | **Name of compound** | **%** |
|---|---|---|---|
| 1. | 6.592 | Benzaldehyde | 0.284 |
| 2. | 11.189 | Linalool | 7.112 |
| 3. | 13.391 | Acetic acid, phenyl methyl ester | 9.004 |
| 4. | 14.173 | α terpineol | 0.954 |
| 5. | 14.817 | Cis- Geraniol | 0.497 |
| 6. | 15.543 | Geraniol | 1.931 |
| 7. | 16.944 | Cyclopentanone, 2-(2- methylpropylidene) | 0.254 |
| 8. | 18.383 | Phenol, 2-methoxy-3(2-prophenyl) | 2.846 |
| 9. | 19.671 | Cis-jasmone | 9.646 |
| 10. | 20.247 | Isopentyl alcohol | 0.278 |
| 11. | 20.616 | Heptadecane 2,6,10,15-tetramethyl | 0.667 |
| 12. | 21.179 | Benzoic acid | 0.694 |
| 13. | 22.055 | Alpha-farnesene | 5.381 |
| 14. | 22.530 | Farnesal | 0.345 |
| 15. | 22.899 | Geranyl linalool | 0.466 |
| 16. | 23.712 | 3-hexenyl benzoate | 8.848 |
| 17. | 24.069 | Cis-3-hexenyl benzoate | 1.318 |
| 18. | 24.463 | Caryophyllene oxide | 0.339 |
| 19. | 25.076 | Tau-muurolol | 0.973 |
| 20. | 25.401 | Alpha cadinol | 1.436 |
| 21. | 28.066 | Benzyl benzoate | 10.103 |
| 22. | 29.104 | 9-eicosyne | 0.262 |
| 23. | 29.330 | Per hydro farnesyl acetone | 1.411 |

| | | | |
|---|---|---|---|
| 24. | 29.680 | Phenylethyl benzoate | 1.043 |
| 25. | 29.949 | 9,12-octadecadienoic acid ZZ phenyl methyl ester | 1.761 |
| 26. | 30.218 | 1 chloro octadecane | 0.315 |
| 27. | 30.931 | Palmitic acid | 3.079 |
| 28. | 31.544 | Iso phytol | 6.397 |
| 29. | 31.782 | 1-heptatriacotanol | 0.274 |
| 30. | 33.014 | Geranyl linalool | 4.217 |
| 31. | 33.721 | [2-(hydroxyphenyl)cyclopropyl](phenyl) methanone | 0.415 |
| 32. | 34.190 | Linolenic acid | 3.131 |
| 33. | 34.484 | Phytol | 2.371 |
| 34. | 34.609 | Methyl stearate | 0.701 |
| 35. | 34.834 | E,E,Z 1,3,12-nonadecatriene-5,14-diol | 0.269 |
| 36. | 35.003 | E,E,Z 1,3,12-nonadecatriene-5,14-diol | 0.399 |
| 37. | 35.416 | Z 3,7,11,trimethyldodec-2-enoic acid, methyl ester | 0.366 |
| 38. | 36.317 | Phytol | 3.876 |
| 39. | 37.080 | 9-tricosene | 0.727 |
| 40. | 37.530 | Tricosane | 1.417 |
| 41. | 40.639 | Pentacosane | 0.816 |
| 42. | 44.236 | Heptacosane | 1.474 |
| 43. | 47.107 | Squalene | 0.627 |
| 44. | 48.390 | Nonacosane | 0.945 |
| 45. | 49.021 | Oxirane 2,2-dimethyl-3-(3,7,12,16,20-pentamethyl-3,7,11,15,19 heneicosapentaeny) E | 0.331 |

**Table 5:** List of common chemical constituents (%) of two *jasminum* species.

| **Sr. no.** | **Compound name** | ***J. grandiflorum*** | ***J. officinale*** |
|---|---|---|---|
| 1. | Acetic acid, phenyl methyl ester | 12.200 | 9.004 |
| 2. | 9-tricosene | 3.088 | 0.727 |
| 3. | Tau-muurolol | 3.596 | 0.973 |
| 4. | Palmitic acid | 2.670 | 3.079 |
| 5. | Methyl stearate | 2.012 | 0.701 |
| 6. | Methyl linolenate | 2.684 | 3.131 |
| 7. | Linalool | 11.044 | 7.112 |
| 8. | 1-Heptatriacotanol | 0.841 | 0.274 |
| 9. | Heptacosane | 1.355 | 1.474 |
| 10. | 3-Hexenyl benzoate | 10.501 | 8.848 |
| 11. | Geranyl linalool | 4.232 | 4.217 |
| 12. | Farnesal 1 | 2.574 | 0.345 |
| 13. | Alpha.farnesene | 1.760 | 5.381 |
| 14. | Alpha.cadinol | 6.709 | 1.436 |
| 15. | Benzyl bezoate | 1.286 | 10.103 |
| 16. | Benz aldehyde | 0.883 | 0.284 |