

SUMMARY AND CONCLUSION

SUMMARY:

This work was carried out to study the effect of some factors on the yield of Egyptian distilled oil and extracted vetiver concretes and their absolutes by benzene and n.hexane, and their characters. The obtained results could be summarized in the following:

I) Effect of different factors on oil yield, physicochemical properties and chemical composition of vetiver essential oil:

(1). The yield of oil from roots of 2 and 3 years age showed nearly the same percentage, as it was 0.507 and 0.498% respectively, which is nearly 5 times of that from those of 1 year age which reached only 0.114%.

(2). Oils from roots of 2 and 3 years age were nearly of close physical and chemical characteristics showing higher refractive index, specific gravity, optical rotation, acid value and ester value than those of oil from roots of 1 year age.

(3). The concentrations of two from the identified five major constituents namely, $M^{+}218$ and 220 (Sesquiterpenoids) increased in oils produced from roots of 2 and 3 years age, while these of $M^{+}202$ and 222 are

lower. The sesquiterpene hydrocarbons ($M^{+}204$), on the other hand, are almost of the same concentration in the three oils.

(4). The odour of oils produced from roots of 2 and 3 years age were significantly better than those produced from roots of 1 year age.

(5). The yield of oil increased gradually after roots storage for 1 month before distillation, as it reached 0.145%, multiplied nearly twice after storage for 5 months as it reached 0.273%. The maximum oil yield was obtained after storage for 6 months before distillation, hence it amounted to 0.507%.

(6). The refractive index fluctuated in oils obtained from roots stored for different periods before distillation, while specific gravity, optical rotation, acid value and ester value showed higher values in oil from roots stored for 6 months before distillation.

(7). The contents of the sesquiterpene hydrocarbons ($M^{+}202$ and 204) were significantly changed as a result of roots storage for different periods before distillation. Those of $M^{+}202$ decreased gradually till storage of 4 months, then increased gradually till the last storage period. No significant differences occurred among the compounds of $M^{+}218$ and 220. Generally,

significant differences in the content of the sesquiterpenic alcohol $M^{+}222$ could be detected between the oils obtained from roots stored for different periods before distillation.

(8). The odour quality of oil obtained from roots stored for 6 months was significantly better than those produced from roots stored for other periods.

(9). The oil yield increased gradually corresponding to increase of roots thickness, hence the oil content was; 0.093, 0.129 and 0.529% from roots of thickness less than 0.2, 0.2-1.3 and more than 1.3 mm. respectively.

(10). There is no effect of roots thickness on the refractive index of oils. However, the specific gravity and optical rotation were significantly affected. They were the higher in oils obtained from roots of thickness more than 1.3 mm. Oils from roots of medium thickness (0.2-1.3 mm.) had the higher acid and ester values, but those of thickness more than 1.3 mm. had the lower ones.

(11). The percentages of the sesquiterpenic hydrocarbons ($M^{+}202$ and 204) were affected significantly by the thickness of roots. They were the highest in oil obtained from thick roots (11.79 and 9.35%) and decreased gradually with decrease in roots thickness (7.29 and 3.50%), (5.53 and 2.41%) in oils from medium and thin

roots respectively. The two identified constituents namely those of M⁺218 and 220 had the higher concentrations in the oil obtained from roots of thickness 0.2-1.3 mm., while its content in the oil from roots of thickness less than 0.2mm. represents the lower. The concentrations of the sesquiterpenic alcohol M⁺222 in oils from roots of thickness more than 1.3 mm. and 0.2-1.3 mm. were about the same, being completely absent in oil obtained from roots of thickness less than 0.2 mm.

(12). The odour of oil obtained from thick roots (more than 1.3mm.) was better than that of the other two oils.

(13). The oil content increased proportionally with the increase in distillation pressure as it was 0.118, 0.458, 0.505, 0.510 and 0.684% under 1, 1.5, 2.0, 2.5 and 3.0 atmospheres absolute pressure respectively.

(14). Refractive indices of oils produced under 1.0 and 1.5 atmospheres absolute pressure were nearly the same, as they were 1.5222 and 1.5229 respectively, which represent the lower values than those produced under other pressures. The specific gravities of oils increased gradually with the increase of distillation pressure till 2.0 atmospheres absolute pressure, then decreased to about the same values under 2.5 and 3.0

atmospheres absolute pressure. Oil obtained by distillation under 1.0 atmosphere absolute pressure had the highest value of optical rotation (35°), followed by that obtained under 2.0 which reached ($+25^{\circ}$), while the lowest values were for those obtained by distillation under 1.5, 2.5 and 3.0 atmospheres absolute pressure as they were only ($+20^{\circ}$). The acid and ester values fluctuated in oils obtained under the different pressures, however, the lowest one was obtained by distillation under 3.0 atmospheres absolute pressure.

(15). The content of the unsaturated sesquiterpene hydrocarbons ($M^{+}202$) changed under different pressures of distillation as they reached 10.59, 10.33 and 11.01% under 1.0, 2.0 and 2.5 atmospheres absolute pressure respectively, which were higher than those obtained under 1.5 and 3.0 atmospheres absolute pressure. The content of sesquiterpene hydrocarbons ($M^{+}204$) increased gradually with increase in pressure. Sesquiterpenoids $M^{+}218$ amounted to the highest values under 1.5 and 2.0 atmospheres absolute pressure, as they were 16.15 and 16.29% respectively. The higher contents of sesquiterpenoids $M^{+}220$ were 33.38 and 32.38% under 1.0 and 2.0 atmospheres absolute pressure respectively. Sesquiterpene alcohol $M^{+}222$ revealed the lower content

under 2.0 atmospheres absolute pressure as they reached 1.22%.

(16). The odour quality of the oil obtained by distillation under 2.0 atmospheres absolute pressure was significantly the better than those of the oils obtained by distillation under other pressures. There was no significant difference between the odour quality of oils produced under 1.5 and 1.0 atmospheres absolute pressure. The panelists gave the lower mean score for the oils produced under 3.0 and 2.5 atmospheres absolute pressure.

II) Studies on vetiver oils distilled from the marc after solvent extraction:

(1). Oil distilled from marc extracted with benzene gave higher yield than that extracted with n.hexane, as it reached nearly the double, as their percentages were 0.415 and 0.225% respectively.

(2). Refractive index, specific gravity and ester value showed higher values for oil distilled from marc extracted with hexane as they reached 1.5251, 1.0214 and 109.9 among 1.5225, 1.0180 and 104.9 for that distilled from marc extracted with benzene. Contrary to the above conclusion, the oil from marc of benzene extraction had the higher acid value.

(3). Three of the five major constituents namely: M^{+202} , 204 and 220 increased in the oil obtained from the marc of hexane extraction than that from marc of benzene extraction, as they were 10.63, 10.93, 26.84% and 7.31, 7.42, 19.39% respectively. On the other hand, the sesquiterpenoids M^{+218} and sesquiterpenic alcohol M^{+222} , are nearly of the same concentration, as they were 14.96, 2.80% and 15.36, 2.87% in oils distilled from marc extracted with benzene and hexane respectively.

(4). The odours of the two oils are not significantly different, and represented a less closed odour to the standard sample of Réunion vetiver oil.

III) Analysis of distilled vetiver oils from different origins in comparison with Egyptian oil:

(1). Egyptian vetiver oil revealed the nearest value of refractive index to that of Réunion as it was 1.5270 and 1.5268 respectively. Also the specific gravity was more than 1.0 in both Egyptian and Réunion oils, while it was below 1.0 in the other oils. The acid value was nearly the same in Egyptian and Chinese oils as it was 42.5 and 42.1 respectively, however the lowest value was that of Haiti which reached only 2.8. Java and Réunion vetiver oils had moderate acid values of 20.4 and 16.5

respectively. The Egyptian vetiver essential oil proved the highest value of ester value, (90.1) while the other oils had ester values between 46.7 and 59.5.

(2). Réunion vetiver oil contained the least percentage of sesquiterpenes $M^{+}202$, however that of Java contained the highest value. Egyptian oil showed nearly the same percentage of these compounds as Haitian and Chinese oils. Sesquiterpenes $M^{+}204$ in Egyptian vetiver oil were of the lowest percentage, while those of Java showed the highest one. Sesquiterpenoids $M^{+}218$ were nearly at the same percentage in both Egyptian and Réunion oils, however the rest of oils contained different percentages. Egyptian vetiver oil revealed the highest percentage of $M^{+}220$, however that of Réunion was nearly close to the Egyptian oil as they were 27.93 and 32.38% respectively. $M^{+}222$ compound was of the lowest percentage in Egyptian oil, while that of Réunion was of the highest value.

IV) Effect of different factors on the benzene concrete content, its physicochemical properties and chemical composition:

(1). Vetiver roots extracted with benzene, revealed concrete content of 1.99, 2.50 and 2.52% from root of 1, 2 and 3 years age respectively, however no significant

difference could be observed between those of 2 and 3 years age.

(2). The refractive index of concrete from roots of 1 year age was 1.5066, while those from roots of 2 and 3 years age were nearly the same as they were 1.5137 and 1.5101 respectively. The melting point took the same trend for the concretes of roots of 2 and 3 years age, hence it was 26°C for both. Concrete from roots of 1 year age, was of less value as it reached only 22°C. Acid value was higher while aster value was less in concrete of roots of 1 year age, as it was 43.3, 29.5, 38.2 and 156.1, 159.0, 177.1 in concretes from roots of 1, 2 and 3 years age respectively.

(3). The absolute percentages of concretes extracted from roots of 2 and 3 years age showed higher value and was nearly the same, as it reached 89 and 90% respectively, while that of concrete extracted from roots of 1 year age was the lower.

(4). The refractive index of absolute of concrete extracted from roots of 1 year age obtained less value than those extracted from roots of 2 and 3 years age, as it was 1.5029, 1.5105 and 1.5091 respectively. On the contrary, the specific gravity took the opposite trend. No significant differences could be observed among the

other properties of absolutes.

(5). The four major constituents namely those of M⁺202, 204, 218 and 220 increased in absolutes from roots of 2 and 3 years age than those from roots of 1 year age. On the contrary, the percentage of the sesquiterpenic alcohol M⁺222 in absolute from roots of 1 year age represented the highest value.

(6). The concrete content increased gradually by increasing storage period of roots before extraction till storage periods of 3 and 4 months, then declined. The concrete content reached its maximum in oil from roots stored for 4 months while the lowest value was achieved after storage of roots for 1 month before extraction.

(7). Refractive index was at its lowest value after storing vetiver roots for 1 month before extraction, then increased without any significant change through the rest of storage periods. The melting point showed its lower value after storage of roots before extraction for 1 month as it was 22°C and increased gradually till storage period of 5 months, where it reached 27°C, then became stable. The acid and ester values fluctuated through storage periods of roots before extraction, however the lowest value of acid amounted to 26.4 after roots storage for 5 months, while the highest value of ester reached

199.0 at the last period of storage (7months)

(8). The absolute content of benzene concrete increased gradually till storage period for 4 months before extraction, then decreased gradually till the storage period of 7 months.

(9). The highest value of specific gravity of absolute was that after 1 month of roots storage as it was 1.2360 while the lowest value was achieved after 5 months, of roots storage hence it reached 0.9987. The optical rotation showed its lowest value in absolute after storage of roots for 1 month before extraction as it was $+10^{\circ}$, then amounted to $+20^{\circ}$ and became stable. The acid value showed the lowest value after storage period of 1 month, then fluctuated through till it reached the highest value at the last period of storage. The ester value fluctuated through the different periods of storage, however the lowest value was observed after 2 months of roots storage.

(10). The 3 sesquiterpenes of $M^{+}202$, 204 and 222 had the lower values in the absolutes of benzene extracts from roots stored for different periods before extraction while those of $M^{+}218$ and 220 obtained higher values, however those of $M^{+}220$ were of the highest values.

(11). The concrete centent increased gradually by

increase the thickness of vetiver roots, as it 0.98 1.52 and 3.82 from roots; less than 0.2, 0.2-1.3 and more than 1.3 mm. respectively. Thick roots gave nearly 4 times of concrete than fine roots.

(12). Refractive index, melting point and acid value increased gradually by increase of roots thickness. On the other hand ester values took the opposite trend and decreased gradually as they were; 174.8, 123.8 and 105.9 from roots of; less than 0.2, 0.2-1.3 and more than 1.3 mm. respectively.

(13). The absolute content in benzene concretes, increased gradually by increase of roots thickness as it was 82.0, 83.0 and 84.8% from concretes of roots of thickness less than 0.2, 0.2-1.3 and more than 1.3 mm. respectively.

(14). The refractive index, specific gravity and acid values of absolutes increased gradually by increase of roots thickness. On the other hand optical rotation and ester value were high in absolutes of roots of less than 0.2 mm. thickness.

(15). The contents of sesquiterpene hydrocarbons (M^{+202} and 204) were affected significantly by roots thickness. The absolute from thin roots contained the higher percentages (3.52 and 4.08 respectively) than absolutes

from needium and thick roots which were; 2.01, 1.30 and 2.29, 2.70% respectively. The concentrations of the two identified constituents namely those of M⁺218 and 220 represent the higher values in absolute from thick roots and decreased gradually towards that from the thin roots. The sesquiterpene alcohol (M⁺222) reached the higher concentration in absolute from thick roots than that from thin roots, being completely absent in the absolute from roots of medium thickness.

(16). The concrete content decreased gradually from the first to the fourth soaking, as it was 1.19, 0.57, 0.42 and 0.32% from first, second, third and fourth soaking respectively. The first soaking gave nearly 3 times of concrete than the fourth soaking.

(17). The refractive index of concretes from first, second and third soaking were nearly the same, as they were 1.5127, 1.5128 and 1.5126 respectively, while that of fourth soaking was 1.5090. The melting point showed its highest value for concrete of the first soaking as it was 29°C, then reached to 25°C for the three other soakings. The acid value showed the highest value for the concrete of first soaking, then fluctuated towards the fourth soaking, hence its concrete has the lowest value. The ester value fluctuated through the successive

soakings, however the highest and lowest values were observed in concretes of fourth and first soakings respectively.

(18). The absolute percentage decreased gradually from first soaking to the last, as it was 92.0, 90.7, 88.0 and 87.0% from concretes of first, second, third and fourth soaking respectively.

(19). The refractive index and specific gravity of absolute decreased gradually from the first to the fourth soaking. The optical rotation of the investigated samples was the same ($+25^{\circ}$), except that of absolute resulted from the third soaking, as it was $+20^{\circ}$. On the other hand, the acid value decreased gradually towards the fourth soaking, while the ester value took the opposite trend and increased gradually as they were; 78.31, 86.52, 86.85 and 94.52 for absolutes of first to fourth soaking respectively.

(20). The concentrations of the two identified constituents of $M^{+}202$ and 204 represent the higher values in absolutes from third and fourth soakings (2.92, 3.56 and 1.94, 2.92 respectively) than those absolutes from first and second soakings (1.71, 1.16 and 1.46, 1.60 respectively). On the other hand, the concentrations of the two sesquiterpenoids of $M^{+}218$ and 220 took the

opposite trend, as the absolutes from third and fourth soakings had the lower values than that absolutes from first and second soakings. The sesquiterpene alcohol (M^{+222}) had the lower value in the absolutes under investigation, and its concentration in absolute from the third soaking represent the highest value.

V) Effect of different factors on the hexane concrete, its physicochemical properties and chemical composition:

(1). The hexane concrete contents were 0.96, 1.10 and 1.09 from roots of plant of 1, 2 and 3 years age respectively. There is no significant difference between those of 2 and 3 years age.

(2). The refractive index of concrete from roots of 1 year age was higher than that from roots of 2 and 3 years age (nearly the same), as they were 1.5765, 1.4987 and 1.4995 respectively. The melting point took the opposite trend, hence it was 39°C for both concretes from roots of 2 and 3 years age. Concrete from roots of 1 year age was of less value, as it reached only 35°C . The acid value was higher while ester value was less in concrete from roots of 1 year age, as it was 25.0, 19.5, 20.5 and 144.6, 156.9, 155.0 in concretes from roots of 1, 2 and 3

years age respectively

(3). The percentage of absolute in concrete extracted from roots of 2 and 3 years age showed higher values and were nearly the same, as they amounted to 81.0 and 80.5 respectively, while that in concrete from roots of 1 year age was the lower (65.0%).

(4). The refractive index, optical rotation and acid value were higher while specific gravity and ester value were less in absolute from concrete extracted from roots of 1 year age. Generally, all the determined constants in absolutes from concretes extracted from roots of 2 and 3 years age, had nearly the same values.

(5). Generally, there are no significant differences between the concentrations of the four major constituents namely those of M^+202 , 204, 218 and 220, in absolutes from roots of 2 and 3 years age. The content of constituents of M^+202 and 204 were higher while those of M^+218 and 220 were less in absolute from roots of 1 year age. The sesquiterpene alcohol (M^+222) reached nearly the same concentration in absolutes from roots of 1 and 3 years age, being completely absent in the absolute from roots of 2 years age.

(6). The concrete content increased gradually by increasing storage period of roots before extraction till

4 months, then declined. The concrete contents of roots stored for 4 and 1 month before extraction represent the maximum and minimum values respectively.

(7). There are no significant differences between the values of refractive index of concretes from roots stored for different periods before extraction. The melting point increased gradually by increasing the storage period before extraction till 4 and 5 months, then decreased gradually and reached the lowest value after storage period of 7 months. The acid and ester values fluctuated through storage periods of roots before extraction, however the lowest values of acid and ester amounted to 17.5 and 53.1 after storage periods; 2 and 1 months respectively, while the highest values reached 45.5 and 156.9 after storage periods of 3 and 4 months respectively.

(8). The absolute content of hexane concrete showed increase by increase of storage period of roots before extraction till the highest after 4 months, then decreased to reach the lowest value after storage period of 7 months.

(9). The refractive index and specific gravity of studied absolutes showed fluctuation after different storage periods of roots however the highest values were achieved

after storage period of 1 and 2 months, as they were 1.517 and 1.1043 respectively, while the lowest values were observed after storage period of 4 months for both, as they were 1.5038 and 1.0263 respectively. The most of studied absolutes have the same value of optical rotation, as they were $+20^{\circ}$, while the highest and lowest values were achieved after storage period of 1 and 2 months, as they were $+40^{\circ}$ and $+15^{\circ}$ respectively. The acid and ester values fluctuated through storage periods of roots before extraction, however the highest value of acid and ester reached 55.50 and 134.11 after storage periods of 7 and 4 months respectively.

(10). The two identified hydrocarbons of $M^{+}202$ and 204 had the lower concentrations in all samples of absolutes under study, while those of $M^{+}218$ and 220 had the higher values. However, the sesquiterpene alcohol of $M^{+}222$ obtained the lowest value, being completely absent in the absolutes from roots stored for 2,4,6 and 7 months before extraction.

(11). The hexane concrete content increased gradually in vetiver roots by increase of their thickness, as it was 0.26, 0.58 and 1.19% from roots of thickness less than 0.2, 0.2-1.3 and more than 1.3 mm. respectively. Thick roots gave about 4.5 times of concrete than fine roots.

(12). The refractive index reached the highest value for the concrete from thick roots, as it was 1.5105. The melting point and acid value increased gradually by increase of roots thickness. On the other hand ester values took the opposite trend and decreased gradually, as they were 94.9, 90.7 and 80.9 for concretes from roots of thickness less than 0.2, 0.2 -1.3 and more than 1.3 mm. respectively.

(13). The absolute content increased gradually by increase of roots thickness as it was 76.0, 77.9 and 89.0% in concretes from roots of thickness less than 0.2, 0.2-1.3 and more than 1.3 mm. respectively.

(14). The absolute from thick roots had the highest refractive index and optical rotation (1.5182 and $+25^{\circ}$ respectively), while the fine roots possess the highest specific gravity (1.0890). The acid value increased gradually by increase of roots thickness, while the ester value took the opposite trend and decreased gradually, as they were 41.42, 44.45, 58.02 and 78.24, 75.09, 50.62 for absolutes from roots of thickness less than 0.2, 0.2-1.3 and more than 1.3 mm. respectively.

(15). The absolute from fine roots characterized by the highest concentrations of the two identified components of M^{+} 202 and 220, as they were 11.76 and 35.59%

respectively. On the other hand, the absolute from thick roots had the highest content of constituents of M⁺204 and 218, as they were 7.88 and 29.46% respectively. Generally, the content of sesquiterpene alcohol (M⁺222) represents the lowest concentration in the studied samples, and completely absent in absolute from thick roots.

(16). The hexane concrete content decreased gradually from the first to the fourth soaking, as it was 0.48, 0.29, 0.19 and 0.14% from first, second, third and fourth soaking respectively. The content of concrete from the first soaking was about 3.5 times of that from fourth soaking.

(17). The refractive index of concretes from the last three soakings were nearly the same, as they ranged from 1.4995 to 1.5002, while that of the first soaking was 1.5097. The melting point showed its highest value for concrete of the first soaking as it was 39°C, then reached 32°C for the other three soakings. The acid and ester values fluctuated through the four successive soakings, however the highest values were observed in concretes of first and fourth soakings respectively.

(18). The absolute percentage in concrete obtained from the first soaking showed the highest value, as it was

37.1, then decreased gradually till the fourth soaking, as they were 21.1, 13.4 and 9.4 respectively.

(19). The refractive index and specific gravity fluctuated among the different investigated absolutes. The optical rotation of the resulted absolutes was the same ($+20^{\circ}$), except that of the fourth soaking, as it was $+25^{\circ}$. The highest acid and ester values were observed in absolutes of concretes from first and fourth soakings respectively.

(20). There are no significant differences in the concentrations of the two sesquiterpene hydrocarbons of $M^{+}202$ and 204 , between the four studied absolutes. The concentrations of the two identified constituents namely those of $M^{+}218$ and 220 represent the higher value in absolute from the first soaking and the lower value in absolute from the last soaking. However, the sesquiterpene alcohol of $M^{+}222$ obtained the lowest value, being completely absent in the absolutes from the first and second soakings.

CONCLUSION:

Vetiver roots of Egyptian vetiver plants were investigated for the production of two aromatic products; distilled oil and concretes extracted by benzene and n. hexane and consequently their absolutes.

From data obtained, it could be mentioned that roots of 2 years age, stored for six months before distillation process under 2 atmospheres absolute pressure and distillation for 25 hours gave the best yield and quality of essential oil.

However, concretes obtained by benzene and n.hexane revealed the best yield and quality when roots of 2 years age were stored for 4 months before extraction.

It is well known that marcs of extraction are usually considered as waste, however distillation of marcs of benzene and n. hexane extracts of vetiver roots gave a significant percentage of oils of different organoleptic properties which are different from the normal vetiver essential oil.