Egyptian Jasmin

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The jasmin flower yields one of the most important and exquisite natural oils employed since time immemorial. The warm and highly diffusive odor of the oil is an integral and indispensable component in high quality perfumery. The old saying "no perfume without jasmin" is not too far from the truth today.[1] Jasmin absolute essence is also used in the formulation of compounded flavors, especially in liqueurs, pastry, condiments and baked goods.[2]

Jasminum grandiflorum L. was first produced commercially in Egypt for the purpose of perfumery at the turn of this century.[3] The name jasmin is derived from the Arabic "Yasmeen." The flower was apparently native to India and was brought to North Africa and Spain by the conquering Moors, where it spread to the Mediterranean basin during the 16th century after being introduced to southern France.[4] During that time, the common white flower *J. officinale* was also introduced to France from Persia.

It was not until the 1860s that large plantations of jasmin for the perfume industry were cultivated. To make the flower more frost resistant and better able to survive conditions other than ideal, *J. grandiflorum* was grafted on *J. officinale*.[5] It was later during the early twentieth century that industrial jasmin production moved to Egypt, where the climate was warmer and very suitable for cultivation. Other production sites were Italy, Spain, Morocco, Turkey, and Lebanon. For many years southern France owed much of its growth and establishment as a source for natural fragrance materials to the production and extraction of jasmin flowers.

The geographical sites of jasmin cultivation have dramatically changed from what it was in the late 19th and early 20th centuries. In the past, cultivation had centered around Grasse (France), Calabria (Italy), and Mitidja (Algeria). Today, Egypt is the primary producer of jasmin flower with more than 60% of the total world production of jasmin concrete. India, at 22%, is the secondary source of jasmin. India's production started in the early 1970s. World production (in 1990) of jasmin concrete was estimated at 10.0 to 10.5 metric tons, having the following ratios:[6]

	Kilos
Egypt	6,000
India	2,200-2,400
China	1,000
Morocco	600-700
Guinea	200
France	45-50
Algeria	15

Today, in Italy and Spain, production is practically nonexistent.

The jasmin flower is delicate and fragile, as is the extraction of its essence. It is a perennial plant with attractive snow white flowers. The time of day when the flowers are picked is crucial to the odorous quality of the extracted essence. In addition, the length of time that the picked petals are left to breathe before extraction is also critical.

The beauty of jasmin is that even after the flowers have been picked from the plant, they continue to develop and diffuse their odor until the flowers fade and dry. This is exhibited in the range of percentages for indole (a natural chemical ingredient of jasmin). This variation is directly related to the time of day the flowers were picked and how long after picking they were extracted. The finest quality jasmin is that which had been picked early in the morning. This is when the flower has optimum yield and fragrance.

Methods of Extraction

There are several extraction procedures which remove the essence or odorous chemicals from fragrant plant materials such as jasmin. These processes may include the use of volatile organic solvents, or may utilize the absorptive prop-

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erties of fats as in the enfleurage process.

Steam distillation and maceration are two other extraction procedures. In steam distillation, most of the volatiles evaporate under the high temperatures involved so this method is never used on the delicate jasmin petal.

Enfleurage will be described briefly not for its importantce at present but for its historical importance, that which gave floral extraction and perfumery their artistic beauty.

Enfleurage is the process whereby the freshly picked flower petals are placed on layers of fat made up predominantly of purified and odorless lard. A number of these fat filled trays, or chassis, are stacked one on top of the other. The flowers are still "alive" after harvesting and continue to produce and release their chemicals which are absorbed by the fats. This is another reason why the flowers must be gently handled after having been picked so as not to disrupt the life process. A new group of fresh petals replace the old after 24 h and the same procedure continues.

This cycle continues for several weeks until the fat layers are saturated with the perfume oils. The fatty layers are then melted and resolidified to create a uniform mass that is then extracted with alcohol. The alcohol is removed by vacuum distillation at low temperatures yielding the flower absolute.

Enfleurage is used primarily for delicate florals such as jasmin and tuberose. It gives relatively high yields of flower oil. However, the process requires a great deal of costly hand labor, time and effort. Today, enfleurage is almost totally replaced by volatile solvent extraction of the flowers.[7]

In volatile solvent extraction, the flowers are placed in large containers known as extractors together with the solvent of choice, usually hexane. Either the extractor rotates or the solvent circulates as in a coffee percolator. The solvent continuously extracts the organics from the flower parts taking advantage of the preferred solubility factors. The oil enriched solvent is then led to a vacuum still where the solvent is distilled off and returned for reuse.

What gets extracted out in this procedure are all the hexane soluble materials such as the desired jasmin fragrant organics as well as undesired plant waxes. The waxes present in this concrete (over 50%) make this product soluble in limited products and hence undesirable in this form. Therefore, the concrete is extracted with several times its own weight with pure alcohol at 54°C, dissolving the perfume and some of the waxes. The precipitated waxes are filtered off. The alcoholic solution is cooled to –25°C to –30°C to precipitate the soluble waxes, and filtered again to remove the remaining precipitated waxes from the solution. The wax-free solution is then concentrated by distilling out the alcohol at 35°C and 20mm/Hg pressure. Traditionally, the absolute is the more preferred of the two and when fresh, is very reminiscent of fresh flowers in odor.[8]

Typical production yields in the extraction of jasmin range from 0.25% for the concrete to 0.1% for the absolute. It is estimated that 7.5-8.5 million jasmin blossoms weigh about 1,000 kilograms. Thus one ton of fresh flowers yield approximately 2.5 kilos of jasmin concrete. One kilo of jasmin concrete yields 530-600 grams of jasmin absolute.[9]

Comparing the process of enfleurage with solvent extraction in terms of yield, calculations are based on percent of volatile oils. As a result of many studies made earlier this century, it was shown that the process of enfleurage gives 2-2.5 times the yield achieved by solvent extraction. It was also shown years later by Naves that the chemical compositions of the two products are relatively the same.[10]

Chemical Analysis

Undoubtedly, the method of extraction of the volatile constituents of the jasmin flower will have a major effect on the results of the analysis. It is well known that water and steam distillation extractive techniques produce inferior oils.[11] The methods of choice today require solvent extraction procedures or the more labor intensive enfleurage processes. Minor differences in the quality of oil exist between those two methods of extraction.[12] The indole content is generally higher in enfluerage processes when compared to solvent extraction methods. Interestingly enough, Mookherjee et al.[13] in their elegant comparative study done on *Jasminum grandiflorum* via head space analysis of picked vs. living flowers revealed similar differences in the indole content. They reported a level of 11% in the living flower vs. 2% in the picked flowers. A value of 11% is an unexpectedly high level of indole in "living" jasmin flower. That is more than we ever detected in "picked" flowers. However, levels as high as 8% in "night" picking jasmins were observed.

The factors affecting the fluctuation of the indole content in jasmin are many and include:

- Indole being present in the buds not as the free form but as a complexed compound freed only when the buds open in the early morning hours.[14]
- It is estimated that the indole is freed and evaporates into the atmosphere upon exposure of the flower to the sun even before it is harvested off the shrub, at the rate of 0.6 to 0.8 mg/100 g of flowers/h. Thus it is clear that the length of time the flowers are exposed to the sun will have a dramatic effect on the percentage of indole present.
- The length of time picked flowers are left to "breathe" prior to the extraction is also an important variable. When the flowers are exposed freely to the atmosphere, the indole evaporates steadily. This explains the relatively high indole content of the enfleurage products.
- The concrete of jasmin will generally have a higher indole content than the absolute which is obtained from the alcohol washings and chilling off the waxes of the concretes. This is due to the relative solubility of the indole in the alcohol.[15]

Jasmin, being a natural plant is also susceptible to geographical and climatic factors which affect the quality of the extracted oils. Such variables include:

- The geographic location of the growing areas.
- The temperature and the amount of sun to which the flowers are exposed. It is well known that during warm and sunny weather the flowers give a better yield and a superior quality oil than during cloudy and especially rainy weather.
- The yield and the quality of the flower oil is higher and superior during the height of the season than at the beginning or at the end of the season.
- The soil and the availability (or lack) of nutrients and fertilizers may influence the quality and quantity of oil produced.[16-19]

With nearly 100 varieties of jasmin flowers in India, Arabia, Egypt and China, and with all of the above considerations relating to natural and/or man-made variations during harvesting, collection, extraction and concentration, it is no wonder that data on the chemical constituents will vary from one source to the other.

Many publications have dealt extensively with the analysis of jasmin,[20-24] yet our data presented in this paper will be the most relevant from a commercial point of view, since our company is a major grower and produces over half of the world's consumption of jasmin. We are thus assured of the authenticity of the samples from different bulkings and picking seasons. Data included reveals the natural variation of Egyptian jasmin absolute during the five months of the 1990 season (June, July, August, September and October) and for the six crop years (1986 through 1990). Finally, the average percentages of the major constituents for the 1991 crop from the five major producing areas in Egypt are also presented for the months of July, August, and September.

It is worthwhile to note here that a few publications on carbon dioxide extraction of jasmin concrete, not the fresh flower, have appeared in the literature.[25-26] The data presented is interesting but would have been more relevant had it been done directly on the flowers rather than on the concrete which has already been solvent extracted. The product obtained from CO_2 extraction is solvent free, however, the process is expensive. When the absolute derived from the alcohol extraction is compared to the absolute from a CO_2 extraction, odor and composition differences are seen. Higher proportions of the more volatile components and lower proportions of the less volatile components have been reported in the CO_2 extraction.[27]

Results

Jasmin absolute samples from more than a dozen different lots from various picking months and years were analyzed. The results from our study are given below for the components volatile enough to elute off the GC. The GC and GC/MS conditions used in our analysis are given in Table I.

Figures 1 and 2 represent GC of Egyptian jasmin absolute run on an Ultra 1 column (Figure 1) and on a cross-linked carbowax column(Figure 2) at 4°C/min. Twenty-five of the more prominent peaks are numbered and identified with their respective levels in Table II. The average percentages listed were quantitated from the non-polar, Ultra 1 column. Other components identified at lower than 0.10% were: benzaldehyde, cis-3-hexenyl acetate, phenyl ethyl alcohol, benzoic acid, cis-3-hexenyl butyrate, iso-eugenol, p-cresyl acetate, phenyl ethyl acetate, methyl anthranilate, trans-nerolidol, hexyl benzoate, ethyl palmitate and methyl linoleate.

The average percentages of the main components of the absolute for the past five production years are shown in Table III.

Major producing areas in Egypt in the Nile Delta are Qutour, Haraneya, Bassyun, Kom El-Nagar, and Gharbiya. We studied samples of jasmin absolute from these five production sites during the optimum seasonal months of July, August, and September of 1991. Table IV contains the average percentages of the five sites by month. Table V contains the average percentages of all three months by production location.

It is a well known fact that the flower is richer and more odoriferous in the early hours of the day. When jasmin is exposed to sunlight, the flower is not as rich in perfume and the oil content is less. Indole gets released from the cells and evaporates or breaks down. Many phenomena happen to the jasmin flower during the daytime and it has been established that the

Table I. GC and GC/MS conditions

	GC	GC/MS
Sample size	0.1 µl	0.05 µl
Oven temp	65°C to 250°C	65°C to 230°C
Program rate	4°C/min	2°C/min
Injection port temp	210°C	210°C
Detector	FID*	TIC*
Carrier flow	1ml/min	1ml/min
Split ratio	150:1	100:1
MS ionization voltage		70 eV
Start, stop masses		35-400
Electron multiplier voltage		2000 eV

* FID= Flame ionization detector
TIC= Total ion chromatogram

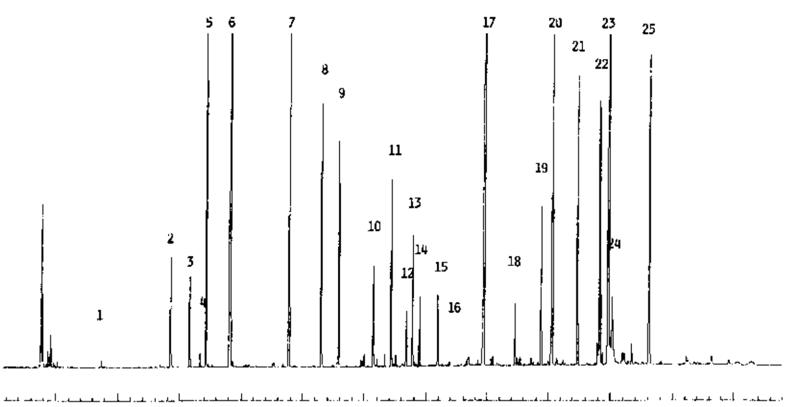

Figure 1. Gas chromatogram of Egyptian jasmin absolute on non-polar column

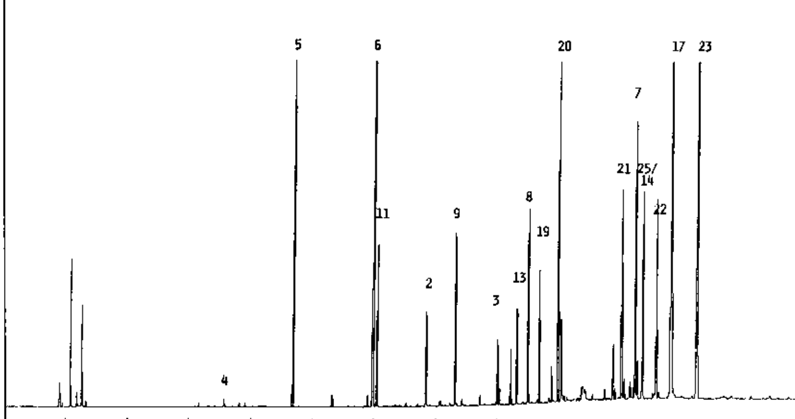

Figure 2. Gas chromatogram of Egyptian jasmin absolute on polar column

Table II. The chemical constituents of Egyptian jasmin absolute

Peak No.	Constituent	Average %
1	cis-3-hexenol	0.07
2	benzyl alcohol	0.80
3	p-cresol	0.95
4	methyl benzoate	0.15
5	linalool	4.58
6	benzyl acetate	25.80
7	indole	3.73
8	eugenol	2.60
9	cis-jasmone	2.36
10	(-)-(R)-(Z)-dec-7-en-5-olide	1.31
11	farnesene	1.96
12	dodec-9-en-5-olide	0.32
13	cis-3-hexenyl benzoate	1.28
14	acetyl methyl anthranilate	0.69
15	cis-methyl jasmonate	0.60
16	trans-methyl jasmonate	0.16
17	benzyl benzoate	11.48
18	phytone	0.63
19	methyl palmitate	1.55
20	iso-phytol	8.15
21/25	phytyl acetate	7.33
22	methyl linoleate + methyl oleate	4.46
23	phytol	12.52
24	methyl stearate	1.64

Table III. Average percentages of the major components of jasmin absolute for crop years 1986-1990

Constituent	1986	1987	1988	1989	1990
benzyl alcohol	0.8	0.9	0.9	0.8	0.6
linalool	4.9	4.5	6.0	5.8	4.5
benzyl acetate	21.1	26.2	26.7	26.3	24.0
indole	3.4	3.3	4.2	4.5	4.4
eugenol	2.9	2.5	3.4	2.9	3.4
cis-jasmone	2.4	2.5	2.7	2.5	3.3
farnesene	3.0	2.2	2.5	2.1	3.5
cis-3-hexenyl benzoate	2.0	1.4	1.3	1.5	1.3
benzyl benzoate	12.0	12.6	14.4	13.8	11.2
iso-phytol	8.6	7.0	7.9	8.5	7.6
phytyl acetate	10.1	7.9	7.8	8.1	7.9
methyl linoleate	4.2	3.6	3.3	3.3	3.4
phytol	12.3	14.8	9.4	14.5	11.3

Table IV. Average percentage of the major components of jasmin absolute for the 1991 season by months

	July	August	Sept.	Average
benzyl acetate	25.08	26.24	25.18	25.50
benzyl alcohol	0.35	0.38	0.41	0.38
benzyl benzoate + phytol	24.50	23.23	25.03	24.25
cis-3-hexenyl benzoate	1.20	1.19	1.16	1.18
cis-jasmone	3.05	3.20	3.00	3.08
eugenol	3.26	3.05	2.77	3.03
farnesene	3.37	3.53	4.41	3.77
indole	5.49	5.40	5.52	5.47
isophytol	8.10	8.07	7.93	8.03
linalool	4.77	5.22	3.78	4.59
methyl linoleate	3.81	3.74	3.80	3.78
phytyl acetate	7.87	7.43	7.04	7.45
all others	9.15	9.32	9.97	9.49

Table V. Average percentage of the major components of jasmin absolute for the 1991 season by geographic location in Egypt

	Qutour	Haraneya	Kom El Nagar	Bassyun	Gharbia	Average
benzyl acetate	24.42	25.07	26.04	25.62	26.38	25.50
benzyl alcohol	0.43	0.49	0.40	0.21	0.37	0.38
benzyl benzoate + phytol	24.80	23.59	25.25	23.39	24.23	24.25
cis-3-hexenyl benzoate	1.19	1.31	1.22	1.14	1.08	1.18
cis-jasmone	2.79	3.51	2.72	3.67	2.72	3.08
eugenol	3.10	3.05	3.11	2.84	3.03	3.03
farnesene	3.95	3.48	3.68	3.94	3.80	3.77
indole	5.30	5.32	4.62	6.49	5.62	5.47
isophytol	8.35	7.86	8.11	7.81	8.04	8.03
linalool	4.45	4.73	4.71	4.40	4.62	4.59
methyl linoleate	3.88	3.67	3.85	3.70	3.84	3.78
phytyl acetate	7.61	7.97	7.32	7.18	7.15	7.45
all others	9.73	9.95	8.97	9.61	9.12	9.49

best jasmin product is that which was picked in the dawn hours of the day or "night-picking."

The massive data presented in this paper which represents only the average of many analyses per data point serves to reveal part of the complexity in dealing with the many natural variations of an agricultural product such as jasmin. Nevertheless, due to the tremendous production capacity available, most of the variations in individual components are generally averaged out. For example, we have analyzed many lots that had as high as 7% and as low as 1.2% indole. However, when such small lots are bulked expertly, a rather consistent product is obtained.

With few exceptions, individual ingredients in the complex jasmin oil vary by as much as 10% (as seen in Tables IV and V) due to the geographic location, the time of day the flowers are picked, or the month of picking in a given season. Generally, the oil content is highest in mid-season and when picking is done prior to 10 AM certain other trends do emerge, such as the content of eugenol and phytol acetate decreasing steadily throughout the season, with July having the most amount and September the least. Farnesene tends to increase steadily throughout the season, with September revealing the highest content. The linalool and benzyl acetate content tends to be highest at mid-season and the benzyl benzoate and phytol the lowest at mid-season (see Table IV).

Geographical location of the flowers is another variable (see Table V), however, the experienced analysts and "noses" at those factories tend to blend the appropriate amounts of the variants in very much the same way as wine or aged whiskey is blended. The combination of experience and instrumental sophistication is necessary to achieve a consistent superior jasmin product for the perfumers and flavorists of the world.

Conclusion

Jasmin is an extremely delicate plant that not only requires the utmost of care in growing and cultivating but is affected by many variables including the time of day the flowers are picked, the precise period in the season, the degree of exposure to sunlight, the method of picking, the length of time the picked flowers are stored prior to extraction, the type of extraction, the duration of the extraction process, the concentration temperatures, the conversion of the "concrete" to the "absolute" and finally the storage period and method of storage prior to the use by the perfumer and flavorist.

The data presented in this paper may shed some light on the complexity of harvesting the jasmin flower and its conversion into a suitable form for the ultimate consumer. The jasmin flower can be likened to a child that needs gentle nurturing and constant attention to thrive and blossom. Interestingly enough, the flowers are picked only by young girls during their summer vacations. It is a joy to witness this picturesque scene where dozens of young girls all dressed in brightly colored clothes and singing while they work. They are required to hand pick the flowers one by one, very gently, without bruising the flower. Crushed or bruised blossoms tend to rapidly oxidize and deteriorate. The bushes are trimmed each year to facilitate their harvesting by children. All attempts to automate or mechanize the process of collecting the jasmin blossoms have failed. Thus regions with expensive labor cost have virtually abandoned the commercial harvesting of the jasmin flowers.

Jasmin is, undoubtedly, one of the most important floral extracts used in perfumery and has, consequently, been subjected to a great number of studies. The absolute represents one of the most striking examples of nature's ability to round off and conceal the odor effect of very simple constituents such as benzyl acetate and linalool. With this precious drop of absolute, the perfumer possesses a gem, an olfactory giant, an essential ingredient in all major worldwide fragrance creations which are recognized successes and would have been impossible without nature's masterful composition of jasmin.

Acknowledgment: *We would like to acknowledge the support of Mr. Ben Benveniste and Eng. Mohamed Osman for their help in providing the necessary samples, data and support to conclude this study. Others who have assisted include Eng. Mohamed Ali from Kato Aromatic in Egypt and Frederick Flores from the Instrumentation Laboratory at Kato Worldwide.*

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