Research Article

Flavour and Fragrance Journal

Received: 2 June 2009; Revised: 25 June 2009; Accepted: 25 June 2009; Published online in Wiley InterScience: 26 July 2009

(www.interscience.wiley.com) DOI 10.1002/ffj.1944

Perfumes in Mediterranean antiquity

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ABSTRACT: Perfumes in antiquity were products of great importance. They played an important role in seduction, feasts and ceremonies, religious rites, funerals and medicine. Antic perfumes were produced by extraction of fragrant raw materials (plants, gum resins, spices, etc.) using vegetable oil (ben, sesame, horseradish, almond, olive oil) as the main excipient. In the frame of new archaeological researches over the antic sites of Delos in Greece and Capua, Herculanum and Pompei in Italy, we sought to understand the composition of perfume in antiquity. How were antic perfumes made? Which fragrances characterized them? The Seplasia[†] project, led by chemists and archaeologists, has the main intention of answering these questions. Copyright © 2009 John Wiley & Sons, Ltd.

Keywords: antic perfumes; raw materials; formulation; antic perfumes analysis

Introduction

Based upon new archaeological findings, our knowledge on perfume during antiquity is currently being reviewed. A wealth of clues spread over the antic sites of Delos (Greece), Capua, Herculanum and Pompei (Italy) has allowed a new interpretation of where and how perfumes were produced and sold during antiquity. Antic perfumes were composed of several ingredients, often including expensive spices and resins. In that way, and more so than today, perfume was a product of social importance, since only affluent people could afford the money to pay for it. In spite of such an importance, we are still missing reliable data about both the composition and the scent of antic perfumes. Unlike other archaeological substances, untouched samples of antic perfumes have never been found. The aim of our project was thus to recreate antic perfumes by means of recipes described with sufficient accuracy in several ancient texts. Our work comprises two main objectives, rediscovering the ancient scents and also providing an aid to interpret and understand antic technologies.

Archaeological State of the Art

The role of perfume in antiquity was much greater than nowadays. Of course, the main use was for personal adornment, as a tool of seduction. Both women and men used perfumes and perfume makers were able to produce unguents particularly suited to one sex or the other. We must remember that Aphrodite wore perfume when she went to the judgement of Paris and ancient literature contains frequent allusions to this use, the only one remaining today. But there were other specific uses in antiquity. A common practice involved being anointed with perfumes after bathing, private or public, and after exercises at the gymnasium. During the Hellenistic and Roman periods, one of the main functions of patron in charge of gymnasia or palestra was precisely to provide oils and perfumes for use by athletes.

Perfumes were also used during feasts: they were worn by individuals and also sprayed around during symposia or processions. Perfumes were also used on bedding and clothing after cleaning.

The Gods wished also to be perfumed. Altars, statues and other cult objects were regularly anointed, for instance the famous omphalos of Delphi was daily perfumed in this way. In his famous book named *Characters*, Theophrastus, characterizing a devoted person, depicts him anointing sacred stones placed at the crossroads.

Funerals were not conceivable without burning perfumes such as incense. They were used also for anointing the corpse, for embalming it, for pouring on the ashes after cremation and for depositing in the grave, contained in small flagons of precious metal, glass or ceramics.

A very important role was played by perfumes in medicine. In truth, there was no difference between perfumes and medicines. Perfume makers were the equivalent of our pharmacists. They made special unguents under the orders of physicians to cure all manners of illnesses and physicians tried to adapt existing types of perfume and perfumed wine to different ailments. The most complete list of perfume recipes extant from antiquity, the *De Materia Medica* written by Dioscorides during the first century A.D., gives all their medical uses. Other specifications can be found in the treatises of the famous physician Galen, who lived during the second century A.D., and in the Graeco-Roman papyri from Egypt.

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[†] Seplasia is the name of a famous place in Roman Capua where perfumers used to make and sell perfumes.

Just as today, perfumes in antiquity were subjected to the vacillations of fashion. Athenaios recorded that fashions altered over the centuries mainly because the origins of aromatics changed and because the favour of the kings and aristocrats varied. According to Pliny, in the past people preferred irinon, a perfume based on the iris from Corinth. Rhodinon, a rose oil from Phaselis in Lykia, was once favoured but was supplanted by those made at Neapolis, Capua and Praeneste. How were ancient perfumes made? According to Pliny,[1] the recipe for making unguents contains two ingredients, the juice and the solid part: the former usually consists of various sort of oil and the latter of scented substances, the oils being called 'astringents' and the scents 'sweetenings'. As early as the end of the fourth century B.C., Theophrastus listed the different types of oils used in perfumery.[2] The most commonly used oils were those obtained from ben, sesame, horseradish, almond and olive, with the latter being the most prolific, since it was easier to produce in large quantities. This oil, made from green olives, was in Latin called *oleum omphacium*. Extracted from unripe berries, the best oil was white, with the inferior one being green; Dioscorides and Pliny mentioned that it was the best to use for perfume and health, probably because of its high resistance to oxidation compared to classical olive oil.[3,4] Theophrastus also notes that it was best to use freshly extracted oil and not the previous year's.[5] Fats were made astringent by being first heated with plants such as sedge, then mixed with aromatic substances, mainly flowers (rose, lily), rhizomes (iris, calamus) but also fragrant woods (cinnamon), spices (cardamom, fenugreek), gum resins (myrrh, Judea balsam, labdanum), etc. Depending on the type of product, the action of imparting the scents onto the fats either required heating or did not. Perfumes were then made less volatile by adding fixing agents, such as resins, and colouring, e.g. red with madder.

If ancient authors give precise details about how to make the oil, the actual perfume recipes remain rare and are often vague concerning the processes used and the quantities of ingredients. The most precise treaty is the *De materia medica* of Dioscorides, cited above, which is the only one that gives exact quantities of aromatics and instructions for making many scented oils employed for the treatment of various ailments. This book is fundamental to any attempts to recreate perfumes following ancient recipes. As a way to get an overview, the most used substances cited in Dioscorides' treaty are listed in Table 1.[6]

Research on ancient perfume has recently been invigorated by new archaeological, chemical and experimental approaches. As perfume is often mentioned in the near-eastern clay tablets, it was tempting to search for perfume-making installations there, especially in the palaces of the Bronze Age. But for these periods, archaeology has so far had little to offer. Perfume making did not require heavy equipment and it is very difficult to detect installations that needed little more equipment than hearths, caldrons and cloths used to squeeze berries or flowers. The same approach has been followed in Crete and Greece, where the palatial archives written in so-called Linear B on clay tablets found at Pylos, Mycenae and Knossos, frequently allude to scented oils. At Pylos, destroyed towards the end of the 13th century, the perfumers' workshops have been tentatively located in front of the building's eastern entrance on account of a discrete quantity of cauldrons, basins and vases found there. But this interpretation is controversial, as was the identification of the workshops recently discovered in the Bronze Age settlement of Pyrgos-Mavroraki, abandoned towards 1850 B.C. The excavators interpret these evidences as oil production plants suited to perfume manufacture; it would have been fitted with hand-made pottery used for primitive distillation.[7–9]‡ No traces of archaic and classical perfume workshops have yet been discovered in Greece, even though they are cited in several ancient texts, such as the well-known speeches of Demosthenes, Hyperides and Lysias.[10]§ In 4th century Athens, perfume shops were located on or near the agora and some perfumers ran prosperous businesses, which the Agora excavations did manage to identify. Archaeological remains of perfume activity can only be found at the end of the Hellenistic period, on the Island of Delos. This island hosted a very famous panhellenic sanctuary of Apollo and reached a considerable development after the year 166 B.C. following a decision by the Roman Senate to grant free port privileges. Delos developed quickly and attracted a large cosmopolitan community of merchants and artisans coming from Greece, Italy and the Orient. This development was interrupted in 69 B.C. by the sack of the city by pirates. During this one century of prosperity, Delos was the crossroads of all kinds of trades between the Orient and the West – mainly slaves but also perfumes which, according to Pliny,[11] the island was renowned for. Several installations have been identified as perfumery on the basis of the discovery of carefully carved marble press beds. They were used to make oil and, in house II B of the Stadium district, they are linked to four furnaces that would have been employed to warm the oil used during the perfume-making process. During this period, the presses employed were fitted with wedges: wooden wedges were inserted between planks maintained by a frame held in place by a frame for pressing the pulp against the stone to extract oils or other juices.[12]

During the Roman period, these techniques spread all over the Mediterranean, not only in the Oriental basin, but also in the western part, especially in Italy, where Campania, during the second half of the first century B.C. and throughout the time of the Empire, became the very centre of perfume production. The phenomenon is well known through literary sources that mention the perfume makers of Capua, and is further confirmed by numerous inscriptions of perfume makers found there and also at Pompei. Indeed, several perfume shops have been identified at Pompei, Herculanum and Paestum; they are dated from the second half of the first century A.D. They are all equipped with central screw presses with a base of stone or wood. This type of press, invented, according to Pliny, during the second quarter of the first century, rapidly replaced the old wedge-type press that was nevertheless still depicted on the Pompeian paintings representing perfume processing. Both of these paintings and the archaeological remains help us to reconstruct the entire process, from the preparation of the different types of oils and fats to the mixing of the ingredients, the heating of the mixture of oil, aromatics and gums, and ending with the filling of the flagons.[12]

During excavation, of course, not all of these stages are detectable: for example, presses are relatively easy to identify but caldrons are always missing (the metal being re-used), mortars have

‡The analyses done by G. Donato *et al.* were not sufficiently sophisticated to give a scientific basis to the authors' conclusions, as N. Garnier *et al.* demonstrated.

§Lysias writes that in Athens, everybody 'is in the habit of paying a call at either a perfumer's or a barber's or a shoemaker's shop, or wherever he may chance to go, in most cases, it is to the tradesmen who have set up nearest the marketplace and in fewest, to those who are farthest from it.' Around 330 B.C., a speech of Hyperides, *Against Athenogenes,* mentions the perfume maker Athenogenes, who owned shops on the Athenian agora. See other examples in Demosthenes (*Against Phormion* 13) and Aristophanes (*Equites* 1375).

Table 1. Raw materials used in antique perfumes

Raw materials	Botanical origin	Function
Vegetable oils		
Olive oil	*Olea europaea*	Excipient
Sesame oil	*Sesamum indicum*	Excipient
Ben oil	*Moringa oleifera*	Excipient
Almond oil	*Prunus amygdalus*	Excipient
Plant materials		
Jasmine (flower)	*Jasminum grandiflorum/Jasminum sambac*	Fragrance
Rose (flower)	*Rosa centifolia/Rosa damascena*	Fragrance
Narcissus (flower)	*Narcissus poeticus*	Fragrance
White lily (flower)	*Lilium candidum*	Fragrance
Henna (flower)	*Lawsonia inermis*	Fragrance
Iris (rhizome)	*Iris pallida*	Fragrance
Calamus (rhizome)	*Acorus calamus*	Thickener, fixative
Nardus (rhizome)	*Nardostachys jatamansi*	Fragrance
Gum resins		
Myrrh	*Commiphora myrrha*	Fragrance, fixative
Judea balsam	*Commiphora opobalsamum*	Fragrance, fixative
Frankincense	*Boswellia carterii*	Fragrance, fixative
Ladanum	*Cistus ladaniferus*	Fragrance, fixative
Galbanum	*Ferula galbaniflua*	Fragrance, fixative
Spices		
Cinnamon	*Cinnamomum verum*	Fragrance
Cardamom	*Elettaria cardamomum*	Fragrance
Saffron	*Crocus sativus*	Fragrance, colouring
Fenugreek	*Trigonella foenum-graecum*	Fragrance
Others		
Juncus odoratus	*Cymbopogon schoenanthus*	Fragrance
Aspalathus	*Alaghi maurorum*	Thickener, fixative
Orcanette (root)	*Anchusa tinctoria*	Colouring
Xylobalsamum	Judean balsam tree bark	Thickener
Spatha	*Phoenix dactylifera* fruit husk	Thickener, fixative
Honey	–	Preservative
Wine	*Vitis vinifera*	Fragrance
Sodium chloride	–	Preservative, drying agent

been removed (except in the perfume shop at Paestum in Italy) and hearths are scarcely characteristic. However, by combining all the information we have on the archaeological remains, the iconography and knowledge of the traditional processes, we can begin to form an impression of how perfume was produced in antiquity, although the archaeology of the installations is not precise enough to answer questions about the composition of the perfume itself – here chemical analysis may help. Scented oils were conserved in flagons of metal, ceramic and glass and in some of them, found mainly in tombs but also in shipwrecks (e.g. the *Saint-Gervais 3* shipwreck) or in the cities destroyed by the eruption of Vesuvius in A.D. 79, the remaining organic deposits can be analysed.[9,13]** For example, analyses of the contents of perfume bottles discovered at Pompeii (in Regio I 11, 5) have shown that they once contained an olive oil-based perfume.[14] Flagons from tombs of the Valadas Roman necropolis at Saint-Paul-Trois-Châteaux contained animal and vegetal fats.

More recently, *unguentarii* found in the necropolis of Rue Charcot at Lyon were analysed by the Laboratoire Nicolas Garnier, who identified several components, not only vegetable oils such as olive oil but also animal fats and vegetal gums; in several cases it seems that the flagons did not contain perfume but instead a vegetable decoction, perhaps a medicine.[15] But in all cases, the volatile components that would have been the most instructive for recreating the perfumes have vanished and chemical analyses of ancient vases are still too few to understand their make-up.

Experiments to recreate ancient perfumes have been carried out since the beginning of the twentieth century.[16] More recently, after the discovery of a possible perfume workshop in the En Geddi oasis, on the western shore of the Dead Sea (Israel), a team from the Italian Centro Nazionale della Ricerca, under the direction of G. Donato, recreated perfumes following indications given by Dioscorides and Pliny. The results of these experiments were presented in an exhibition in Atlanta (GA, USA).[17] Experimental archaeology projects have also been developed in other fields, such as wine production. Working in conditions similar to those of antiquity, a team of academic researchers and

** Gas chromatography analysis proved that zoomorphic vases from Corinth contained a base of vegetable and animal fats.

wine makers tried to recreate antique wines, mainly using indications given by Columella, a first century A.D. agronomist. A Roman-type wine cellar was constructed, with a huge lever press and several tanks and jars for fermentation. By adding aromatics and fruits such as fenugreek, iris and quince, and mixing the must with grape juice concentrated by heating to increase the alcoholic degree, the final product had similar characteristics to southern Spanish wines, such as sherry.[18]

In the same way, the aim of the project 'Seplasia'†† is to recreate perfumed oil on the basis of ancient sources and archaeological data, using processes similar to those of antiquity, in order to rediscover ancient fragrances and to characterize their components.

Experimental

Botanical Sources and Plant Materials

Botanical sources, plant materials and their origins were as follows:

Sesame oil	*Sesamum indicum* L.	India
Jasmine flowers	*Jasminum grandiflorum* L.	France
Cardamom	*Elettaria cardamomum* (L.) Maton	Sri Lanka
Cinnamon	*Cinnamomum verum* Presl.	Sri Lanka
Saffron	*Crocus sativus* L.	Iran
Myrrh	*Commiphora myrrha* Engler.	Somalia

Honey (Lune de Miel, Gan, France) and sodium chloride were purchased from supermarkets in Nice, France.

Antique Perfume Formulation

Jasmine flowers (50 g) were placed in 500 ml sesame oil and the mixture was stirred with hands previously rubbed with honey. After 24 h the mixture was filtered and jasmine flowers were pressed (with hands rubbed with honey) to recover the fragrant oil. Sodium chloride was added and after a few minutes it was filtered; 50 g new jasmine flowers and 4.5 g cardamom were added to the oil and the mixture was stirred with hands rubbed with honey. After 24 h, the steps were repeated to six cold digestions to obtain about 125 ml oil. Then 14.4 g myrrh, 7.5 g cinnamon and 1.7 g saffron were added to the oil. Finally, after 2 h, the mixture was filtered into a glass bottle coated with honey.

HS–SPME

Headspace–solid-phase microextraction (HS–SPME) was performed with an SPME Combipal autosampler (CTC Analytics, Switzerland) equipped with a divinylbenzene/carboxen/polydimethysiloxane fibre (DVB/CAR/PDMS, 50/30 μm) purchased from Supelco (Bellefonte, PA, USA). The fibre was conditioned according to the manufacturer's recommendations prior to analysis. 2 g oil were placed in a sealed 20 ml SPME vial. The stabilization of the headspace was obtained by equilibration of the vial at room temperature during one night. The extraction was carried out with a sampling time of 90 min at 25°C. After sampling, the fibre was thermally desorbed into the GC injection port, which was equipped with a 0.75 mm i.d. inlet liner, for 2 min at 250°C. Before sampling, the fibre was reconditioned for 5 min at 250°C.

†† Seplasia is the name of a famous place in Roman Capua where perfumers used to make and sell perfumes.

GC–FID and GC–MS

GC and GC–MS analyses on apolar columns were carried out using an Agilent 6890N gas chromatograph apparatus equipped with a flame ionization detector (FID) and coupled to a quadrupole Agilent 5973N network mass selective detector working in electron impact (EI) mode at 70 eV (scanning over a 35–400 amu range). The gas chromatograph was equipped with two fused-silica capillary columns, HP-1 (polydimethylsiloxane, 50 m $\times$ 0.2 mm i.d., film thickness 0.33 μm). The analytical parameters were identical for GC and GC–MS analyses; carrier gas, helium; constant flow, 1 ml/min; injector temperature, 250°C; mode, splitless (2 min); temperature programme, raised from 50°C to 250°C at 2°C/min; FID temperature, 250°C; transfer line temperature, 280°C; ion source temperature, 230°C.

GC–MS analyses on polar column were carried out using a Hewlett-Packard 5890 series II/5971A system equipped with a fused-silica capillary column HP-20M (polyethyleneglycol, 50 m $\times$ 0.20 mm i.d., film thickness 0.1 μm): carrier gas, helium; constant pressure, 190 kPa; injector temperature, 220°C; mode, splitless (1 min); temperature programme, raised from 50°C to 220°C at 2°C/min; transfer line temperature, 230°C, ion source temperature, 140°C; ionization energy, 70 eV; electron ionization mass spectra were acquired over the mass range 35–350 amu.

Compound Identification

Identification of the constituents was based on computer matching against commercial libraries (NIST98, Wiley), laboratory-made mass spectra libraries built up from pure substances and MS literature data.[19] Identification of the components was also based on the comparison with literature data of GC retention indices (RI) on apolar and polar columns, calculated using a series of *n*-alkanes (C_5–C_{18}).[20,21]

Results and Discussion: Reconstitution of Antic Perfumes

Our attempt of reconstitution was based upon a modern traduction of Dioscoride's treatise, *De Materia Medica*.[6] Dioscoride usually used the same general scheme to describe all his recipes. First, the name of each recipe was mentioned; each perfume was known by the name of the principal ingredient or that of the city in which it was mainly produced, such as *cardamomon*, made from cardamom seeds or *Mendesion*, from Mendes in Egypt. However, some famous perfumes were also known by the name of their inventor, e.g. the *megalleion*, a very desirable perfume, took its name from Megalos.

Dioscoride further described the various steps of the recipe giving in more or less detail the quantities of raw materials (vegetable oil, flowers, spices, resins . . .), the heating of the mixture, the time of exposure to the raw materials, the filtration steps, etc. When necessary, Dioscoride gave additional indications on the vegetable oil that had to be processed before the addition of the main fragrant raw material.

In several recipes, we noticed that Dioscoride described the production of different grades of perfumes by recovering the main fragrant material after each filtration in order to pour on again a new batch of fresh vegetable oil. We assume that these perfumes of inferior qualities, probably cheaper than the first quality, were sold to inferior social classes. Dioscoride also indicated the ideal number of repetitive exposures to the fragrant material in order to obtain the most fragrant oil. Once the perfume had been obtained, Dioscoride gave advice about storage conditions. He sometimes mentioned the description of existing varieties, the place where the perfume was mainly produced and also the origin of the best quality. Finally, he described in detail the expected effects on the body and its 'good' way of use.

The reconstitution and analysis of a jasmine perfume named *iasmelaion* was selected among Dioscoride's recipes. In order to understand the way of reproducing an antique perfume, we present here the translation of the text used for this study, taken from: T. A. Osbaldeston, R. P. A. Wood. *Dioscorides: De Materia Medica, Book One: Aromatics.* Ibidis Press: Johannesburg, **2000**:

> 1–77. IASMELAION.
> That which is called jasme is made among the Persians from the white flowers of jasmine – two ounces of which are placed into an Italian pint of sesame oil, then changed and softened again as described in the manufacture of liliaceum [1–62]. The use of this is entertained among the Persians at their banquets for the sweet scent that it yields. It is good for the whole body after bathing, for those who want warmth and relaxation. It has a heavy sweet smell, so that many do not willingly use it.'

This brief recipe referred to a more detailed recipe, *liliaceum*, presented below:

> 1–62. SUSINON.
> Susinum is also called lilinum or liliaceum and is made as follows. (. . .) Take three and a half pounds of this thickened oil and a thousand (counted) lilies, and having stripped off their leaves, put them in a broad but not deep jar. Pour in the oil, stir it around with your hands (that have been previously rubbed with honey) and let it stand for a day and a night. The next morning pour it into a cupped strainer and presently (when it is strained) separate the oil on top from the water that is strained out with it, because it will not permit the water with it, like rosaceum [1–53], but when heated together it grows hot again and is spoiled. Pour it out again into other jars smeared with honey, first sprinkling a little salt in there and taking away the filth carefully as it gathers together. Take the strained aromatic stuff out of the basket, and placing it into a broad jar pour in on it again the same amount of the aromatised oil as at first. Put in ten teaspoons of bruised cardamom, stir it well with your hands, and after waiting a little strain it out, removing the filth off from that which runs out. Pour on the oil again a third time, repeat [the procedure] throwing in the cardamom and the salt with it, and press it out (first smearing your hands with honey). That which was the first strained out will be the best, the second the next after that, and the third the least. Then take another thousand lilies and strip off their leaves, lay them in order and pour on them the oil that was first strained out. Work methodically, doing the same things over again as you did at first, mingling cardamom [as before and afterwards straining it out]. Do the same the second and the third time, placing into it the cardamom, afterwards straining it out and repeating the procedure. As often as you steep fresh lilies in there, [by so much] you shall have the ointment stronger. Finally when it seems to you that you have enough, mix with every preparation seventy-two teaspoons of the best myrrh [1–77, 1–73, 4–116], ten teaspoons of crocus and seventy-five teaspoons of cinnamon. Some take the same amount of crocus and cinnamon (having pounded and sifted it), put it into a jar with water, and pour on it the ointment from the first pressing: afterwards (leaving it alone a little while) they put it into little dry jars (first smeared around with gum or myrrh and saffron and honey diluted with water). Do the very same things to the second and third pressings. (. . .)'

One first notices the difficulty in interpreting the method of producing the perfume, since several steps of the recipe are not described in enough detail. In addition, another problem was to identify the raw materials in order to make sure that our botanic identifications were correct. Was jasmine cultivated during this period at this place? How did perfume makers purchase all their ingredients?

From the current knowledge of archeology, coming either from various excavation sites or profound reading of the ancient texts, the existence of a commercial 'route of spices' that supplied the entire Mediterranean basin is now commonly accepted. It is thus likely that perfume makers would have certainly disposed of several raw materials mentioned in Dioscoride's treaty, depending, however, on their availability and price.

We tried to determine which part of the *liliaceum* recipe Dioscoride referred to in the *iasmelaion* recipe. Apparently, Dioscoride referred to the part of the recipe presented above, including the use of flowers and the addition of spices.

The measuring units used in Dioscoride texts are well known; the Italian pint corresponds to 500 ml, pounds and ounces correspond to roman units and thus represent 324 and 27 g, respectively. Concerning the teaspoons described in translated texts, a spoon of about the size of that found during archaeological excavations was used. The raw materials used and the main steps of the *iasmelaion* reconstitution are presented in Table 2.

The first cold digestion consisted of mixing jasmine flowers and sesame oil with hands rubbed with honey. Honey was used also to coat the jar receiving the intermediate mixtures and the final perfume. The role of honey was not clearly explained in the ancient texts, even though its use was widespread during antiquity.[22] One can assume that such a specific use for perfume preparation and storage might be related to its antioxidant and antiseptic properties. After 24 h, the mixture was filtered. Dioscoride mentioned the necessity of removing any residual water released by the flowers in order to avoid the degradation of the fragrant oil. The subsequent addition of salt in the recipe could thus be interpreted as a way to remove water. In addition, salt was extensively used as an excellent preservative during antiquity.[23] The next steps (not shown in Table 2) described the production of a second and a third grade of perfume by re-using the flowers used for the best quality. Following Dioscoride's recipe of *iasmelaion*, we submitted the first oil obtained to a second cold digestion, using bruised cardamom as an additional ingredient. Every step was repeated again to obtain premium quality oil until a total of six cold digestions had been performed, since the number and the time of cold digestions were not specified in the recipe: 'after waiting a little, strain it out . . . when it seems to you that you have enough'.

The last step consisted of the final addition of myrrh, saffron and cinnamon. Although a final filtration was not mentioned in the recipe, we chose to remove the solid part of the mixture after 2 h of exposure in order to perform olfactory evaluation under more convenient conditions. Depending on the desired aspect of the final product, one could decide to use it as either an unguent or as an oil for body care.

Table 2. Raw materials and steps in antique perfume formulations

Raw materials

Jasmine
(*Jasminum grandiflorum* L.)

Sesame oil
(*Sesamum indicum* L.)

Cardamom [*Elettaria cardamomum* (L.) Maton]

Cinnamon
(*Cinnamomum verum* Presl.)

Saffron
(*Crocus sativus* L.)

Myrrh
(*Commiphora myrrha* Engler.)

Principal formulation steps

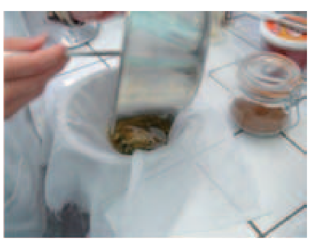
1. Addition of sesame oil to jasmine flowers (first cold digestion)

2. Mixture with hands rubbed with honey

3. After 24 h, oil filtration

4. Sodium chloride addition

5. After sodium chloride filtration, the oil is blended with new jasmine flowers and cardamom (second cold digestion)



6. After 24 h, each step is repeated until six cold digestions have been done

7. Final addition of myrrh, cinnamon and saffron

8. 2 h exposure

9. Filtration and storage in a vial coated with honey

The fragrant oil was subsequently analysed by SPME–GC. In a former study this solvent-free sample preparation technique was applied successfully to determine the volatile constituents of olive oil.[24] Thus, we used the extraction and analysis conditions previously optimized to characterize qualitatively the volatile composition of our fragrant oil. The quantitative composition will be discussed in a future study. The results are summarized in Table 3. Ethyl acetate and acetic acid (19.5%), 1.8-cineole (16.3%), linalool (11.8%), α-terpinyl acetate (10.5%), β-myrcene (7.1%) and benzyl acetate (4.6%) were identified as the main constituents. Each raw material used for the production of *iasmelaion* was analysed using the same method to assign the origin of the compounds identified in the fragrant oil. The contribution of jasmine was related to the presence of linalool, benzyl acetate, limonene, benzyl alcohol and (*Z*)-jasmone. We also observed the presence of styrene (0.1%). This constituent was not previously identified among the volatile constituent of jasmine flowers but its occurrence in the volatile extracts of many natural products is widely described in the literature. However, the presence of this constituent needs to be carefully considered because its high affinity with the DVB/CAR/PDMS fibre may lead to an overestimation, as we demonstrated in a previous study on benzoin gum.[25] Cardamom provided significant amounts of α-terpinyl acetate, 1,8-cineole, sabinene and linalyl acetate in the SPME extract.

Table 3. Volatile constituents of Iasmelaion obtained by HS-SPME–GC

Compound[a]	LRI[b] HP-1	LRI[b] HP-20M	% ± SD[c]	Identification method[d]	Assumed origin[e]
Ethanol	–	905	1.5	MS, LRI, Std	jasm., card., myrr., cinn., saff.
Methyl acetate	514	828	4.0	MS, LRI	jasm., card., myrr., cinn., saff.
Ethyl acetate	602	870	19.5 ± 0.4[f]	MS, LRI	jasm., card., myrr., cinn., saff.
Acetic acid		1387		MS, LRI	jasm., card., myrr., cinn., saff.
Propyl acetate	698	–	tr	MS, LRI	
Isobutyl acetate	752	984	tr	MS, LRI, Std	
2,3-Butanediol	756	–	tr	MS, LRI	
Hexanal	773	1041	0.2	MS, LRI, Std	jasm., card., myrr., cinn., saff., ses. oil
Ethyl butyrate	780	–	tr	MS, LRI	
Butyl acetate	795	1032	0.2	MS, LRI	
(*Z*)-3-Hexenol	834	1335	tr	MS, LRI, Std	jasm.
(*E*)-2-Hexenol	843	1358	tr	MS, LRI	
Hexanol	846	–	tr	MS, LRI, Std	
p-Xylene	851	–	0.1	MS, LRI	myrr.
3-Methylbutyl acetate	852	–	0.1	MS, LRI, Std	
2-Methylbutyl acetate	855	–	0.1	MS, LRI, Std	
styrene	868	–	0.1	MS, LRI, Std	jasm.
α-Thujene	928	1004	0.9	MS, LRI	card., myrr., cinn.
Benzaldehyde	933	1462	0.1	MS, LRI, Std	jasm., cinn.
α-Pinene	934	1000	0.9	MS, LRI, Std	myrr., card., cinn.
Dihydromyrcene	940	–	0.1	MS	
Camphene	944	1027	tr	MS, LRI, Std	myrr., cinn.
6-Methyl-5-hepten-2-one	966	1288	tr	MS, LRI, Std	jasm., myrr., cinn., saff.
Sabinene	970	1082	3.4 ± 0.1	MS, LRI, Std	card., myrr., cinn.
β-Pinene	974	1064	0.3	MS, LRI, Std	myrr., cinn.
Octanal	981	–	tr	MS, LRI,Std	jasm., cinn.
β-Myrcene	983	1126	7.1 ± 0.1	MS, LRI, Std	card., jasm., myrr., cinn.
α-Phellandrene	1000	1134	0.4	MS, LRI, Std	cinn., saff.
Benzyl alcohol	1004	1812	0.1	MS, LRI, Std	jasm.
α-Terpinene	1013	1136	0.9	MS, LRI, Std	card., cinn.
p-Cymene	1017	1223	0.6	MS, LRI, Std	jasm., myrr., cinn.
1,8-Cineole	1027	1166	16.3 ± 0.6	MS, LRI, Std	card., myrr., saff,
Limonene	1028	1155	4.3 ± 0.1	MS, LRI, Std	jasm., myrr., cinn., saff.
(*Z*)-β-Ocimene	1031	1196	0.3	MS, LRI, Std	jasm., cinn.
(*E*)-β-Ocimene	1043	1211	0.5	MS, LRI	jasm., card., cinn.
γ-Terpinene	1054	1203	0.9	MS, LRI, Std	card., jasm., myrr., cinn.
(*E*)-Sabinene hydrate	1058	1418	0.1	MS, LRI	card.
Terpinolene	1084	1236	0.7	MS, LRI, Std	jasm., card., myrr., cinn.
Linalool	1085	1512	11.8 ± 0.1	MS, LRI, Std	jasm., card., cinn.
Isophorone	1089	1529	0.2	MS, LRI	saff.
4-Ketoisophorone	1102	1630	0.4	MS, LRI	saff.
allo-Ocimene	1115	–	0.1	MS, LRI	jasm., card.

Table 3. *Continued*

Compound[a]	LRI[b]		% ± SD[c]	Identification method[d]	Assumed origin[e]
	HP-1	HP-20M			
Benzyl acetate	1132	1676	4.6	MS, LRI, Std	jasm.
Terpinen-4-ol	1159	1571	0.4	MS, LRI, Std	card., myrr., cinn.
α-Terpineol	1171	–	1.0[f]	MS, LRI, Std	card., cinn.
Safranal		1587		MS, LRI	saff.
Octyl acetate	1191	–	0.1	MS, LRI, Std	card., myrr.
Nerol	1209	–	tr	MS, LRI, Std	jasm.
2-Phenylethyl acetate	1223	–	tr	MS, LRI, Std	
Cinnamaldehyde	1229	–	1.2 ± 0.1	MS, LRI	cinn.
Geraniol	1234	1801	0.1	MS, LRI, Std	
Linalyl acetate	1240	1526	2.7 ± 0.1	MS, LRI, Std	card., cinn.
Bornyl acetate	1265	–	tr	MS, LRI	card., cinn.
Terpinen-4-yl acetate	1281	–	0.1	MS, LRI, Std	
Methyl geraniate	1299	–	tr	MS, LRI	
α-Terpinyl acetate	1336	1656	10.5 ± 0.4	MS, LRI, Std	card., cinn., myrr., jasm., saff.
Neryl acetate	1342	1688	0.1	MS, LRI, Std	card.
Geranyl acetate	1360	1719	0.3	MS, LRI, Std	card.
(*Z*)-Jasmone	1365	1875	0.1	MS, LRI	jasm.
α-Copaene	1375	1452	0.1	MS, LRI, Std	cinn., card., myrr.
β-Bourbonene	1382	1478	0.1	MS, LRI	myrr.
β-Elemene	1387	1553	0.7	MS, LRI	myrr., card.
β-Caryophyllene	1416	–	0.4	MS, LRI, Std	cinn., jasm., card., myrr.
Furanodiene	1483	1818	0.4	MS	myrr.
α-Selinene	1490	–	0.1	MS, LRI	card., myrr., cinn.
γ-Cadinene	1505	–	tr	MS, LRI	card., myrr.
δ-Cadinene	1514	–	tr	MS, LRI	myrr., cinn.
Furanoeudesma-1,3-diene	1609	–	0.4	MS	myrr.
Lindestrene	1616	–	0.2	MS	myrr.

[a] Compounds are listed in order of their elution time from an HP-1 column. Compositional values <0.1% are denoted as traces (tr).
[b] LRI, retention indices as determined on HP-1 and HP-20M columns, using the homologous series of *n*-alkanes.
[c] Reported GC–FID percentages are relative; SD, standard deviation over three analyses.
[d] Identification methods: MS, comparison of the mass spectrum with those of the computer mass libraries or from the literature; LRI, comparison of retention index with those from the literature; Std, analysis of an authentic sample.
[e] jasm., jasmine; card., cardamom; myrr., myrrh; cinn., cinnamon; saff., saffron; ses. oil, sesame oil.
[f] Coelution, percentage corresponding to the total integration of the two compounds.

Furanodiene, furanoeudesma-1,3-diene and lindestrene were assigned to myrrh. Isophorone, 4-ketoisophorone and safranal were characteristic of saffron, and cinnamon was mainly represented by cinnamaldehyde and β-caryophyllene. Sesame oil afforded very few volatile compounds. As checked by means of a blank analysis, only hexanal was identified as a constituent contributing to the odour profile.

The olfactory evaluation of the fragrant oil was performed by an experienced perfumer. *Iasmelaion* was described as giving a very pleasant jasmine note, quickly hidden by strong spicy notes (cinnamon, clove, pepper). The perfume also provided terpenic notes and a persistent styrax bottom note, making it recall as the ancestor of Opium™ (Yves Saint-Laurent). Although cinnamon was predominant, this floral composition was very surprising and interesting.

Acknowledgements

The authors are grateful to Albert Vieille S.A., Grasse, France, for furnishing raw materials and helpful discussions, to the 'Bastide du Parfumeur' Mouans-Sartoux, France (Mr Gabriel Bouillon), for providing plant material, and Perfum.com, Saint Vallier de Thiey, France (Mr Raphaël Haury), for olfactive evaluation. We thank N. Garnier and T. Silvino, who provided us with a full report on their analysis. We thank CNRS for financial support.

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