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# "ROSE SCENTED GERANIUM" A *PELARGONIUM* GROWN FOR THE PERFUME INDUSTRY

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## INTRODUCTION

The interest shown in growing scented-leaves geranium dates from the mid nineteenth century. At that time, the real "rose of the Levant", *Rosa damascena* Mill. (*Rosaceae*), became rare and its essential oil reached excessive levels of price in Paris (Héricart De Thury, 1845). This situation forced the perfumers to look for new essential oils with a rose scent and they remembered the distillation trials of rose geranium carried out by Recluz in Lyon (France) in 1819. In 1844, Demarson established the first plantations in the sunshine of Provence, near Grasse (France).

But, from a physiological point of view, the geranium cultivation requires generous sunshine and well-drained soils, rich in organic matter. The crop dreads frost and temperatures below 2°C are harmful to the growth and even to the survival of the plant.

And, from an agro-economic point of view, the traditional cultural practices are very labour consuming : preparation of cuttings for plantation, muck-spreading, hoeing for weed control, manual harvesting and firewood duty for distillation are costly operations. Furthermore, in the countries with pronounced winter season, the risk of frost leads to an annual crop management and needs to shelter the cuttings till the next plantation.

For these reasons and because of the lack of mechanized solutions, the production of rose geranium oil did not take a long time to abandon the South of France and to move to more merciful skies, where an abundant and cheap labour existed. From 1850, the French colonies of Northern Africa and Indian Ocean seem predestined for growing geranium. The crop is introduced to Algeria in 1847 and developed on an industrial scale by Léon Chiris and Monk towards 1865 (Naves, 1934; Igolen, 1941). In 1880 the plant is introduced to Reunion Island where it will be distilled for the first time in 1882. After the turn of the twentieth century, the crop was attempted in Corsica, Italy, Morocco, Tunisia, Egypt, Southern Russia, Congo, Kenya, Tanganyika, Madagascar, India, Spain, Portugal, Brazil, Comoro Islands... The most recent development was in the People's Republic of China in the fifties.

## TAXONOMY AND GENETICS

Since the beginning, and as pointed out by Guenther (Guenther, 1977), "the taxonomy of the plants which are cultivated in various parts of the world for the production of commercial geranium oil has been a matter of much controversy and has given rise to considerable confusion". Basically, the name geranium itself is incorrect, since the species involved in the production of rose geranium oil are all *Pelargonium*, not *Geranium*. All these plants belong to the *Geraniaceae* family, but *Geranium* and *Pelargonium* form two different genera beside three other which are *Erodium*, *Monsonia* and *Sarcocaulon*.

In the same way, the cultivars which, by a great misnomer, are referred to as *P. capitatum* or *P. graveolens* or *P. radens* are not natural species. According to the revision of the genus *Pelargonium*, carried out since 1977 under the supervision of J.J.A. Van der Walt at the University of Stellenbosch (Republic of South Africa) (Van der Walt, 1985), we now know that the true *P. radens* H.E. Moore (Moore, 1955) and *P. graveolens* L'Hérit. (L'Héritier Dom De Brutelle, 1789) are mint-scented species (Van der Walt and Demarne, 1988) with 88 chromosomes (Albers and Van der Walt, 1984), while *P. capitatum* (L.) L'Hérit. is a 66 chromosomes species very poor in essential oil and with a very faint rose scent (Demarne, Viljoen et al., 1993; Viljoen, Van der Walt et al., 1995). All these natural species are originated from Southern Africa.

According to past and recent researches in genetics (Ducellier, 1933; Kuchuloriya, 1964; Kuchuloriya, 1968; Demarne, 1989; Demarne and Van der Walt, 1989), we also know that all the cultivars grown worldwide for rose geranium oil production are interspecific hybrids between *P. capitatum,* on one hand, and *P. graveolens* or/and *P. radens*, on the other hand. Most of these cultivars have 77 chromosomes (Tamai, Tokumasu et al., 1958; Tamai, Tokumasu et al., 1963; Payet, 1981; Demarne, 1989) and their main features (chromosome number, habit, leaf and flower shape, essential oil yield and composition...) are in-between those of their natural parents. The oil composition in particular depends on the *P. capitatum* parent which transmits the ability to synthesize geraniol and citronellol rather than isomenthone. Furthermore, the presence of 6,9-guaiadiene and/or 10-epi-γ-eudesmol, which usually distinguishes between the African type and Bourbon type oils (Vernin and Metzger, 1983), is also inherited from the *P. capitatum* parent.

Thus, the cultivars of rose-scented geranium should be named *Pelargonium* hybrids of *P. capitatum* x *P. radens* or *Pelargonium* hybrids of *P. capitatum* x *P. graveolens*. The choice of the proper variety, when establishing a new production anywhere in the world, is fundamental : it will directly influence the yield and the quality of the oil.

## CULTIVATION

Growing rose geranium for essential oil production is not easy. For a profitable crop, this fragile bush must be cultivated as a perennial on a 3 to 5 years basis, and every harvest can question its survival. This is especially true when the plantations are established in tropical countries, where fungal and bacterial diseases are numerous and particularly virulent.

Probably because of that fragility, rose geranium is usually grown on small plots of about one to two hectares, sometimes less. Most of the cultural practices are done by hand, making the cultivation resemble horticulture and keeping the farmer busy throughout the year.

### *Propagation*

All the cultivars of rose geranium are heterozygotic and highly polyploid (x=11; "2n"=7x=77 chromosomes). Due to their hybrid character and their unusual chromosome number, the plants are

usually male-sterile and do not set seed (Ducellier, 1933; Tokumasu, 1970). Their agronomic performances and the quality of their essential oil depend on complex genetic balances. Therefore, asexual propagation is the rule and the cultivars are usually propagated by terminal stem cuttings.

Rooting of the cuttings is influenced by the nutritional state (carbohydrates/nitrogen ratio), the sanitary state and the age of the mother-plants (Bricheteau, Alegre et al., 1980; Dartigues and Lemaire, 1980; Vidalie, 1980), by wilting and application of root-promoting hormones and fungicides (Michellon, 1975; Michellon, 1976; Demarne, 1981; Demarne, 1992; Rajeswara Rao, Bhattacharya et al., 1993), and by water supply, temperature and growing medium.

From a common experience, the best rooting is obtained from herbaceous terminal stem cuttings of 12-20 cm long (7-10 nodes) with 4-5 terminal leaves, grown in nursery under partial shade and mist irrigation. Day/night temperature should be 21-27/12-17°C and the growing medium should provide a sufficient porosity as well as a good water-holding capacity to allow good aeration and good water supply. Treating the base of the cuttings with a mixture of indolebutyric acid (IBA/0.1-0.2%) and captane (10%) in talcum powder is also recommended. Under those conditions, the cuttings quickly develop a profuse root system and are ready for transplanting after 40-60 days.

However, beside these ideal conditions of propagation, a traditional method is still in use in most of the producing countries. It consists of planting unrooted woody cuttings, 30-40 cm long, directly into the field, just after the rainy season, but unfortunately, the mortality rate after planting is usually high.

*Planting and cropping system*

The profitability of a geranium crop is directly correlated to the number of plants per hectare. Depending on soil and climatic conditions, plant spacing varies between 80x30 cm and 100x60 cm; the optimum being of about 35000 plants/ha. Rose geranium is generally cultivated as a pure crop installed on a cleared land. Cuttings are planted in holes or furrows dug manually, at the bottom of which the farmer usually puts a handful of manure (farm manure or composted distillation residues) - and sometimes, some fertilizer, lime, and a slow-released insecticide to prevent the young roots from white grubs and other soil insects' attacks.

During the first six months, geranium grow quite slowly, leaving large portions of the inter-row uncovered. Intercroping geranium with short duration legumes or maize allows a profitable use of this space and an efficient weed control (Lougnon, 1924; Prakasa Rao, Singh et al., 1984; Narayana, Prakasa Rao et al., 1986; Narayana, Prakasa Rao et al., 1986; Prakasa Rao, Singh et al., 1986; Garin, 1987).

In the traditional cropping systems, rose geranium is thus a perennial weeded crop, and in the tropical countries, rainfall combined with hoeing for weed control causes severe erosion. Fields on a slope must be arranged in bench terraces with swathes, and new concepts of sustainable agriculture, based on planting geranium into a controlled cover crop, are now developed. These techniques favour the microbiological activity of the soil and lead to a better preservation of soil fertility and a better water management (Michellon, Dejante et al., 1994; Michellon, Dejante et al., 1994; Michellon, Seguy et al., 1995; Perret, Michellon et al., 1996). The foliage production and the life span of the crop are thus improved, but the drawback is that this agriculture is more technical, especially in keeping the cover crop under control, without damaging the rose geranium.

*Fertilization*

In traditional cultivation, the fertilization of rose geranium usually combines chemical fertilization, restitution of organic matter and sometimes liming (Garin, 1987). Because of their relatively high costs, the chemical fertilizers are sparingly used (Chabalier, 1992). The composted residues of distillation and/or an organic manure bring back to the field some fertilizer elements, but the time of application and the placement of these residues mainly benefit the intercrops. Generally speaking, the fertilization level is low and the traditional system is not stable.

*Chemical fertilization*

The first fertilization trials on rose geranium date from 1894. According to Fritz (Fritz, 1976), a high yield crop can produce 7 tons of dry matter/ha/year, which correspond to average exportations of 100 kg N, 32 kg $P_2O_5$, 165 kg $K_2O$, 250 kg CaO, 28 kg MgO, 15 kg Na and 10 kg S. The dry matter is about 18 to 20% of the total biomass. These figures obtained on Reunion Island have been confirmed in India (Prakasa Rao, Singh et al., 1988).

It is now well known that rose geranium responds to nitrogen applications but on the condition that balanced fertilization is used and other elements are not limited, especially phosphorus (Chabalier, 1992). On Reunion Island, Fritz has observed linear increases of herb yield with nitrogen applications up to 150 kg/ha/year (Fritz, 1976). Similar results were obtained in India with doses up to 225 kg/ha/year (Prakasa Rao, Singh et al., 1986; Prakasa Rao, Singh et al., 1988).

Rose geranium also responds to phosphate applications. Fritz (Fritz, 1976) observed that the essential oil yield is linked to the P content of the plant and, as far back as 1899, Dantes (Dantes, 1899) reported that massive applications of superphosphate (2 t/ha) doubled the herb yield. Weiss (Weiss, 1967) reported similar results in Kenya and advised the renewal of the application of 200 kg/ha of $P_2O_5$ after the fifth harvest of a geranium crop. Other hints are given by Chabalier (Chabalier, 1992) to grow rose geranium in andic soils and to prevent phosphorus deficiencies when this element is made unavailable by the absorbing complex of the soil.

Rose geranium can withstand low levels of potassium before displaying deficiency symptoms. Those symptoms and a yield drop appear when the K content of the plant drops down 0.3% of the dry matter (Chabalier, 1992).

Lastly, spraying miconutrients (B, Cu, Zn, Mo...) is sometimes recommended (Ladaria, 1968; Arumugam and Kumar, 1979; Dhakshinamoorthy, Arumugam et al., 1980; Prakasa Rao, Singh et al., 1984). Under certain conditions, those trace elements can favour the oil production.

In practice, the type of chemical fertilizer will depend on the soil conditions and on the cropping system; the doses and the applications' timetable will depend on the possible level of intensification. For an intensive crop, the theoretical fertilization, based on the restoration of the exported nutrients and on the normal losses is 150-200 N/60-80 $P_2O_5$/150-200 $K_2O$.

N, P and K can be combined by using a complete fertilizer at the time of planting and then once a year at the end of the rainy season. In intensive cultivation N (urea or calcium ammonium nitrate) can be applied in 2-4 split doses. In any case, when the fertilizer is put in the hole at the time of planting, it is important to avoid the contact between the cutting and the fertilizer to prevent burning.

*Lime and organic manure*

When rose geranium is traditionally grown without a cover crop, its erosive character often requires incorporated applications of lime and organic matter for restoring the soil fertility. In Reunion Island, an additional practice consists in a several years' growth of bush fallow of *Acacia decurrens* before planting.

In all cases, the use of organic manure in combination with fertilizers at the time of planting is advocated. The beneficial impact of organic matter to the microbial activity and soil structure is well known. Michellon (Michellon, 1987) observed that application of 5 to 10 tons of distillation residues on a geranium intercropped with legumes leads to a better root development and a two-fold increase of essential oil yield compared with geranium grown as a sole crop. Further to that observation, Chabalier (Chabalier, 1992) gave the composition of different sources of organic matter suitable for rose geranium cultivation.

*Irrigation*

Rose geranium is rather drought resistant and dreads excess of water. Irrigation is usually left to nature and when a waterlogging risk exists, the cuttings are planted on ridges for insuring a good drainage, as in the Nile valley in Egypt.

But, attempts made to grow geranium in harsh weather conditions in Egypt (Koriesh and Atta, 1986), in Israel (Sanderovich, Putievsky et al., 1983) or in India (Singh, Chandrashekhara et al., 1996; Sabina Aiyanna, Farooqi et al., 1998; Singh, 1999), demonstrated that rose geranium can take advantage of different techniques of irrigation and fertigation to increase both its herb and essential oil yields per hectare. However, a negative correlation is often found between plant water content and the essential oil content, as a result of irrigation treatment (Sanderovich, Putievsky et al., 1983).

In intensive cropping systems, both plasticulture and cover crops allow the reduction of the water needs of the crop.

*Weed control*

Weed control is important in geranium cultivation. Under tropical and sub-tropical climates, weeds grow very quickly and tend to invade the fields, competing with the crop for space, light, water and nutrients. When left uncontrolled, the weeds smother the young cuttings which become etiolated; they limit growth and branching leading to poor yields (Trémel, 1992; Rajeswara Rao and Bhattacharya, 1997). Moreover, some odoriferous weeds can be harvested and distilled with the crop and will affect the quality of the oil.

In most producing countries, manual weeding is practised. This operation is very labour-consuming and requires from 60 to 125 days of labourer/ha/year. Even when herbicides are used in the inter-row, weeding the row is always done manually. In practice, weed control is a combination of manual hoeing, herbicide application, cultural tricks (mulching, plasticulture...) and suitable rotations or co-cultivation with soil-cover plants.

In all cases, man must keep in mind that hoeing tends to deconstruct and to dry the soil, increasing the erosion. On the other hand, pre-emergence, systemic or contact herbicides can be used but they must be checked against their ability to be traced later in the essential oil. Different active ingredients have been tested on numerous weeds in geranium cultivation (atrazine, diuron, glyphosate, paraquat, metribuzine, nitrophen, alachlor...); their selectivity and the way to use them is available in the literature (Gravaud, Roura et al., 1976; Srinivasan, Sambandamurthy et al., 1979; Didelot, Garin et al., 1985; Garin, 1987; Trémel, 1992).

*Pest and diseases*

Rose geranium in cultivation is subject to attacks of insects, nematodes, fungi and bacteria. Pests and diseases are so numerous that it seems inconceivable to give the chemicals a miss and to produce organic geranium oil in profitable conditions.

A little information is available on pests in Egypt and China, but in Reunion Island, a general survey (Quilici, Vercambre et al., 1992) of the harmful insects revealed that geranium is attacked by at least fourteen different species belonging to the *Hemiptera* (6 species), *Coleoptera* (3 species), and *Lepidoptera* (5 species). Among the most important pests are the white grubs of *Hoplochelus marginalis* Fairmaire (*Coleoptera*, Fam. *Scarabaeidae*), the cockchafers *Cratopus humeralis* Boh. and *C. angustatus* Boh. (*Coleoptera*, Fam. *Curculionidae*), the whitefly *Trialeurodes vaporariorum* Westwood (*Hemiptera*, Fam. *Aleyrodidae*), the scale insect *Pseudaulacaspis pentagona* Targioni-Tozzetti (*Hemiptera*, Fam. *Diaspididae*) and the defoliating caterpillar of *Lobesia vanillana* (*Lepidoptera*, Fam. *Tortricidae*). An efficient integrated control of these insects exists as a combination of light trapping, agronomic controls (minimum tillage, cover-crop...), chemical controls (insecticides spraying, chemical trapping...) and biological controls (with insects and fungi which parasitize the larva) (Quilici, Vercambre et al., 1992).

Nematodes have also been reported as important pests, especially in India. According to Rajeswara Rao (personal communication, 1999) several species of *Criconemoides*, *Helicotylenchus*, *Meloidogyne*, *Pratylenchus*, *Scutellonema* and *Xiphenema* damage the crop and inflict yield losses up to 75.8% (Doraswamy, Sundaram et al., 1979; Kumar and Nanjan, 1985; Anita and Vadivelu, 1997). Chemical control seems possible as well as biological control with a nematophagous fungus and/or companion cropping with nematicidal plants (periwinkle, marigold...) (Anita and Vadivelu, 1997).

Fungal and bacterial diseases are also numerous and very virulent, and a considerable literature is available on these subjects. Several lists were established by different plant pathologists who have studied and referenced the pathogens in the producing countries (Bouriquet, 1946; Sinaretty and Trémel, 1992) (Rajeswara Rao, 1999, personal communication). Wilt, dieback, leaf blight, leaf spot, root and stem rot, anthracnose, are common and can be ascribed to several species of fungus (*Colletotrichum*, *Botrytis*, *Septoria*, *Cercospora*, *Armillaria*, *Rosellinia*, *Phomopsis*, *Pythium*, *Fusarium*...) and bacteria (*Pseudomonas solanacaerum*). Certain of those pathogens cause severe damages leading sometimes to the total destruction of the crop and the impossibility of growing geranium again on the same plot (eg. wilt caused by *Pseudomonas solanacearum*). Generally speaking, the suggested control measures of these pathogens are only partially satisfactory, even if anthracnose and botrytis can be controlled by spraying specific fungicides. Despite the need for resistant varieties, a genetic improvement program does not seem reasonable or economically viable regarding the economic importance of the crop in the individual oil-producing countries.

## HARVESTING

The geranium oil is contained in glandular trichomes (Demarne and Van der Walt, 1989) which are mainly located on both surfaces of the young leaves, on the young stems, on the buds and on different parts of the inflorescences. Oil is thus obtained from the top young parts of the plant and, because of the perennial character of the crop,

harvesting geranium must fulfil two opposing objectives : the maximum production of herb and the preservation of the subsequent shoot development ability (Demarne, 1992).

Therefore, the performance of a particular harvesting technique depends on the actual herb and essential oil yields, but also on the regrowth of the plants and the rate of mortality. The quality of the shoot development will govern the frequency and the yield of the subsequent harvests on individual plants, while the rate of mortality will drastically influence the number of plant per hectare, the time spent to fill in the gaps and hence the survival and the medium-term profitability of the crop.

From that point of view, manual harvesting with secateurs, knives or sickles is the best way for harvesting geranium as it prioritizes an individual approach of the plant architecture and tends to optimize both the herb yield and the regrowth ability. On the contrary, mechanical harvesting carries out an intermediate trimming of the whole field and only optimizes the labour productivity. Whatever principles they are based on (Korezawa, Tanida et al., 1967; Boyko, 1977; Paillat, 1987; Ducreux, 1993), the mechanical harvesters developed up till now partly solve the question of plant regrowth in cutting the utmost top of plants only (Demarne, 1992) and in the different trials conducted mainly in Reunion, the loss of earnings was offset in increasing the harvesting frequency.

For all these reasons, geranium is still largely harvested by hand. The crop is ready for the first harvest 6 to 8 months after planting. Subsequent harvests are made at 3-5 months intervals, depending on plant development, weather conditions, crop management, labour availability... Details on the way to prune rose geranium in different conditions are available in the literature (Doraswamy and Sundaram, 1982; Demarne, 1992).

In all cases, harvests must be performed on clear and sunny days. The plant material is usually left one day on the inter-row to wilt. It is then transported to the distillery. At that time, heaping up or chopping the plant material is inadvisable to avoid fermentation, and distillation must be done quickly.

## DISTILLATION

Distillation is the ultimate step of geranium cultivation. Because the essential oil is located in secretory sacs at the end of glandular hairs, its extraction is easy. Therefore, all the techniques of distillation and organic solvent or supercritical fluid extraction give results; the differences being only in the yield and the quality of the products. In practice, there is only a market for water-distilled essential oil. Only Egypt produces and markets on request small quantities of geranium concrete and/or absolute.

Different kinds of distillery can be found in the producing countries, which correspond to different technical, economical and social situations. The numerous direct-fired stills of about 1000 litres capacity operating in the countryside in Reunion Island, in China, in Egypt or in India have nothing in common with the batteries of 3 000 litres stills equipped with a separate boiler and encountered in certain factories, in Egypt or in Reunion Island.

However, the major part of the world production of geranium oil is distilled in small units, belonging to small holders or to village communities. Processing 300-350 kg of plant material in a 1000 L direct-fired equipment lasts 90 to 150 minutes and gives a 0.1 to 0.2 % yield of oil. A complete description of the traditional equipment, including the limiting factors, the possible improvements and the detailed field distillation procedure is available in the literature (Demarne, 1992). However it is interesting to point out that under such conditions, the distillation of one hectare can take 40 to 100 days/year, without counting the time necessary for firewood or charcoal duty.

## ESSENTIAL OIL PRODUCTION

### *World production*

After more than one century of attempts, the so-called "rose geranium oil" is now produced mainly in the People's Republic of China, Egypt, Reunion Island and India. A few kilos are also exported from Madagascar and smaller quantities are sometimes produced in Algeria, Morocco and Russia.

China is now by far the biggest producer of geranium oil. The cultivation is concentrated in the Yunnan Province, in the district of Binchuan. From an agricultural point of view, geranium is grown as a perennial (3 years) and makes the best of the horticultural techniques of the Chinese agriculture; the fields can be harvested four times a year, and the average oil yield is pretty good (±45 kg/ha). An accurate assessment of the Chinese production and domestic consumption is precluded by the lack of reliable statistical data. Nevertheless and for several years, the annual production seems to be about 80 to 110 t, of which 20 to 30 tons are used on the Chinese domestic fragrance market and 60 to 80 tons are exported

Egypt is the second biggest producer. Major areas of production are primarily in upper Egypt, mainly Bani Sweif and Fayoum. In this country the crop is annual, with two harvests a year. The oil is of the African type, as it contains substantial quantities of 10-epi-γ-eudesmol. According to different sources, the production in the past 10 years has averaged 50 to 55 tons per year, with important year to year fluctuations.

On Reunion Island, despite big efforts made by the French government and the local authorities to support the production through research and development programs, and the remarkable results obtained by the agronomists, the production of rose geranium oil has drastically declined over the years. The unequalled quality of the real Bourbon oil, universally recognized in high class perfumery, has not been sufficient to maintain the price at levels that can balance the increasing standard of living of the population and nowadays, the annual production is about 6 tons, all of which is exported.

India has a small production (±2 t) in south hills and plains (Nilgiri hills, Pulney hills) which is used for its internal market.

### *International trade and demand*

The main importers of geranium oil are France and USA. However, whereas the bulk of US imports are consumed domestically, there is a big re-export trade from France. The French exports consist, on one hand, in pure geranium oil and, on the other hand, in a proportion of oil that has been reprocessed and blended to users' specifications. In most cases these oils are of very high quality.

"The international trade appears to be relatively stable and it is very likely that the USA and France will continue to dominate the import trade for the foreseeable future" (Robbins, 1984). The average annual imports of geranium oil into the USA are about 60 to 65 tons, and into France about 90 to 95 tons. Japan imports about 15 tons and the remainder of the European Community an other additional 30 tons. Other appreciable markets include Brazil, India and a few others, but the trade statistics often fail to show geranium oil separately, and it is difficult to gauge the extent of these trade flows in the majority of cases.

For all these markets combined, the total need is about 220 tons, when trade between the USA and European Community, and within the European Community, is excluded. All in all, the supply no longer fits the demand, and despite a production adjustment between the main sources, there is still an extra demand for 20 to 25 tons of a very high quality geranium oil to replace the Bourbon type.

Geranium oil is purchased and assessed mainly on the basis of its odour character and, in a lesser extent on its suitability for the preparation of rhodinol. For many years it was traded almost exclusively on the basis of samples and this procedure still applies, even if, for certain origins (Bourbon and certain Yunnan oils), it is possible to buy on the basis of product description only.

In the importing countries, geranium oil is traditionally marketed through a comprehensive network of intermediaries of various types including import merchants, dealers, commissioned brokers, agents and the processing and compounding houses. Geranium oil prices vary widely depending on the quality, the origin and also unforeseen supply fluctuations. Bourbon oil from Reunion Island traditionally commanded a price premium over oils from other sources, but the shortage of this quality in the recent years has caused switches to oils from alternative sources, causing the price of the latter oils to rise sharply.

## OIL COMPOSITION AND QUALITY

Geranium oil is one of the most important raw materials used in perfume and soap industries. This product is rather well known and has been studied for the end of the 19th century. Its physical properties and its chemical composition have given rise to a great number of publication, as the analytical techniques progressed.

The works of Naves *et al.* (Naves, 1934; Naves, Brus et al., 1935; Naves, 1944; Naves, 1951; Naves, 1957; Melera and Naves, 1961; Naves, Lamparsky et al., 1961; Naves and Tullen, 1961; Naves and Ochsner, 1962; Naves, Oschner et al., 1963; Benesova, Chou et al., 1964; Romanuk, Herout et al., 1964; Krepinsky, Samek et al., 1966), Teisseire *et al.* (Teisseire and Corbier, 1964; Pesnelle, Corbier et al., 1969; Pesnelle, Corbier et al., 1971), Giannotti *et al.* (Giannotti, 1966; Giannotti and Schwang, 1968; Giannotti and Schwang, 1968), Wolff *et al.* (Wolff, Ma et al., 1963), Klein *et al.* (Klein and Rojahn, 1977; Rojahn and Klein, 1977), have been amongst the important contributions to the knowledge of the chemical composition of the geranium oil. Since 1960, the development of gas-chromatography and associated identification techniques (GC-MS, GC-FTIR...) have sped up this acquisition of knowledge with the works of Garnéro *et al.* (Garnéro and Buil, 1969; Garnéro, 1974; Garnéro, Joulain et al., 1980), Peyron (Peyron, 1962; Peyron, 1962), Teisseire *et al.* (Corbier and Teisseire, 1966; Pesnelle, Corbier et al., 1969), Kapétanidis *et al.* (Heuss, Kapétanidis et al., 1969), Timmer *et al.* (Timmer, Heide et al., 1971; Ter-Heide, De Valois et al., 1975), Kami *et al.* (Kami, Otaishi et al., 1969), Kaiser *et al.* (Kaiser, 1984), Demarne *et al.* (Guérère and Demarne, 1985; Demarne, 1989), Vérin *et al.* (Vérin, Bianchini et al., 1998), and others. More recently, techniques of multidimensional chiral gas chromatography coupled with mass spectrometry (enantio-MDGC/MS) (Ravid, Putievsky et al., 1992; Kreis and Mosandl, 1993; Casabianca, Graff et al., 1996; Wüst, Beck et al., 1996; Allemant, 1998; Wüst and Mosandl, 1998; Wüst, Rexroth et al., 1998; Wüst, Rexroth et al., 1998; Doimo, Fletcher et al., 1999; Wüst and Mosandl, 1999; Wüst, Reindl et al., 1999) has led to a better knowledge of the enantiomeric distribution of chiral compounds in the oils of different origins and, in a certain extent, could be used as criteria for authenticity assessment.

Based on this mass of academic information, a lot a applied research programs were carried out on the variation in the quality of the oil and to determine the influence of different internal, external and management factors like the variety (Gailleton, 1959; Korezawa and Tanida, 1969; Naragund and Divakar, 1983; Obaladze and Kuchuloriya, 1986; Angadi and Vasantha Kumar, 1989; Demarne and Van der Walt, 1989; Zobenko and Arinshtein, 1989; Kulkarni, Baskaran et al., 1997), the climate and the soil (Charapov, 1968; Kalix, 1968; Michellon, 1980; Sun Handong, Ding Jingkai et al., 1985; Rajeswara Rao, Sastry et al., 1990; Bhattacharya, Kaul et al., 1993; Yan Dongwei, Zhang Zhengju et al., 1994; Southwell, Stiff et al., 1995; Rajeswara Rao, Kaul et al., 1996; Chakib, 1998), the plant part (Pieribattesti, 1982; Mallavarapu, Rajeswara Rao et al., 1997), the crop age (Kaul, Rajeswara Rao et al., 1998), the shade (Kaul, Rajeswara Rao et al., 1997), the fertilization (Korezawa, 1961), the presence of weeds and diseases (Rajeswara Rao and Bhattacharya, 1997; Rajeswara Rao, Bhattacharya et al., 1999), the irrigation (Singh, 1999), the time of harvesting and the technique used (Yoshida, Ikawa et al., 1968; Mani and Sampath, 1981; Ravid and Putievsky, 1983; Tiberghien, 1989; Baret, 1991; Prakasa Rao, Ganesha Rao et al., 1995; Rajeswara Rao, Kaul et al., 1996), the post-harvest treatments (Mani, Mohandass et al., 1981; Rajeswara Rao, Bhattacharya et al., 1992; Rajeswara Rao, Kaul et al., 1999), the distillation (Guérère, Demarne et al., 1985), the conditions and duration of storage (Mani, Mohandass et al., 1981; Mahmoud, El-Gmal et al., 1983; Kaul, Rajeswara Rao et al., 1997)...

All these studies would tend to confirm that as far as the production scale is concerned, the three main important factors influencing the oil quality are the variety, the soil and climate conditions and also the distillation procedure. The quality variations of the oil mainly concern the relative concentrations of eight important compounds which are (E)- and (Z)-rose oxides, linalool, geraniol, citronellol, isomenthone, guaia-6,9-diene and 10-epi-$\gamma$-eudesmol.

*Chemical composition of the oil*

Several reviews were published by Lawrence (Lawrence, 1976; Lawrence, 1978; Lawrence, 1984; Lawrence, 1985; Lawrence, 1986; Lawrence, 1988; Lawrence, 1992; Lawrence, 1992; Lawrence, 1994; Lawrence, 1996; Lawrence, 1999) during the last years, which amalgamated the information published in the scientific literature on the composition of different geranium oils from different geographical origins, but also on the identification of new components of the oil and on the enantiomeric ratios of some chiral compounds ($\alpha$-terpineol, rose oxides, linalool, citronellol, menthone, isomenthone). Rose geranium oil is a very complex product that contains hundreds of compounds, some being hydrocarbons (aliphatic, aromatic, monoterpenic, sesquiterpenic with different skeletons), and the others being oxygenated with alcohol, phenol, oxide, aldehyde, ketone, acid, ester and ether functional groups.

Only thirty compounds are regularly and individually present at more than 0.3% of the oil; together they represent about 90% of the essential oil and are sufficient to form the basis of the essential oil quality. The other components appear as traces, the correct identification of which is sometimes difficult, especially when sesquiterpenic hydrocarbons are concerned.

*Quality*

So, the quality of rose geranium oil used in the fragrance industries depend on

several criteria, from analytical determinations to compliance with international standards. The standards classify the geranium oils into three types, according to their origins : the Chinese type is applicable to the essential from the People's Republic of China, the African type is for the oils from Algeria, Morocco and Egypt, and the Bourbon type for the essential oil from Reunion Island. The other oils are produced in too small quantities to be the subject of particular standards; when they appear on the international market, they are bought on the basis of their similarities with one of the three types above-mentioned.

According to the standards (Anonymous, 1996), the geranium oils are easily characterized by their physical properties (specific gravity, optical rotation, refractive index, flash point, solubility in ethanol and colour), chemical properties (acid value, ester value, carbonyl value) and above all by their organoleptic properties (aspect, colour, odour) and their gas chromatography profiles. The rose geranium oils are liquid, limpid and less dense than water ($0.882 \leq {}^{20}d_{20} \leq 0.892$). Their colour vary from amber-yellow (Egypt) to yellowish-green, brownish-green or green (Bourbon). They all have a strong rose scent with a more or less pronounced mint note depending on their origin.

Capillary gas chromatography using polar or non-polar phases reveals that the major components of the oils are the monoterpenic alcohols (citronellol, geraniol, nerol, linalool, α-terpineol) and their esters (citronellyl formate, citronellyl butyrate, citronellyl propionate, citronellyl tiglate, geranyl formate, geranyl butyrate, geranyl propionate, geranyl tiglate, neryl formate), the monoterpenic ketones (menthone, (-)-isomenthone), the monoterpenic aldehydes (neral, geranial), the monoterpenic oxides ((E)- and (Z)-rose oxides), monoterpenic hydrocarbons (α-pinene, myrcene, limonene...), an aromatic ester (phenylethyl tiglate), several sesquiterpenic hydrocarbons (guaia-6,9-diene, germacrene D, β-bourbonene, β-caryophyllene), two sesquiterpenic ketones (furopelargones A and B) and a sesquiterpenic alcohol (10-epi-γ-eudesmol).

Among these components, geraniol, citronellol, linalool, isomenthone, geranyl formate, citronellyl formate, guaia-6,9-diene and 10-epi-γ-eudesmol play a particular role, as they allow to distinguish between oils of different origins and different varieties of rose geranium. The Bourbon type oil from Reunion Island contains large quantities (ca. 5-7%) of guaia-6,9-diene but does not contain 10-epi-γ-eudesmol. Chinese geranium does not contain 10-epi-γ-eudesmol as well. On the contrary, the African types contain 10-epi-γ-eudesmol (ca. 4-5%) but do not contain guaia-6,9-diene (Pesnelle, Corbier et al., 1969; Pesnelle, Corbier et al., 1971). However, when the varieties and the growing conditions are not fixed for long, both guaia-6,9-diene and 10-epi-γ-eudesmol can be found together, as in some Indian oils (Kaul, Rajeswara Rao et al., 1997), but on the international market this lead to suspicions of blending between oils of different origins.

Linalool is a good indicator of the extraction process as it seems to be formed from geraniol during steam water distillation. When the herb is extracted with solvent, the concrete thus obtained contains only small quantities of linalool (<0.5%). But when the herb is distilled for more than two hours in a traditional field equipment, the oil contains 5 to 10% of linalool; the sum (linalool+geraniol) being constant in both the solvent extract and the water-distilled oil (Guérère, Demarne et al., 1985). In the industrial process, because of a limited contact between the herb and hot water, the linalool percentage is in-between those limits (ca. 4-5%).

Geraniol, linalool and citronellol together also typify the different qualities. Citronellol is usually higher in Chinese oil (38-40%) and Egyptian oil (31-33%) than in Bourbon oil (19-21%). On the contrary, geraniol and consequently linalool are higher in Bourbon oil (16-19% / 7-10%) than in Egyptian oil (13-15% / 5-6%) and Chinese oil (7-10% / 2-3%); the ratio (linalool+geraniol)/citronellol varies from 0.3 (Chinese oil) to 0.6 (Egyptian oil) and 1.4 (Bourbon oil). The amount of geranyl formate and citronellyl formate are proportional to the corresponding alcohol in the oils; isomenthone is slightly higher in Bourbon oil (8-9%) than in the others oils (ca. 6%).

As far as the Indian oils are concerned, these figures vary depending on the variety under cultivation. The oil of certain cultivars even content more geraniol than citronellol.

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