32nd International Days of Essential Oils & Extracts

Cinnamon, patchouli, vetiver, osmanthus, extraction techniques and more.

Jean-Jacques Étienne

The 32nd International Days of Essential Oils & Extracts was held this past autumn in Digne, France. What follows is a collection of highlights.

—Editor

Perfume Creators and Their Public

Speaker Véronique Nyberg (IFF) arrived in the field of perfume creation after receiving her doctorate in scientific studies. From this training she developed a strong motivation to create and experiment restlessly.

Today, she said, perfumers must understand the "brand DNA" of customer projects in order to find the specific characteristics that will translate into a proper fragrance expression. This can necessitate a large number of trials. For example, Nyberg's creation of Paco Rabanne's *Invictus* required something like 5,500 mods. She compared the process of servicing 50 briefs per year to an actor who is supposed to play six or seven different roles at the same time. This comes in addition to cost controls dictated by the market. This confluence of challenges, she said, stimulates creativity.

Sources of perfumery inspiration differ, she said, and include architecture, fashion, literature and painting. Meetings and dialogues with other creators are a source of inspiration; such enrichment can be encountered at the École de la Mode (Fashion School) or at the ENSAD (National High School of Decorative Arts).

Nyberg told the audience how she composes a fragrance similar to how a painter creates a picture, finding the fragrant "shapes" that will express the brand DNA. One way to captivate the consumer is to be inspired by a well-known and familiar smell and to transcend it in a new inspired expression, such as "black olive" fragrance.

To conclude, with a great optimism, Nyberg told the audience that she keeps her "nose in the wind" to feed her creativity and ensure "creative eclecticism."

Indonesian Cinnamon

Indonesian cinnamon trees (*Cinnamomum burmannii* Blume) are grown mainly on the island of Sumatra, near Mount Kerinci, said Olivier Bernard (Tripper Nature). Most of these fields were created during the Dutch colonial era. The production of spices (cinnamon, nutmeg, clove, vanilla, pepper, ginger, etc.) is a secondary activity compared to the production of palm oil, coffee and cocoa. Bernard discussed the process of insuring a

The event's scientific committee comprised, from left: Jean-Claude Bayle (IFF), Jean-Jacques Etienne (ATN Conseil), Michael Moisseeff (Asquali),Yves Comte (chairman, organizing committee), Xavier Fernandez (Nice University) and Hugues Brevard (Robertet).

Veronique Nyberg (IFF) discussed the contemporary job of the perfumer, including the need to understand "brand DNA."

Olivier Bernard (Tripper Nature) discussed Indonesian cinnamon.

complete traceability from the tree to the preparation of the cinnamon products.

Fifteen- to 20-year-old trees are cut and left to grow again naturally. The composition of the products depends upon the age of the tree part collected. The bark is separated from the trunk and dried in the open air where it rolls in on itself while taking on a red-brownish color. Pieces of bark are collected in nets and transported to Jakarta where steps of preparation will provide the different qualities offered to clients: rolls, chips, and ground and sieved powders.

The fragrance of the material results from its volatile oil. Annual production of cinnamon products is 30,000 to 40 000 tons, said Bernard. The aromatic materials are available as tinctures (0.3–0.4% cinnamaldehyde), extracts in ethanol or PPG (1.1–1.2% cinnamaldehyde), super resins (66–68% cinnamadehyde), and ground powders ranging from 18–60 mesh.

Uses are mainly centered on the food industry: pastries, sweets, flavored tea and ice creams.

Indonesian Patchouli and Vetiver

The availability of patchouli essential oil has frequently suffered irregularities, either due to climate disorders (for example, the 2004 tsunami) or as the result of economic issues (farmers moving on to more profitable opportunities), explained Frederic Badie (Payan Bertrand). Consequently, development of a reliable source is a major need. This can be achieved by instilling financial security into the supply chain and exploiting the speed of information technology.

Badie discussed just such a project on the island of Sulawesi, near Aceh. About four to five tons of fresh leaves are obtained per ha (hectare) of growing area, leading to approximately 1 ton of dried material. Once the material is dried and ground, the leaves are distilled with water vapor in iron tanks, yielding a colored essential oil. About 200 kg of essential oil can be produced based on the collections of four collecting families during one year, during which three operations process 32 tons of fresh leaves via 320 traditional distillations. Variations of yield are observed according to regions. Aceh mountain essential oils have a content of 33–36% patchoulol, while those of the valley contain 31.5% and those of Java contain 29%, said Badie.

The development of the Sulawesi project allowed the building of two stainless steel units equipped with a reliable electric line, while the quality control of products obtained was managed by Payan Bertrand's laboratory in Grasse. Several qualities of patchouli oil are produced from the operation, including those with the following: chocolate, cocoa nib, dry and woody, humus, and fresh and camphor.

This production channel yields 1,600 tons/year and delivers, via an association of producers, financial help and investment. This support provides producers guarantees to sell at a stabilized price, pushing away temptations to stop the cultivation. This creates a safe, sustainable source of patchouli essential oils.

The same approach has been developed for the production of vetiver essential oil, said Badie. About 200 kg of vetiver essential oil can be produced from 50 to 90 tons of fresh roots, which in turn cover 15–25 ha on land cultivated and managed by 40 people per day for 20 days, and leading to 35–60 distillations. The distillation is operated under pressure:

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Sandrine Presle (Gattefossé) discussed the extraction of water present in plants while retaining all its richness of composition, including metabolites, oligoelements and other ingredients of interest for cosmetic preparations.

The analysis of natural products involves chromatography (GC, HPLC) completed with the identification of separated components with the help of spectrographic techniques such as infrared spectroscopy (IR, FTIR) and mass spectroscopy (MS, MS/MS), which provide the identification of the separated components, said F. Tomi (Ajaccio University).

- at 2–2.5 bars for 18 hours, a product rich in vetiverol is obtained
- at 4–5 bars for 12 hours, a product rich in β-vetivone is recovered

The supply channel is limited at the moment, with 4–5 tons per year of essential oil being produced. Built and organized on the same basis as the Indonesian patchouli channel described by Badie, the vetiver supply chain is at present able to satisfy the company's needs for compositions.

Osmanthus: from Flower to Extract

Osmanthus is a large wild tree found in the Anhui and Guangxi provinces in China, said Fabien Durand (IFF). Its flowers are small and collected with the help of a cloth on the ground. Three different botanical varieties are:

- *Osmanthus fragrans thunbergii*, "gold" with yellow flowers
- *Osmanthus fragrans latifolius*, "silver" with white flowers
- *Osmanthus fragrans aurantiacus*, "reddish" with darker flowers.

Once collected, flowers are submitted to a maceration for two to five months in pickling brine realized via the addition of 25 kg of salt to 100 kg of water in containers. Post-collection treatment induces the formation of lactones, the transformation of carotenoïds leading to ionones, and the degradation of α-ionone, creating theaspiranes such as 7-oxo-dihydrotheaspirane, which has a woody/cedar, dry fruit and patchouli leaf odor.

The extraction is made with hexane, which yields 0.15% of concrete. The washing of the concrete with ethanol and the elimination of waxes produces an absolute (yield: 70%). Osmanthus concrete is used to flavor teas and pastries, while the absolute is used in perfumery.

The fragrance differs according to the flowers extracted. The heart is fruity, dry with lactonic notes and, for certain qualities, notes of hay, tobacco and even animalic aspects (castoreum, civet-like). It is sometimes waxy, while other varieties have a fruity-floral profile suggesting rum ethers, or can express some green, sour or garlic notes. The GC analysis of osmanthus reveals components such as:

- dihydro-β-ionone
- dihydro-β-ionol
- β-ionone
- organic acids (linoleic, linolenic)
- lactones (γ-decalactone).

Water: a Solvent for Extraction

Loïc Pétigny (BASF/Avingon University) discussed cooperation between BASF and the University of Avignon, which has created the GREEN laboratory (Groupe de Recherches en Eco-Extraction des produits Naturels). The purpose of the lab's activities is to find alternative approaches to the use of organic solvents, particularly water, which presents, depending upon the conditions in which it is used, solvent properties that are not exclusively polar. In fact, water presents physical and chemical properties that allow it to vary its solvent abilities according to the process conditions and parameters such as pressure, temperature, physical state and possible addition of auxiliary molecules.

Different examples have been presented to show these abilities:

- Maceration and percolation to treat Reine des prés (syn: meadowsweet)
- Hydrodistillation with a preliminary grinding for rosemary
- Enzymatic extraction with the use of specific catalysts to extract particular active agents and organic acids
- Extraction using the water *in situ*, inside the plant, and heating with microwave at appropriate pressure
- Extraction by water drawn at its subcritical state and at a temperature above 100°C under high pressure; in these conditions the water polarity decreases and its behavior is similar to that of organic solvents (*Gingko biloba* extracts)
- Extraction involving surfactants, allowing the extraction of organic components insoluble in water with the help, for example, of Triton X-100 (INCI name: octoxynol-9)[a]

[a]Triton X-100 is a trade name of Dow Chemical

Frederic Badie (Payan Bertrand) discussed patchouli from Indonesia.

All these processes permit the exclusion of the use of current organic solvents such as acetone, methanol and dichloromethane, which may be deemed environmentally unsafe or uneasy to handle (warehousing, waste treatment, etc.), and shifting toward water, for which industrial handling is easier and free from environmental blame.

Organic Extracts

Sandrine Presle (Gattefossé) discussed the extraction of water present in plants while retaining all its richness of composition, including metabolites, oligoelements and other ingredients of interest for cosmetic preparations. These original extracts can be used to replace the water in formulas, partially or completely. These water extractions are prepared in an organic-certifiable manner. Presle described the processes as follows:

- Hyperfrequence: the material is submitted to an instantaneous sublimation at the appropriate temperature supplied by a microwave oven; water is recovered by condensation.
- Flash distillation: the vegetal is submitted to a reduced pressure at ordinary temperature; the water vapor escapes and is recondensed.
- Vacuum dehydration: operated at 40°–60°C; the vaporized water is cooled and recondensed.

The water obtained is sterilized through filtering membranes and can be concentrated. It is kept in sterile conditions. Various examples of the extraction technology were presented, which demonstrated cosmetic interest, including:

- antiradical/antioxidant effects (ginger, lotus, rice)
- TGF-β stimulation (cherry, kiwi)
- anti-tyrosinase activity (kiwi, lemon)
- stimulation of keratinocytes growth (apple)

Analytical Performances of NMR on Natural Products

The analysis of natural products involves chromatography (GC, HPLC) completed with the identification of separated components with the help of spectrographic techniques such as infrared spectroscopy (IR, FTIR) and mass spectroscopy (MS, MS/MS), which provide the identification of the separated components, said F. Tomi (Ajaccio University). Their combination generally allows a good identification of the different components of a mixture.

NMR spectroscopy studies can be operated either on already separated components in view to identify their nature, but NMR can also operate on a mixture, even quite complex mixtures such as an essential oil or fractions prepared from a more complex mixture. On previously purified compounds, it will be possible to perform an NMR study of proton ^{1}H and ^{13}C, even involving the bidimensional NMR. As far as the examination of complex substances is concerned, ^{13}C-NMR is preferred to ^{1}H-NMR, despite its low natural isotopic occurrence and its lower sensitivity compared to that of ^{1}H, because:

- C makes the skeleton of all organic molecules; there will be as many resonance lines as the number of C in the molecule
- appropriate instrumental adjustments allow for one to achieve simpler spectra and improved signal dispersion
- it allows work at ambient temperature

These ^{13}C-NMR studies are able to give details about the composition of essential oils and go deeper in the knowledge of certain minor components that nevertheless frequently represent an important parameter linked with the quality, genuineness and source of a product.

Many striking examples have been described, including showing the identification of isomers:

- in the essential oil of *Cupressus officinalis*, the presence and contents in α- and β-cedrene and in α- and β-funebrene
- in the essential oil of *Xylopia aethiopica*, the identification of different vinyl cyclohexenes
- the transformation of thermosensitive molecules of the essential oil of *Cleistopholis patens*, including the transformation of germacrene B into γ-elemene

The very precise character of these studies also allows researchers to elucidate the geographical distribution of *Rosmarinus officinalis* around the Mediterranean Sea:

- the α-pinene/verbenone/bornyl acetate variety is spread in Corsica, Sardinia and Egypt
- the 1,8-cineole/α-pinene /camphor is growing in North Africa, the South of France, Italy, the Adriatic Coast, Greece and Turkey
- the myrcene variety is found in Portugal

Consequently, ^{13}C-NMR is revealed to be an extremely fruitful approach to directly analyze highly complex mixtures of components and, at the same time, a very sophisticated technique to detect isomers that cannot be detected by the usual analytical techniques.

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