

This article appeared in the August 2020 issue of Perfumer & Flavorist magazine

The Modern Way: Supercritical Fluid Extraction

Sustainable, affordable and delivering high-quality products, supercritical fluid extraction is a method for the ages.

BY EDEN STUART, associate managing editor

If any three words may define current consumer demand, a strong argument could be made for "clean, green and affordable." A 2019 survey by Grand View Research and Statista estimated the natural flavor and fragrance market in the United States will reach \$2,302 million in 2025, up from \$1,818 million in 2018^a. Natural ingredients, however, pose unique challenges—namely sustainability, cost and quality of product.

Enter supercritical fluid extraction (SFE). Though the building blocks of the process emerged nearly 200 years ago—in 1822, French physicist and engineer Cagniard de la Tour discovered the supercritical state of carbon dioxide; 150 years later, The Research Institute of Krasnodar in the Soviet Union had extracted more than 80 different plants¹—growing consumer demand has made the process more relevant than ever in F&F.

"Supercritical fluid extraction enhances the ability to address growing customer demand for authentic, natural taste and scent experiences," says Pierre Chauchadis, process engineer in charge of new developments at Firmenich. "This process preserves all the facets and the natural profile identity of the raw material, [delivering] genuine odor and taste, true to botanical freshness and complexity.

"It perfectly answers the clean label requests of the market: natural, 100% from the named source, process solvent-free. The expertise for all types of biomass extraction (fresh flowers, gums, dry and liquid materials) offers broad tonality coverage."

THE PROCESS

"Under its supercritical state (>75 bar; >31°C) CO₂ works as a very effective solvent to selectively extract the volatile part from a large choice of diverse biomasses used for perfumery and flavoring purposes," says Chauchadis. "[In this] state, CO₂ benefits of the high diffusivity of a gas and the good solvent efficiency of a liquid, optimizing the extractability of raw materials in terms of time and yield. The extraction selectivity and precision can be fine-tuned, changing the applied temperature and pressure of the CO₂ modifying its intrinsic density."

Due to the relatively low temperature in comparison to traditional technologies, thermal degradation of the extracted molecules is minimized. Additionally, the chemical inertia of the CO₂ does not induce transformation of the extracted molecules.

After the supercritical extraction, the CO₂ is depressurized in its gaseous state in a separator, releasing the extracted substances. In the separator the retrieved extract will have no solvent residue, a potential problem with traditional solvent extraction technologies.

Over the years, the number of raw materials available for supercritical CO₂ extraction—including fresh or dry solids and liquids—has increased, and knowledge on the different ways to prepare biomasses (including fermentation, roasting, sweating and drying) has improved extract quality and composition.

"At the beginning, we were working only with dry raw materials," says Firmenich perfumer Fabrice Pellegrin. "Then, we could do CO₂ extracts on fresh biomasses, and now liquid ones, which enables us to extend the palette offered by the extraction technique. SFE provides new colors to the perfumer's palette, increasing the creative possibilities."

"Technically, the method has not evolved much [though] extraction units are more and more performant and parameters can be adjusted more precisely," says Mane's Mathilde Voisin. "What has changed however is our knowledge in supercritical fluid extraction. Having our own production unit for several decades has considerably increased our empirical learning."

CLEAN AND GREEN

"All [these] described properties give supercritical CO₂ technology a clear advantage, extracting biomasses under very smooth conditions," says Chauchadis.

The low temperature, paired with the lack of solvent evaporation, means SFE demands significantly lower energy than standard extraction technologies, Chauchadis notes. Additionally, CO₂ can be selected from retrieval or recycling processes that offer lower environmental impact and reduce the carbon footprint further.

"They are also interesting from a regulatory point of view as they can enter clean label formulations," says Voisin. "Supercritical CO₂ extraction allows [the] label 'CO₂ extract of X' in food labelling. In cosmetic applications, the ingredient can enter the composition of a COSMOS bio certified product.

"Supercritical fluid extraction is listed as a natural process according to the ISO 9235 standards. Performed at moderate temperature, it requires a limited quantity of energy and water. Supercritical CO₂ extraction uses CO₂ gas, collected from ambient air or from industry wastes. It is recycled and reused several times during the extraction and allows the obtention of solvent-free extracts and waste. This waste material can thus be easily recycled as compost for soil to limit our environmental impact."

TOP NOTCH QUALITY

Along with sustainability and clean formulation benefits, supercritical fluid extraction has another positive—the process yields a superior product. "From an organoleptic point of view, supercritical fluid extraction is the ideal process to transcript the original scent and taste of a raw material," says Voisin.

"Thanks to their good penetration abilities and excellent solvent capacities, supercritical fluids allow [us] to extract the raw material's heaviest compounds as well as its most volatile molecules. Then, by adjusting the process parameters (pressure, temperature, time of contact), we are able to create a perfect balance between top and bottom notes and capture the full profile of the original raw material.

"When smelling our Lavender Pure Jungle Essence, its floral breeze and coumarin notes will immediately transport you to lavender fields; when tasting our Timur Pure Jungle Essence, you will experience both the fresh, citrus effect and the spicy profile of the Nepalese berries. Supercritical extracts, thus, naturally bring a complex note to fragrance or flavor applications."

Chauchadis noted that since Firmenich's acquisition of Evonik's supercritical CO₂ extraction technology unit at the end of 2019, the company's portfolio has been extended and diversified. The teas and coffees SFE range will create new opportunities in fragrance and flavor, such as oakwood and peanut, as well as for food applications.

POTENTIAL LIMITATIONS

The process is not without its challenges, however. Chauchadis notes that the equipment requires a relatively high investment and must be "flexible enough to work on multi-tonalities and, also, depends on the related natural biomass availabilities (timing and volumes)." Though this typically leads to relatively higher extraction costs, these costs are offset by "the high sensory and supercritical authenticity characteristics of the SC CO₂ extracts and, consequently, their usage at low dosage and reduced cost in use."

These challenges can be addressed by optimizing and fine-tuning production planning and management based on extract type, yield, timing and volumes.

"A further limitation of CO₂ extraction is the extractability of bioactive ingredients for food and health care," says Chauchadis. "These substances are usually of higher molecular weight and their solubility in CO₂ is limited. Therefore, auxiliary measures for extraction such as co-solvents or appropriate pre-treatment of the raw materials can be used."

"In theory, any raw material can be extracted with supercritical fluids; but natural extraction remains an experimental science, and some raw materials show little interest regarding their organoleptic properties but also their cost-effectiveness," says Voisin. "Thanks to our nearly 150 years of experience in naturals, we have been able to find ways to extract some challenging raw materials such as fruits (including coconut and prune), and, more recently, we have been working on delicate flowers with promising results."

PAST IS PRESENT (AND FUTURE)

Despite any potential challenge, SFE remains the method to beat when it comes to extraction technologies. "Supercritical fluid extraction ... makes it possible to create several different ingredients by adjusting the extraction parameters," says Voisin. "Thus, with one process performed on a unique raw material we can create a full range of extracts with different organoleptic profiles. Today, this is the only process that allows such versatility and precision."

Says Pellegrin: "Even though we have been pioneer in this technology, which has existed for several decades, I think, as a leader today, we have still got a lot to discover ... this is one of the most advanced, clean and sustainable technologies—an extraction method for the future."

a www.statista.com/statistics/830229/us-flavor-and-fragrance-market-revenue-byproduct/

Reference

1. Pellerin, P. (1991). "Supercritical Fluid Extraction of Natural Raw Materials for the Flavor and Perfume Industry." *Perfumer & Flavorist* (July/August).