# COMPARING EXTRACTION BY TRADITIONAL SOLVANTS WITH SUPERCIRITICAL EXTRACTION FROM AN ECONOMIC AND ENVIRONMENTAL STANDPOINT

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This study concerns the extraction of diverse natural aromatic raw materials (15 to 20 different raw materials).

## 1- Solid/liquid and solid/supercritical fluid extraction

Both of the above consist in separating one or more constituents contained within a solid matrix via solubilization in a liquid or a fluid in its supercritical state. We will compare the three following procedures:

- Batch solid/liquid extraction
- Continuous solid/liquid extraction
- Solid/supercritical fluid extraction in batch

## 2 – Comparing the three processes

### 2-1 Cost of industrial equipment:

The study was based on standard equipment of sufficient capacity to handle a minimum of 250 tonnes of plant matter per year per work station

- Batch extraction: two 4000-litre extractors, one evaporator and one finisher = 610,000 euros
- Continuous extraction: one screw extractor (diameter 0.5m, length 10m), one evaporator and one solvent remover =1,500,000 euros
- Extraction by supercritical fluid (CO2): two 600-litre extractors = 2,300,000 euros

### 2-2 Handling capacity per work station in 8 hours:

- Batch extraction: 200 to 300 tonnes of plant matter.
- Continuous extraction: 400 to 600 tonnes of plant matter. (It is to be noted that this type of equipment is designed to work continuously, as its name suggests, tripling its annual handling capacity).
- Extraction by supercritical CO2: 250 tonnes of plant matter.

### 2-3 Preparation of the raw material:

- Batch: rare, generally loaded as it comes, sometimes crudely crushed.
- Continuous: often necessary to ensure it proceeds correctly into the spiral (grinding, chopping, etc).
- Supercritical fluid: the product must be reduced to a powder to ensure quality extraction, correct loading and unloading (grinding, freeze-grinding, etc).

### 2-4 The extraction solvent:

- Choice**:** for extraction by traditional solvents, choice is determined by legislation governing the use of the extract to be obtained (food, cosmetics or perfumery) as well as client spec.s - which may be more restrictive. The choice influences the composition of the extract (different solubility parameters), organoleptic quality, and extraction yield. In the case of extraction by $CO_2$ in its supercritical state, the problem of choice only arises if one wants to use a co-solvent to modify the polarity of the $CO_2$ as an extraction solvent. In this case, one must not forget to use a co-solvent that also features on the food list in order to maintain the food status of the solvent.

- Equipment approval: any extraction unit using flammable and/or explosive volatile solvents is subject to legislation controlled by the Regional Authority for Industry, Research and the Environment (DRIRE). In other words, this applies to batch and continuous extraction but not to extraction by $CO_2$ (unless the co-solvent falls within the bounds of the legislation).

- Volume of solvent introduced:
  - Batch: a great deal, in the region of 10 to 20 times the weight of the plant matter. The volume of solvent that evaporates is lower (generally 2/3 of the volume introduced) because the final wash is used to do a first.
  - Continuous: three times the weight of the plant matter introduced, on average. All the solvent evaporates.
  - Supercritical $CO_2$: small, due to the permanent recycling of the solvent. In general, the same weight as the plant matter.

- Solvent loss:
  - Batch: 10 to 15% of the weight of solvent introduced.
  - Continuous: 3 to 5% of the weight of solvent introduced.
  - Supercritical $CO_2$: the $CO_2$ is recycled in its entirety during the extraction of any one raw material. But for this type of application (where the volume of $CO_2$ introduced does not justify the additional cost of setting up reservoirs and compressors to store $CO_2$ used to extract a given plant matter until it is needed to extract the same plant matter in the future) 100% of the solvent introduced and containing organoleptically active traces of the plant extract is lost and cannot be used to extract a different plant matter.

- Safety:
  Traditional solvents are generally flammable and explosive, hence the hefty safety measures imposed on their use.
  Non-flammable $CO_2$ offers safety guarantees. However, introducing a flammable co-solvent puts it in the same position as traditional solvents.

**2-5 Energy:**

- Batch: most energy consumption is accounted for by the solvent evaporating units that recover the extract, and "blowing" (injecting steam) to eliminate residual solvent from the "dregs" (plant residue after extraction). Energy is consumed at about 8 kWh per kilo of plant extract.
- Continuous: as the quantity of solvent introduced is three times lower, energy consumption is reduced by one third, i.e. approximately 3 kWh per kilo of plant extract.
- Supercritical $CO_2$: as solvent elimination takes place by simple decompression, energy consumption is low. It covers compressing liquid $CO_2$ and heating it to bring it up to supercritical state, then - after the extract has been separated - cooling the $CO_2$ which has become gassy in order to make it into a liquid. Energy consumption is in the region of 0.8 kWh per kilo of plant extract.

**2-6 Residues:**

- Batch: once the solvent has been removed by blowing with steam, residues from extraction by traditional solvents must be eliminated in centres approved by the DRIRE. In Grasse, the main plant extraction centre in France, the cost of elimination amounts to 120 euros per tonne if the solvent content of the residue is under 5000 ppm (composting) and 380 euros per tonne if it is higher (incineration). Transport costs must also be added in function of the distance to the treatment centre, plus a pollutant activity tax of 9.15 euros.
- Continuous: the cost per tonne is the same, only the weight of the residue to be eliminated is approximately 60% lower because solvents are removed by a heating process that gives a dry residue. This weighs less than residue impregnated with steam.
- Supercritical CO2: residues that have not come into contact with organic solvents can be used again - to feed cattle, for example.

**2-7 Wastewater:**

- Batch: residual water from blowing and rinsing the vats must be stored and eliminated by a centre approved by the DRIRE. There are approximately 500 litres of water for each tonne of plant matter treated. Elimination costs amount to 160 euros per tonne in Grasse, to which transport costs and the pollutant activity tax must be added.
- Continuous: as there is no blowing, just rinsing the equipment, the cost is halved.
- Supercritical CO2: no expense here because no residual organic solvent.

**2-8 Extraction costs:**

Whatever the technique, this cost will be situated within quite a wide bracket. The duration of extraction, the number of washes, the load per extractor and the preparation or not of the plant matter will all greatly influence the cost.
In order to minimise such parameters as cleaning when defining extraction costs, we looked at treatment costs for introducing a minimum of 5 tonnes of plant matter.

- Batch: between 0.35 and 4 euros.
- Continuous: between 0.175 and 2 euros.
- Supercritical CO2: between 3 and 8 euros.

It is important to note that the more costly extraction process by CO2 gives an extract that is already refined, is more concentrated and can therefore be used in smaller doses.

## 3 Incidence in terms of energy savings, the environment and health

**3-1 Energy savings:**

We have seen that energy consumption varies greatly between the extraction processes:

- Batch: 8 kWh/kilo of plant matter with the emission of 800 litres of CO2.
- Continuous: 3 kWh/kilo of plant matter with the emission of 300 litres of CO2.
- Supercritical CO2: 0.8 kWh/kilo of plant matter with the emission of 80 litres of CO2.

We must not forget that carbon dioxide is the principal agent responsible for the greenhouse effect. These figures come from a study carried out by APAVE as part of a request for aid submitted to the French Energy Management Agency (AFME), today known as the Agency for the Environment and Energy Management (ADEME). It is to be noted that extraction by supercritical CO2 is particularly advantageous in terms of energy consumption because of the small volume of solvent introduced, the separation of the extract by decompression, plus the fact that it is possible to recuperate the calories produced by the cold group (passage from gas form to liquid form) to feed the heating system (passage from liquid form to supercritical state). For information purposes, the hydro-distillation of one kilo of plant matter consumes between 10 and 30 kWh.

**3-2 Environment:**

Apart from the energy aspect - linked to the emission of $CO_2$ responsible for the greenhouse effect and the destruction of the ozone layer -, it is important to underline the following points:

- The emission of organic solvents into the atmosphere: between 100 and 150 Kg per tonne of plant matter treated for batch extraction, between 10 and 30 Kg per tonne for continuous extraction, and zero for supercritical $CO_2$ extraction in so far as the $CO_2$ emitted has been taken from the atmosphere or from the emission source. Under the joint action of the sun and $NO_x$ emitted by traffic, emissions of solvents into the atmosphere contribute to the forming of ozone (responsible for pollution peaks in summer).
- Residual solvents to be destroyed: they are eliminated by incineration (which produces $CO_2$) under the control of the DRIRE. The cost of elimination in Grasse is 100 euros per tonne (plus transport, plus pollutant activity tax).
- Plant residues: these are put in class 2 dumps (incineration, biological treatment, etc).

**3-3 Health:**

DRIRE and Industrial Healthcare regulations govern the exposure of workers to volatile organic solvents:

- Controlled solvent content in workshops.
- Safety measures to combat the risk of fire or explosion (fire-walls, ADF electrical equipment, automatic extinguishers, protected workshops, wastewater detention tanks in case of fire, alarms, etc.).

In the case of supercritical $CO_2$ extraction, risks are far from inexistent:

$CO_2$, even in a weak concentration, is poisonous. What's more, this deadly gas is odourless. Hence the need to equip premises where it is present with a device that analyses $CO_2$ in the air fitted with an alarm bell. The risks inherent in working with very high pressures must also be mastered.

Tanks that have been tried and tested (in France by the weights and measures body), vacuum rupture disks, valves with maximum settings, evacuation of liberated $CO_2$ to outside in case of incident, $CO_2$ pressure-building pump mechanically cannot exceed maximum authorised pressure, all tanks set at the same maximum pressure, etc.

## 4 **Conclusion**

Energy saving and environmental parameters must imperatively be built into any new development; in this domain, as in others, they are a pre-requisite of long-term survival.