

EQUIPMENT DESCRIPTION:

Supercritical Fluid Extraction plant was design by **NATEX** Prozesstechnologie GesmbH, Ternitz, Austria (<http://www.natex.at>) and supplied in 2011 under Fabr. No. 10-023.

The plant has a modular design for solid-liquid extraction with main components characteristics:

✓ **Extractor**

- ➔ gross volume about 3.5 liters, payload basket about 2 liters;
- ➔ design pressure 5.5 MPa and maximum temperature 120°C;
- ➔ parts in contact with medium in stainless steel;
- ➔ equipped with CO₂ pre-heater, integrated in the heating jacket and quick closure system.



✓ **Two separators**

- ➔ volume about 1.5 liters;
- ➔ design pressure 2.5 MPa (first separator) and 100 bar (the second separator);
- ➔ parts in contact with medium in stainless steel;
- ➔ heating jacket.



✓ **CO₂ working tank**

- ➔ volume about 8 liters;
- ➔ design pressure 100 bar;
- ➔ equipped with CO₂ condenser and integrated in the cooling jacket.

✓ **CO₂ circulation pump with diaphragm**

- ➔ mass flow 20 kg/h (at 500 bar);
- ➔ suction pressure 45 – 55 bar;
- ➔ discharge pressure 5.0 MPa.

The supercritical CO₂ plant is equipped with mass flow meter, cooler, evaporator, CO₂ recycling system equipped with cooling unit, pressure and temperature sensors and process software.

PROCESS PRINCIPLES

The supercritical CO₂ extraction is based on the principle that the CO₂ gas is compressed to a pressure and elevated to a temperature greater than that of its critical point (the critical pressure is $p_c = 7.3$ MPa and the critical temperature is $t_c = 31.18^\circ\text{C}$). Thereby, in fluid condition (above the critical pressure and the critical temperature) dissolve larger quantities of compounds than the partial pressure of the substance in the solvent normally permits.

The **ADVANTAGES** of supercritical fluid extraction and supercritical CO₂ are:

- for CO₂ the supercritical parameters are easy to controlled, maintained and environmentally friendly;
- CO₂ in supercritical state is non-toxic, non-inflammable, not explosive, chemically inert, germicidal, free of bacteria, low-cost fluid, readily available, Generally Recognized as Safe (GRAS) solvent and approved by Food and Drug Administration FDA for use in food, cosmetics and pharmaceutical industries;
- supercritical CO₂ has a good solvent characteristics for non-polar and slightly polar solutes and higher density (thus solubility);
- CO₂ is recycled within the plant, is physiologically harmless and does not cause problems for operators like some conventional solvents;
- is no need of any organic solvents for purification of the extract;
- the selective and thermo-sensitive compounds can be extracted;
- pure extracts with aroma unchanged and free of solvent are achieved;
- oxidation reaction are not possible once the pressure process excess in the equipment and prevents oxygen admission during extraction.

The supercritical fluid formation steps are presented in figure 1.

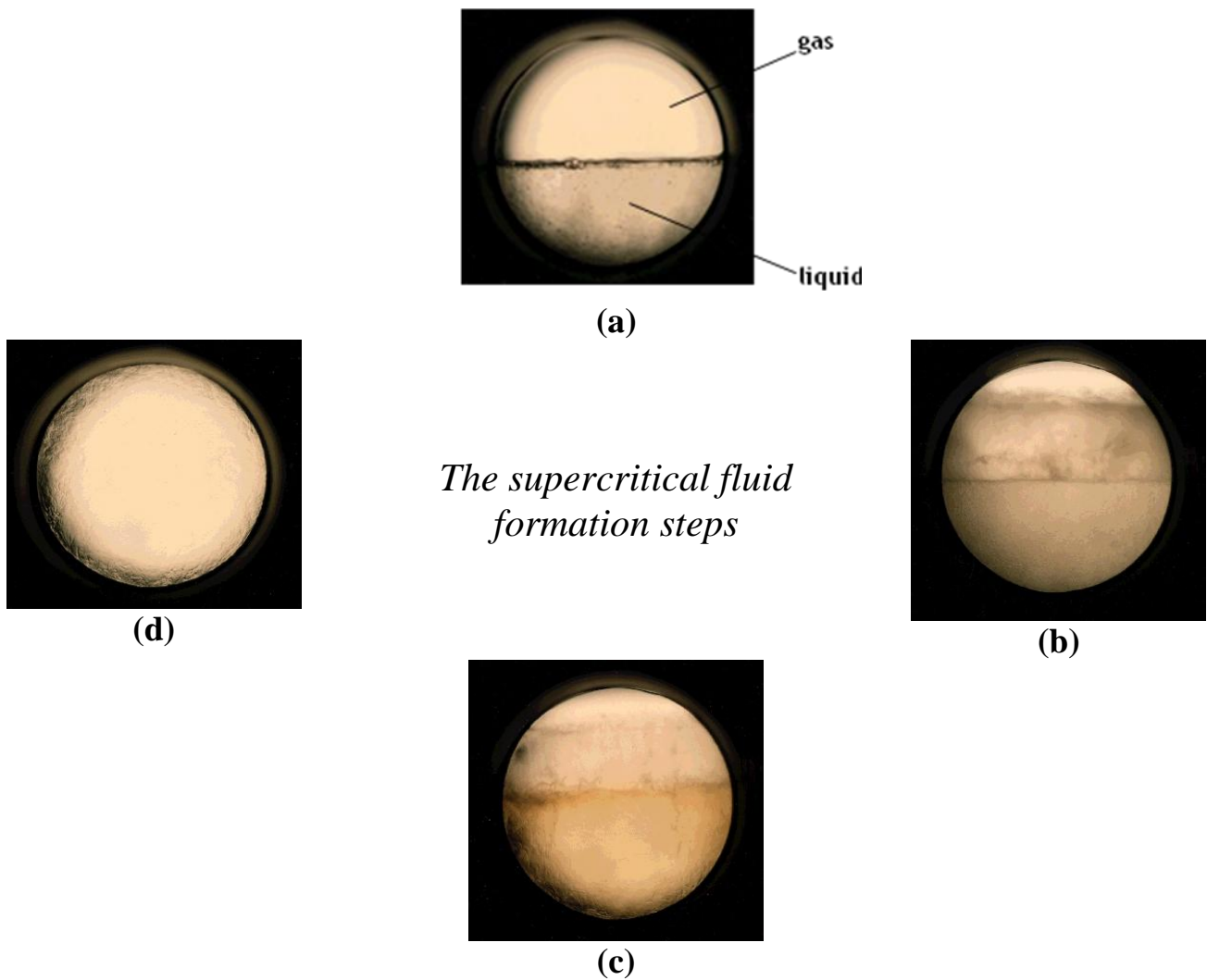


Fig. 1. Formation of supercritical fluid phase

(Source: <http://www1.chem.leeds.ac.uk/People/CMR/criticalpics.html>, accessed December 2015, 10th)

- (a) Can be observed the separate phases of carbon dioxide and the meniscus.
- (b) With the temperature increasing the meniscus begins to diminish and the system is in subcritical area.
- (c) Increasing the temperature further causes the gas and liquid densities to become more similar. The meniscus is less easily observed but still evident. The system is in subcritical area.

(d) Once the critical temperature and pressure have been reached the two distinct phases of liquid and gas are no longer visible. The meniscus can no longer be seen. One homogenous phase called the "supercritical fluid" phase occurs which shows properties of both liquids and gases.

DESCRIPTION OF THE SUPERCRITICAL FLUID EXTRACTION PROCESS

Liquid CO₂ from the storage is pressurized, heated and transferred to the extractor, where it gets loaded according to adjusted conditions. A change of these conditions (pressure, temperature) causes precipitation of the dissolved substances in the separation vessels. The now gaseous CO₂ is condensed, intermediately stored and recycled again (Source: <http://www.natex.at/processdescriptionextr.html>, accessed on March 2016, 31st).

The photo and scheme of supercritical CO₂ extraction plant from Faculty of Food Science and Engineering are presented in figure 2 and 3, respectively.



Fig. 2. Supercritical CO₂ extraction plant (photo)

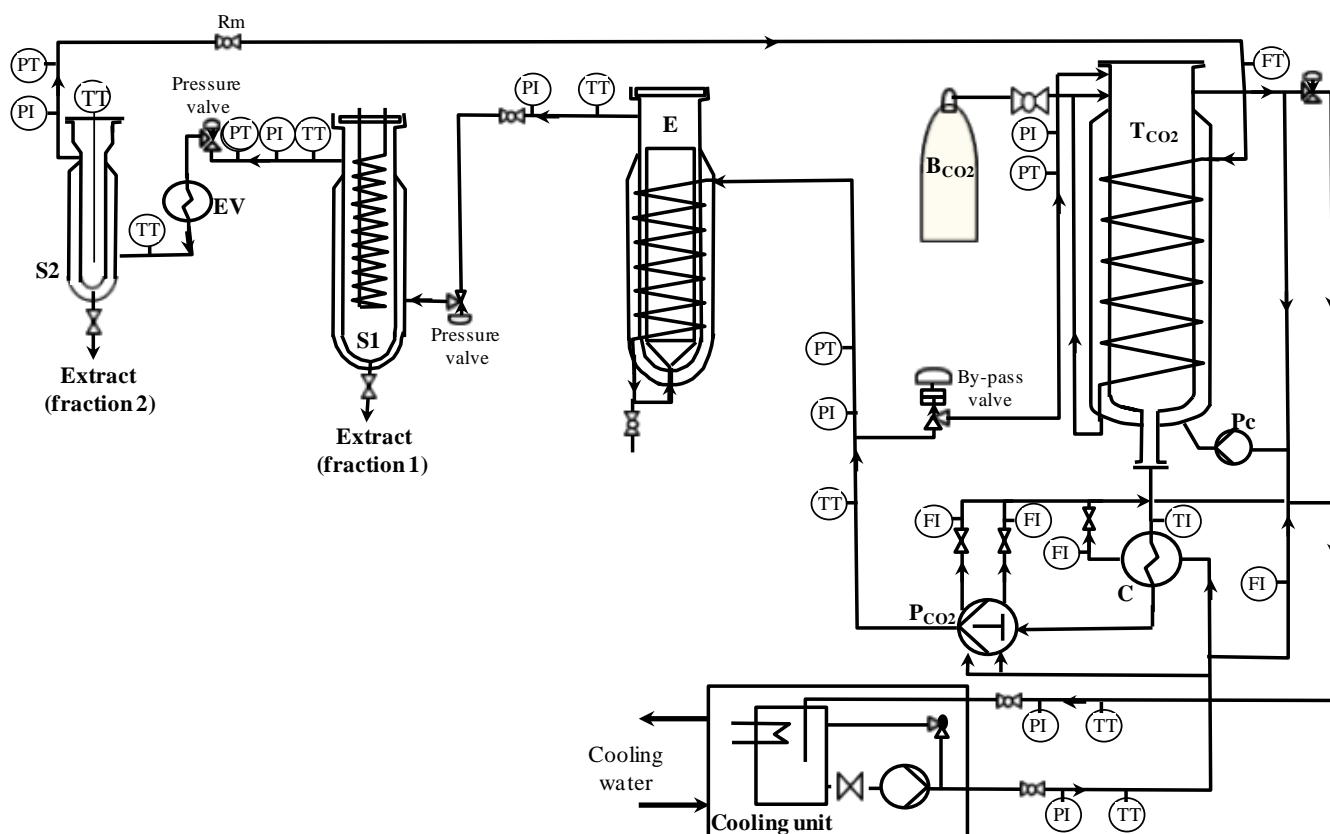


Fig. 3. Scheme of supercritical CO₂ extraction plant (Courtesy of NATEX)

FEW IMPLEMENTED PROJECTS:

- Postdoctoral projects (supercritical CO₂ extraction of *Allium* spp. bulbs and leaves; walnut green husk and leaves);
- PhD thesis (extraction of coriander and allspice seeds);
- Erasmus project (extraction of *Thymus vulgaris* L. leaves);
- Training in May 2011 (extraction of *Calendula officinalis* flowers);
- Private companies collaborations (SC BIOFARMNET SRL Tandarei, Ialomita, Romania – extraction of sea buckthorn).