



NATECO₂ GmbH & Co. KG

Extraktion von Naturstoffen mit überkritischem CO₂

Decaffeination of tea with pressures up to 1000 bars

Josef Schulmeyr

NATECO₂ GmbH & Co. KG
Auenstraße 18 –20
85283 Wolnzach, Germany
e-mail: Josef.Schulmeyr@nateco2.de

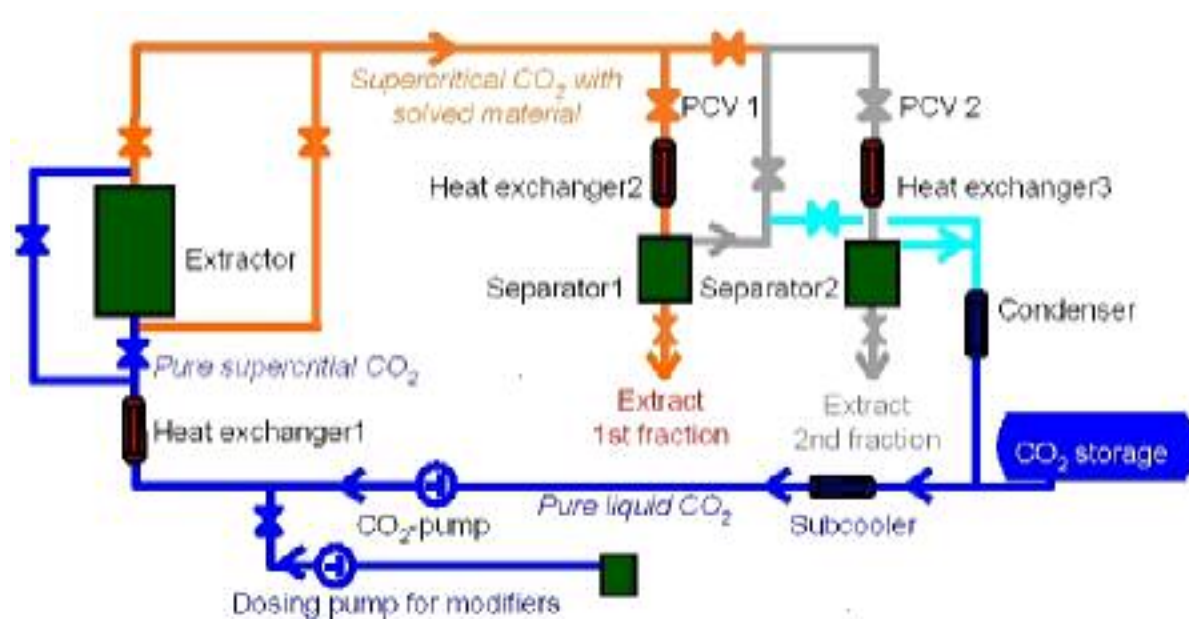
1 INTRODUCTION

Most of the commercial production plant works at moderate pressures lower than 300 bars. The design pressure is limited to approx. 500 bars because equipment costs augment disproportionally with raising pressure. From literature it is well known that with ascending pressure density as well as the dielectric constant of CO₂ are increasing thus the solubility for different components is rising. Through the change of interactions at higher pressures molecules, which are difficult to dissolve at lower pressures will be allowed to be extracted. This paper describes a test series for the decaffeination of tea in a pilot plant which was built in order to be able to examine new extraction areas, system-dependent requirements, special chemical engineering features and economic effects.

2 DESCRIPTION OF THE 1000 bars PILOT PLANT

The plant design is shown in Figure 1. The pump compresses the CO₂ to the desired pressure and the solvent is tempered to extraction conditions in the heat exchanger 1. The carbon-dioxide flows either from the bottom to the top of the extractor or vice versa. When operating with fractionating separation, the load gas is depressurized at the PCV 1 (pressure control valve) to the separation pressure of the first separation step. Temperature adjustment (heating or cooling) follows in heat exchanger 2. Due to the now lower solubility parts of the dissolved substances fall out and are collected in separator 1. At the PCV 2 the gas pressure is finally reduced to the CO₂ storage pressure and the carbon-dioxide is set to the required temperature of the second separation step by heat exchanger 3. The substances still soluble in separator 1 precipitate in separator 2. It is also possible to extract within an extraction cycle at different conditions (pressure and / or temperature) and collect the resulting extracts separately in the two separation vessels (fractionating extraction). The regenerated solvent is then liquified in a condenser, subcooled and recirculated to the CO₂ pump.

Figure 1: Flowsheet of the 1000 bar pilot plant



The plant is PLC-controlled allowing complete automated operation from pressurisation to depressurisation of the extractor. Furthermore all relevant measurements (pressure, temperature, ...) are recorded and logged. Therefore the results are well repeatable and the proceeding of one extraction can be traced afterwards well. The plant can be operated at the following conditions:

- Extraction pressure: 70 – 1000 bar
- Extraction temperature: 5 – 120 °C
- Separation pressure: 65 – 300 bar
- Separation temperature: 15 – 120 °C

The extraction volume of 50 litres allows extraction of approximately 20 kg raw material and therefore the preparation of relevant product samples. The sample is big enough to perform pilot tests for the preparation of new recipes. Furthermore calculations of production costs is possible. A dosing pump allows addition of modifiers between CO₂ pump and heat exchanger 1.



Figure 2: Filling 1000 bar extractor with basket

STATUS OF THE DECAFFEINATION

Infusions of coffee and tea are popular beverages because of their flavorful and stimulating characters. Consumers that would avoid the intake of caffeine according to health and other reasons can revert to decaffeinated products.

The decaffeination is performed by extracting with a solvent. In case of coffee usually the green beans are treated. Caffeine is reduced to the desired level. Only after this processing step the beans are roasted to develop flavor and aroma components. Sensory nearly no difference can be detected between regular and decaf coffee.

Opposite to the decaffeination of coffee the green or black tea ready for consumption is decaffeinated. Therefore the excellence of the extraction step is determining the quality of the final product. Especially long exposition of the tea leaf to wet and warm solvent degrades the quality considerably.

Today mainly supercritical carbon dioxide is used as solvent applying pressures between 200 and 300 bars and temperatures between 10 and 80 °C. At these extraction conditions the significant (anticancerogenic) polyphenols Epigallocatechine-gallat and Epicatechine-gallat are not soluble.

DECAFFEINATION OF TEA WITH PRESSURES UP TO 1000 bars

The intention of the test series was to make a comparison of the quality between teas produced with pressures of 500 and 1000 bars and by the commercial extraction.

Starting Materials

The test series were done with a Kenyan black tea and a Chinese green tea. The various tea species were moistened up to 26 weight-% water content before the extraction process.

Testing method

The different tea types were filled into the extractor's basket of the 1000 bar pilot plant. Carbon dioxide flowed through the extraction material from the bottom to the top. The removal of caffeine was done by pressure drop in a separator. To the CO₂ which was cycled water as entrainer was added before entering the extraction vessel to balance the solubility between separation and extraction.

Result and discussion

- **Kenyan black tea**

Starting caffeine content: 3.1 weight-%

Extraction pressure	300 bars	300 bars	800 bars
Extraction temperature	66 °C	66 °C	66 °C
Feed ratio (kg CO ₂ /kg tea)	260	105	105
Caffeine content after extraction	0.22 weight-%	0,58 weight-%	0,15 weight-%

- **Chinese green tea**

Starting caffeine content 2.9 weight-%

Extraction pressure	300 bars	300 bars	900 bars
Extraction temperature	66 °C	66 °C	66 °C
Feed ratio (kg CO ₂ /kg tea)	260	95	95
Caffeine content after extraction	0.15 weight-%	1.11 weight-%	0.04 weight-%

As could have been expected the extraction time could be reduced considerably. Surprisingly the aroma impact was improved additionally. Likely no aroma components have been removed.

The finding of the experiments have been:

- The extraction time could be shortened to approx. one half of the time needed for the commercial process.
- Sensory evaluation demonstrated higher quality of the infusion.
- The polyphenols Epigallocatechine-gallate and Epicatechine-gallate are not soluble in CO₂ up to pressure of 1000 bars

CONCLUSION

The decaffeination of tea with carbon-dioxide up to pressures of 1000 bars can be done without losses of quality in comparison to the commercial extraction. The next step must be to check exactly the production and investment costs of such an extraction process. Nevertheless the higher extraction pressure open a chance for the extraction of high grade and expensive specialities, like as food additives and raw materials for the production of Functional Food.

