EXTRACTION OF CAROTENOIDS WITH SUPERCRITICAL CO_2

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1 INTRODUCTION

"We live in a world of mixtures - the air we breathe, the food we eat, the gasoline in our automobiles ...:" [1]. Therefore raw materials as well as synthetic products have to be separated into desired and undesired components for further use. Since 1979 the gentle CO_2 extraction process is used at NATECO$_2$.
Most of the commercial CO_2-extraction-plants work at pressures lower than 300 bars. With ascending pressure the solubility for different substances is rising. Consequently also the extraction of carotenoids with CO_2 in an effective and economical way is possible through higher pressures.

1.1 THE CAROTENOIDS

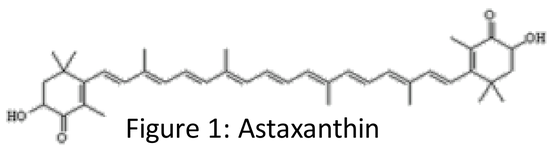

Figure 1: Astaxanthin

Carotenoids are organic pigments naturally appearing in chromoplasts of plants and some other photosynthetic organisms like algae and some bacteria. Structurally they are in the form of a polyene chain which is sometimes terminated by rings. Currently over 600 different Carotinoids are identified.
Carotenoids in plants absorb light energy for use in photosynthesis, and they protect chlorophyll from photodamage. In humans, carotenoids are a precursor to Vitamin A and they can act as antioxidants.
The best known member of the carotenoid-family ß-carotene is even exceeded by lycopene, lutein, astaxanthin (fig. 1) and zeaxanthin concerning the physiological functions. Thus carotenoids prevent some diseases like cancer, rheumatism, cataract or skin aging.

1.2 THE CO_2-EXTRACTION PLANT

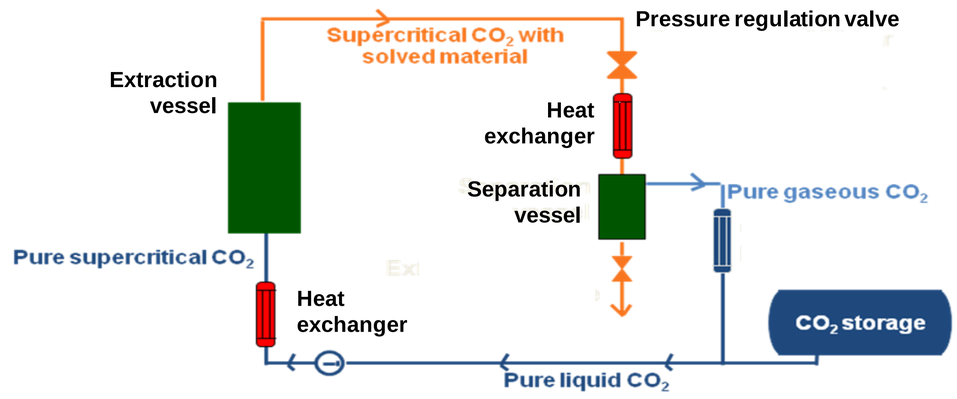

Figure 2: CO_2- extraction-plant

For the test series a CO_2-plant with a design pressure of 1000 bars and a volume of the extraction basket of 2 l was used. The flow sheet of the plant is presented in figure 2. To reach a high yield, extraction pressures were varied from 200 to 800 bars and temperatures from 50 to 80 °C.

2 RESULTS

To recover carotenoids, the preliminary CO_2-extraction tests have been carried out applying maize and saw palmetto berries.

2.1 MAIZE

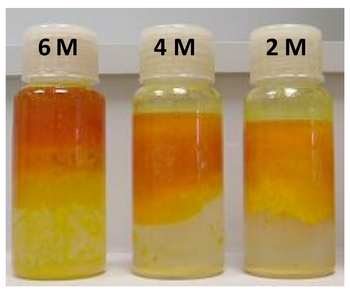

Figure 3: Maize-extracts

After grinding the maize, extractions have been performed in a pressure range between 200 to 600 bars and at temperatures between 50 to 80 °C .
The yield of extract related to the quantity of starting material varied from 4 to 7 % with rising process- parameters. The extracts are composed of approx. 60 % water and 40 % oily phase (fig. 3). Before preparing the orange oil for analysis with HPLC, the two phases have been separated by freezing out.
By increasing pressure and temperature the zeaxanthin-concentration in the extract was nearly doubled from 24 ppm at 300 bars and 60 °C to over 50 ppm at 600 bars and 80 °C (tab. 1).

Table 1: Parameters and analytical results of the extracts of maize

sample	extraction pressure [bar]	extraction temperature [°C]	lutein [ppm]	zeaxanthin [ppm]	ß- carotene [ppm]
starting material	-	-	8	4	n. d.
1 M	200	50	38	24	18
2 M	300	60	37	24	17
3 M	300	80	48	29	20
4 M	600	60	53	36	24
5 M	600	70	61	40	24
6 M	600	80	75	54	11

2.2 SAW PALMETTO BERRIES

Table 2: Parameters and analytical results of the extracts of saw palmetto berries

sample	extraction pressure [bar]	extraction temperature [°C]	lutein [ppm]	zeaxanthin [ppm]	ß- carotene [ppm]
starting material	-	-	2	1	97
1 S	200	50	n. d.	n. d.	583
2 S	300	60	7	2	510
3 S	600	60	14	5	611
4 S	800	80	14	6	627

The extraction parameters as well as the carotenoid concentrations for the minced saw palmetto berries are presented in table 2. The extraction-yield was between 14 and 17 % at 200 to 800 bars (fig. 4).
Nearly no influence concerning the ß-carotene concentration in the extract was achieved with rising pressure. The lutein and zeaxanthin content increased with higher pressures, whereas non of both substances were detectable in the 200 bar-extract.

Figure 4: Saw palmetto extracts

Moreover spent saw palmetto material out of NATECO$_2$'s production has been processed a second time in the lab-plant but at higher pressures (tab. 3). Applying these elevated parameters the extracts contain more than 1400 ppm ß-carotene. Also lutein and zeaxanthin have been concentrated. Pressures higher than 600 bars do not increase the carotenoid content substantially.

Table 3: Parameters and analytical results of the extracts of spent saw palmetto berries

sample	extraction pressure [bar]	extraction temperature [°C]	lutein [ppm]	zeaxanthin [ppm]	ß- carotene [ppm]
5 S	600	80	169	42	1434
6 S	800	80	132	37	1445

2.3 OTHER MATERIALS

In industrial-scale NATECO$_2$ processes dried red algae containing 2-4 % astaxanthin in the biomass to an extract with up to 14 % astaxanthin. Also the fabrication of lutein-extracts out of marigold achieves production-quantities. Extractions of lycopene from tomato skins, ß-carotene from dried carrots and carotenoids from fungi are already in different development stadiums at NATECO$_2$.

3 CONCLUSION

With higher pressures the extraction of carotenoids with supercritical CO_2 becomes more effective and economic. The resulting products are mostly applied as additives in functional food. Small-scale-productions has already been established successfully at NATECO$_2$. For this purpose a plant with an extraction volume of 50 l and pressures up to 1000 bars has been installed on the premises. Two separators allow the recovery of two fractions with different carotinoid contents in one extraction step. Also procurement for an additional plant with an extraction volume of 3 x 1 m³ is ongoing at the moment.

[1] Prausnitz, J.M. Molecular Thermodynamics of Fluid-Phase Equilibria. Prentice-Hall, Engelwood Cliffs, N.J. Library of Congress Catalog Card NO. 69-16866