EXTRACTION OF ESSENTIAL OILS IN THE SUPERHEATED STEAM FLUIDIZED BED

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Abstract: The fluidized bed extraction using superheated steam can be used for the production of volatile ingredients (essential oils) from plant materials. Depending on its properties the plant material must be pretreated. Some experimental investigations of this process and their results are presented. A simple model to describe and calculate the oil release from the plant material and the oil concentration in the steam is also represented.

Keywords: fluidized bed, superheated steam, essential oils, experiments, model

INTRODUCTION

The fluidized bed extraction using superheated steam can be used for the production of volatile ingredients (essential oils) from appropriate endemic plants. Depending on its properties the plant material must be pretreated (Mörl et al. 2005).

Some new experimental investigations of this process and their results are presented. A simple model to describe and calculate the oil release from the plant material and the oil concentration in the steam is also represented.

EXPERIMENTAL INVESTIGATIONS

The experimental investigations were performed by means of a fluidized bed pilot plant DN 200 with steam circulation (Behns, C. et al. 2008). The extraction is a batch process. The gas distributor bottom can be turned so that the whole bed mass can be removed immediately. A portion of the vapor phase is condensed. The condensate is a mixture of water and oil. An oil phase can be decanted. The watery phase also contains essential oils – Fig. 1.

The concentration of essential oils (total hydrocarbon concentration) can be measured in the vapor phase by means of a flame ionization detector (FID, company Eismann & Stöbe). The determination of the oil content and oil quality was performed by means of an MicroDistiller (Eppendorf) and by subsequent gaschromatography (Agilent).

Certain plant materials must be pretreated by pelletizing or milling to improve the fluidization properties or to open the oil cells in the plant tissues – Figures 2, 3, 4.

Fig. 1. Sight glass below the condenser with condensate and oil phase

Fig. 2. Example for oil containing plant material: camomile

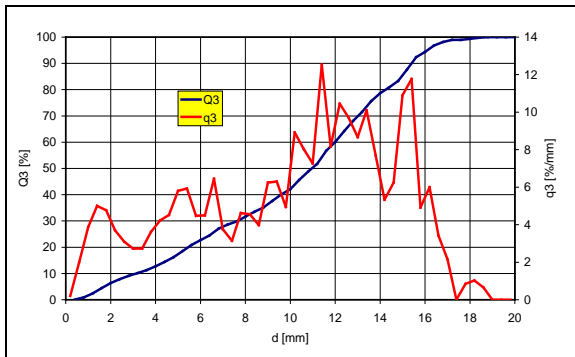Fig. 3. Pelletized camomile plant material (raw material for the fluidized bed extraction)

Fig. 4. Particle size distribution of raw camomile pellets (example KAM 10-01-18-2, xFemax)

RESULTS OF EXPERIMENTS

Fig. 5 shows the typical graph of the total hydrocarbon concentration (FID-signal) versus time. In this case - extraction of chamomile pellets - the concentration tends to zero in about 1500 s or 25 minutes. This is a very long period compared to other plant material (Mörl et al. 2005; Behns, C. et al. 2008) where the extraction is finished in less than 3 minutes.
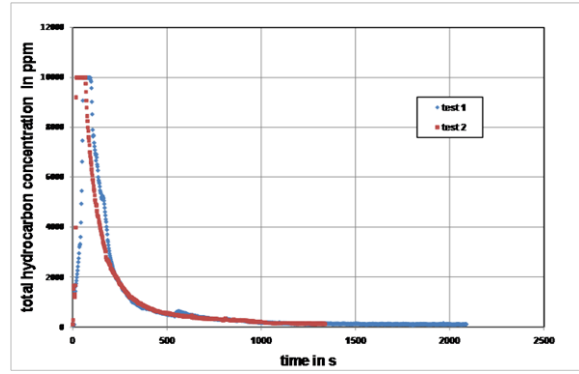

Fig. 5. Extraction of chamomile pellets – FID-signal versus time for two different tests

MODELING OF THE OIL RELEASE

The extracted oil mass per unit of time can be described by a simple approach

$$\dot{m}_E = a \cdot \exp(-b \cdot t) \tag{1}$$

with the substance specific parameters a and b. The cumulative released oil mass $m_E(t)$ can be determined by integration between the times $t = 0$ and $t = t$:

$$m_E(t) = \frac{a}{b} \cdot [1 - \exp(-b \cdot t)] \tag{2}$$

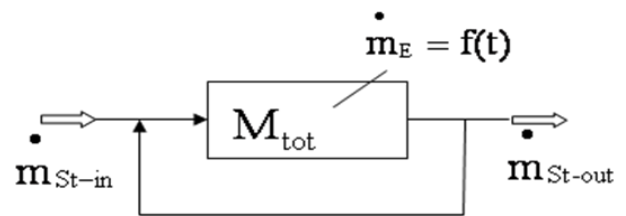## MODELING OF THE ESSENTIAL OIL CONCENTRATION ALTERATION IN THE STEAM LOOP

Fig. 7. Schematic of the fluidized bed and the steam loop

It can be written simplified

$$M_E = M_{tot} \bullet x_E \tag{3}$$

$$\frac{dM_E}{dt} = M_{tot} \cdot \frac{dx_E}{dt} \tag{4}$$

$$\frac{dM_E}{dt} = \dot{m}_E - \dot{m}_{E-out} \tag{5}$$

$$\dot{m}_{E-out} = \dot{m}_{st-out} \cdot x_E \tag{6}$$

Now the essential oil concentration alteration in the steam loop can be described using equation (1):

$$\frac{dx_E}{dt} = \frac{a \cdot \exp(-b \cdot t) - \dot{m}_{st-out} \cdot x_E}{M_{tot}} \qquad (7)$$

This differential equation can be solved with $x_E\,(t = 0) = 0$

$$x_E(t) = \frac{a}{(\dot{m}_{st-out} - M_{tot} \cdot b)} \cdot \left[\exp(-b \cdot t) - \exp\left(-\frac{\dot{m}_{st-out}}{M_{tot}} \cdot t\right) \right] \qquad (8)$$

$$m_{E-out}(t) = \frac{\dot{m}_{st-out} \cdot K}{(A-b)} \cdot \left[\frac{\exp(-A \cdot t)}{A} - \frac{\exp(-b \cdot t)}{b} + \frac{(a-b)}{A \cdot b} \right] \qquad (9)$$

Using this equations the influence of the motive steam flow rate on the essential oil concentration in the circulating steam can be determined. Fig. 8 shows an example.
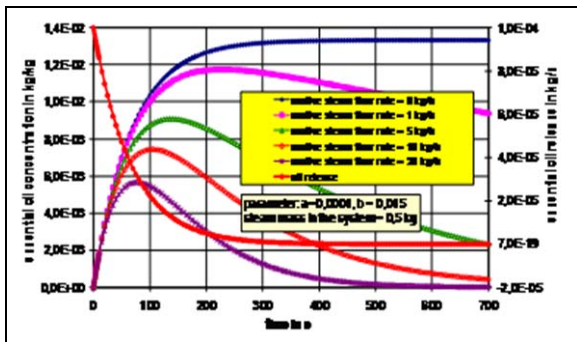

Fig. 8. Calculated essential oil concentration in the circulating steam vs. time for different motive steam flow rates - example

A comparison between the measured and the calculated essential oil concentration in the vapor phase can be seen in fig. 9.
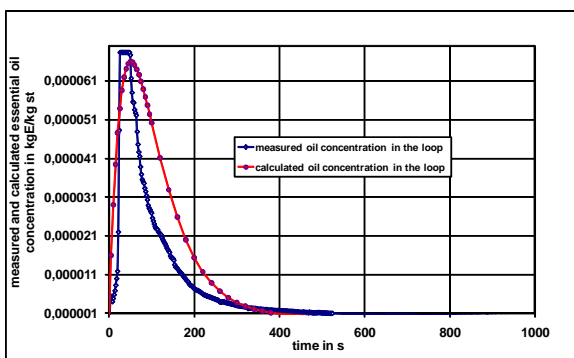

Fig. 9. Measured and the calculated essential oil concentration in the vapor phase

CONCLUSIONS

A high extraction efficiency is achieved. The extraction residue is dried, deoiled and free of solvents. A pretreatment of the plant material by compaction (pelleting) to improve the oil release and the fluidization behaviour is necessary when indicated. The description of the oil release and the oil concentration in the steam loop as function of time by means of an exponential function corresponds qualitatively closely with experimental data.

NOMENCLATURE

M	mass (plant material)	kg
$\dot{m}$	mass flow rate	kg/s
x	concentration in the vapor phase	kg/l
t	running time	s

Subscripts

st	steam
in	input
out	output
E	essential oil
tot	total

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Mörl, L. et al. (2006): Fluidized bed extraction of essential oils using superheated steam, Renewable resources and plant biotechnology, New York, NY, :Nova Science Publishers, pp. 69-76