**Dynamics and process parameters evaluation of citric essential oils vapor distillation extraction**

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**Highlights**

* The study was carried out at a pilot plant level.
* The extraction dynamics differed significantly from one citrus to another
* Steam pressure/packing factor of the bed, determined the extraction dynamics

**1. Introduction**

This work evaluates at a pilot plant level, the dynamics of the process of extraction by steam distillation of essential oils in citrus (*citrus sinensis*, *citrus limetta*, *citrus latifolia* and *citrus reticulata*) from different Peruvian regions. The processing conditions have been determined in the extraction and phase separation units, ensuring efficiency while maintaining a high oil quality.

The steam distillation method of extraction has been selected, because it is the only one that is normalized and that is widely used in industry 1. New technologies for extracting essential oils are being tested, but they do not necessarily provide better results than the steam distillation method2,3, if oil quality and process yield are main guidelines.

The dynamics of extraction, may be represented by the model developed by Ho & Oumarou 4:

$ \frac{dCt}{dt}=k(Cs-Ct)^{2}$

Where:

𝑘 second order constant of velocity of extraction (𝐿𝑔−1 − 𝑚𝑖𝑛−1 );

𝐶𝑠 concentration of oil in saturation (𝑔𝐿−1 );

𝐶𝑡 concentration of oil in the solution at any time instant (𝑔𝐿−1 ) t (min)

**2. Methods**

The steam distillation process of extraction took place in a 15 L pilot plant provided with pressure and temperature sensors. Oil production was monitored with a precision of 0.2mL. The following variables have been manipulated: the packing factor of the citrus peel in the extractor, the inlet pressure of the steam and its flow, as well as the extraction time. Also, in order to understand the mass transfer mechanism of the essential oil located in the citrus peels to the vapor phase, microscopic analysis have been performed to the skin of the citrus materials.

**3. Results and discussion**

The process of extraction presents a dead time (θ) that depends on the citrus species, vapor pressure (*u*) and packing factor (φ). We have formulated a model including these variables, based on the approach of transfer functions for process control. The model we obtained is characterized by the equation $y\left(t\right)=$ {1-exp $[-⁡(t-θ)/τ$]} K Δu(t).

Both the gain *K* and the time constant *τ* depend fundamentally on the type of citrus material. Our results showed a substantial difference (up to 80%) in the dynamics of orange with respect to other citrus species. The optimal time for extraction for all four citrus materials is very much alike, in the range of 10 to 15 minutes, where 80% of the essential oil has been already extracted.

The relation between vapor pressure and packing factor as a function of oil yield is exponential for the orange, hence an optimal point for maximal efficiency can be obtained. However, for the other citrus species it has a linear inverse relation, therefore the best results are obtained with a minimal relation between steam inlet pressure versus packing factor.

In the oil extraction process the dead time could indicate the time required to meet the conditions for the oil to flow out of the glandular trichomes where it is contained, in order to be dragged by the vapor flowing across de bed. These conditions refer to the pressure for breaking the walls of the trichomes, and the temperature to vaporize the essential oil. The measured dead time is in the range of 1.5 to 3 minutes.

Along the extraction, a convective process takes place since there is a continuous vapor flow through the extraction bed; and a diffusive process as well, with the oil flowing across the solid phase due to a concentration gradient. Therefore the gain *K* might represent a mixed transfer coefficient that accounts for both, the convective and diffusive phenomena pointed out, that will be helpful in determining a global efficiency of the extraction process. On the other hand, the time constant is referred to the velocity of the extraction, and indicates the time needed to extract most of the oil. This parameter allows estimation of extraction process ending.

**4. Conclusions**

The dynamics of the extraction of essential oils from citrus is best explained considering a first-order model with dead time, process gain and time constant. These parameters account for bed resistance, convective/diffusive coefficient, and extraction velocity, respectively. A critical zone was achieved where maximum efficiency is obtained, which lies in the range of 10 to 15 minutes of extraction time. The relation between steam inlet pressure and packing factor is a good indicator of the extraction process dynamics.

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