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**THE EFFECTIVE DIFFUSIVITY OF CLOVE (*Eugenia caryophyllus*) ESSENTIAL OIL IN PRESSURIZED CO<sub>2</sub><sup>1</sup>**

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## SUMMARY

The effective diffusivity of clove essential oil in subcritical liquid CO<sub>2</sub> was estimated. The experimental apparatus employed was a fixed-bed extractor. The fixed bed was formed with grounded (mesh  $-32 \pm 65$ ) and compacted clove buds which were considered a solid element. The effective diffusion coefficient was evaluated by fitting the experimental concentration profile to the unsteady state mass balance equation for unidirectional diffusion in a finite solid medium. The diffusion coefficient was related to the concentration of oil in the solid by an exponential function. The estimated values of the effective diffusion coefficient varied from 3.64 to  $5.22 \times 10^{-10}$  m<sup>2</sup>/s. The average relative errors were lower than 3.1%.

**Key words:** Diffusion coefficient, mass transfer, finite difference, clove essential oil, supercritical extraction.

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## RESUMO

ESTIMATIVA DA DIFUSIVIDADE MÁSSICA EFETIVA DO ÓLEO ESSENCIAL DE CRAVO-DA-ÍNDIA (*Eugenia caryophyllus*), EM CO<sub>2</sub> PRESSURIZADO. O coeficiente de difusão efetivo do óleo essencial de cravo-da-índia em CO<sub>2</sub> líquido subcrítico foi estimado. O equipamento experimental usado foi um extrator de leito fixo. O leito fixo foi formado por cravo-da-índia moído (mesh  $-32 \pm 65$ ) e compactado, sendo assim considerado como um meio sólido. O coeficiente de difusão efetivo foi estimado pelo ajuste do perfil de concentração experimental à equação de balanço de massa em estado transiente, para difusão unidirecional em um meio sólido. A dependência do coeficiente de difusão com a concentração de óleo no sólido, foi descrita por uma função exponencial. O

coeficiente de difusão determinado variou de 3,64 a  $5,22 \times 10^{-10} \text{m}^2/\text{s}$ . Os erros médios relativos foram menores do que 3,1%.

**Palavras chave:** Difusividade mássica, transferência de massa, diferenças finitas, óleo essencial de cravo-da-índia, extração supercrítica.

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## 1 - INTRODUCTION

Among the studies on estimation of effective diffusion coefficient of natural product essential oils using pressurized CO<sub>2</sub>, is one by Goto *et al* [5]. These authors proposed a mathematical model to evaluate extraction rate considering local equilibrium adsorption of mint essential oil by lipids and mass transfer. The intraparticle diffusion coefficient of  $\alpha$ -menthol, the main constituent of mint oil, in supercritical solvent varied from  $0.17 \times 10^{-8}$  to  $1.46 \times 10^{-8} \text{m}^2/\text{s}$ .

Reverchon *et al* [9] measured the mass diffusivity of basil, marjoram and rosemary essential oils in CO<sub>2</sub> for particles of different sizes at 100 bar and 313 K. They found that the effective diffusion coefficient varied from  $1.4 \times 10^{-13}$  to  $2.8 \times 10^{-13} \text{m}^2/\text{s}$ . These authors indicated that internal resistance in herbaceous matters controls the mass transfer process.

Roy *et al* [10] used experimental data obtained for the extraction of ginger essential oil at different CO<sub>2</sub> flow rates to estimate the effective diffusion coefficient. They found that the value of effective diffusivity was  $2.5 \times 10^{-10} \text{m}^2/\text{s}$ .

Nevertheless, the effect of bed compactness, has not been studied to date, with the exception of the work by Del Valle and Aguilera [3]. For mushroom powder, bed compaction prior to extraction with supercritical fluids resulted in a solubilization rate apparently controlled by solid matrix solute diffusion; therefore, extraction rate could be predicted by solid diffusion models. These authors evaluated mushroom oleoresin extraction for CO<sub>2</sub> densities from 538.3 to 678.7 kg/m<sup>3</sup> and temperatures from 336 to 363.3 K. They determined that oleoresin fractions located in pores were easily extracted and the diffusion coefficient varied from  $1.0 \times 10^{-8}$  to  $1.5 \times 10^{-8} \text{m}^2/\text{s}$ . Remaining oleoresin located inside the solid matrix was extracted at a rate controlled by a diffusivity 100 to 150 times lower.

The diffusion of solutes in biological system during extraction is a phenomenon which is difficult to explain because it is influenced by the interaction among components and structure. Thus, it is possible that the diffusion coefficient depends on concentration.

The present work objective was to estimate the effective mass diffusion coefficient of clove essential oil in CO<sub>2</sub> for supercritical extraction in a fixed-bed extractor. Operational conditions employed were pressures of 64.7 and 69.7 bar and temperatures of 10 and 12°C. The effective mass diffusion coefficient dependence of essential oil concentration in solid phase was also investigated.

## 2 - MATERIALS AND METHODS

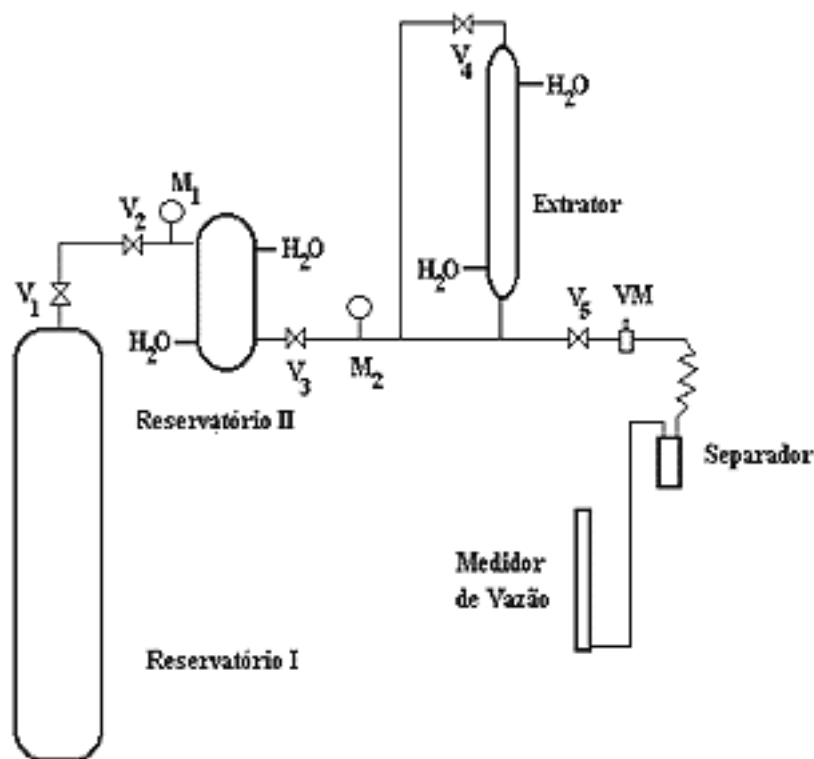
Clove buds were bought from a local store (Campinas, São Paulo, Brazil). Material was cleaned manually with the use of sieves with meshes of 6 and 8 (Granutest Tyler series, Brazil) to remove branches, leaves, stones, powder and other foreign materials. Cleaned and selected clove buds were placed in plastic containers which were hermetically closed and stored in a domestic freezer (Metalfrío, 620 L, horizontal, Brazil).

The amount of material required for each experimental run was ground in a laboratory helix-type mill (Marconi, Model MA-345, Brazil) with water circulation. The solid was ground in a frozen state to avoid thermal degradation of the volatile oil. Solid particles were sifted in a sieve shaker for 25 min. to obtain particles with meshes of -32 and +65. Solid material was manually compacted with the aid of a glass rod (1 cm of diameter) into a surgical tubular cloth (T. M. Orthopaedic, 100% cotton) which was placed into a hollow cylindrical matrix made of disposable hypodermic syringes. Solids with a cylindrical shape of approximately 2.17 cm diameter and 6.8 cm length and a uniform density were obtained. Each experiment required 7 cylinders that were weighed in an analytic balance (Sartorius, Model A 200 S,  $\pm 0.001$  g, USA) and placed inside of an extractor (with length of 60.5 cm and inside diameter of 2.16 cm).

### 2.1 – Experimental procedure

A flow sheet of the experimental apparatus is shown in [Figure 1](#). Valves V<sub>1</sub> and V<sub>2</sub> were always open to guarantee that reservoir-II was always full of CO<sub>2</sub> (Liquid Carbonic do Brazil, 99% pure). System pressure was controlled with the aid of a heating tape (Fisaton, Model -5.5 cm width, Brazil) wrapped around reservoir-I and linked to an automatic temperature controller

(Dyna-sense, Model 2156-00, USA). One hour before connecting the extractor to the extraction line, the recirculating water bath (Tecnal, Model TE-184,  $\pm 1$ K, Brazil) for reservoir-II was turned on. Ninety minutes later, having reached preselected operational conditions, valve V<sub>3</sub> was opened and CO<sub>2</sub> began to flow through the bed. Up to this point valve V<sub>4</sub> was kept closed. Once extractor pressure was equilibrated, its upper end was isolated by closing valve V<sub>4</sub>. Thereafter, valve V<sub>5</sub> and the micrometering valve used to expand and to control the CO<sub>2</sub> flow rate were opened. These valves were heated with a heating tape (Fisaton, Model -5, of 2.5 cm width, Brazil) linked to an electronic temperature controller (Dyna-Sense, Model 2156-00, USA). These valves (V<sub>5</sub> and micrometering) were opened in such a way so as to maintain constant and at a minimum the CO<sub>2</sub> flow rate. This care was taken to avoid the possibility of oil accumulation in the fluid phase. Oil was collected in 20 cm<sup>3</sup> glass flasks. CO<sub>2</sub> flow was maintained for seven hours. After this valve V<sub>3</sub> was closed and CO<sub>2</sub> was completely removed from the extracting line.



**FIGURE 1.** Flowsheet of the Experimental Unit.

After complete depressurization (0 bar of pressure), cylinders formed with compacted solid were removed from the extractor and placed in a *dessecator* until they reached room temperature; afterwards, they were weighed and the solid phase concentration profile was calculated.

## 2.2 – Composition of the extract

The composition of clove oil was determined by gas chromatography using the procedure described by Rodrigues *et al* [11].

## 2.3 – Mass transfer in a finite solid with a concentration dependent mass diffusivity

To study oil extraction kinetics using CO<sub>2</sub> as solvent in a fixed bed formed with compacted clove particles, compacted substratum was considered as a solid, in agreement with Del Valle and Aguilera [3]. For the experimental system employed (see [Figure 2](#)), diffusion occurred only in the axial direction. Therefore, mass balance for the solid phase is given by the unidirectional, nonstationary mass transfer equation, or Ficks Second Law, and can be written as [12]:

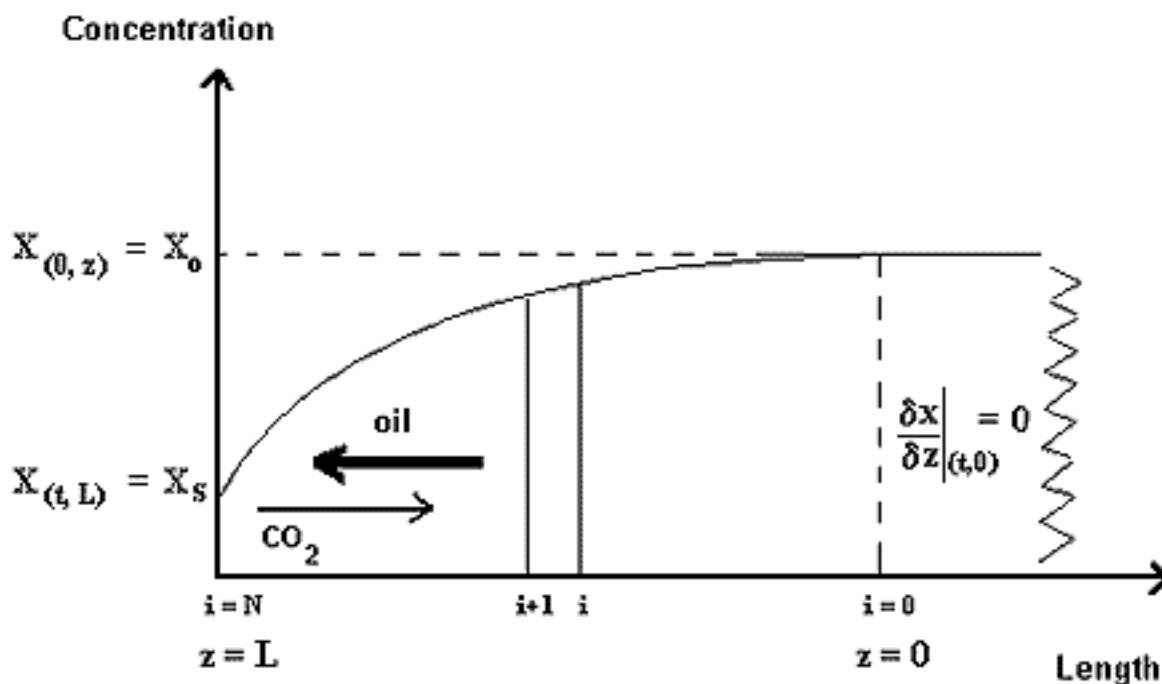


FIGURE 2. Experimental conditions.

$$\frac{\partial X}{\partial t} = \frac{\partial}{\partial z} \left[ D(X) \frac{\partial X}{\partial z} \right] \quad (1)$$

Where the diffusion coefficient depends on oil concentration in the solid:

$$D(X) = f(X) \quad (2)$$

The initial and boundary conditions are given by:

C.I.: Initially the oleoresin content was evenly distributed within the solid:

$$X(0, z) = X_o \quad (3)$$

C.C.: During extraction, the solid surface concentration is:

$$X(t, L) = X_s \quad (4)$$

C.C.: The finite solid condition is:

$$\left. \frac{\partial X}{\partial z} \right|_{(t,0)} = 0 \quad (5)$$

The following dimensionless groups will be defined:

$$\psi = \frac{X - Y_1 / k(\bar{X})}{X_o - Y_1 / k(\bar{X})} \quad (6)$$

$$F_o^+ = \frac{D_o^+ t}{L^2} \quad (7)$$

$$Z = \frac{z}{L} \quad (8)$$

where  $D^+$  is the value of the variable diffusivity that corresponds to the initial concentration [2, 6].

For the concentration-dependent diffusion coefficient for the solution of the mass transfer equation the following dimensionless variable may be used [1, 6]:

$$D = \frac{D(X)}{D^+} \quad (9)$$

Expressing Equation (1) in terms of dimensionless groups, we have:

$$\frac{\partial \psi}{\partial F_o^+} = \frac{\partial}{\partial Z} \left[ D \frac{\partial \psi}{\partial Z} \right] \quad (10)$$

Introducing the transformation variable defined as [2]:

$$S = \int_0^{\psi} D \, d\psi \quad (11)$$

Substituting Equation (11) into Equation (10) results in:

$$\frac{\partial S}{\partial F_o^+} = D \frac{\partial^2 S}{\partial Z^2} \quad (12)$$

To solve Equation (12) we have assumed that the solute concentration dependence of the diffusion coefficient follows an exponential function as given by the relationship [6]:

$$D(X) = D_o \exp(k_1 X) \quad (13)$$



Where  $D_0$  is the diffusion coefficient when  $X = X_0$  and  $D^+$  is the variable diffusion coefficient in  $X = X_0$ , defined by [6]:

$$D^+ = D_0 \exp(k_1 X_0) \quad (14)$$

Therefore, variable  $D$  of Equation (9) is defined as:

$$D = \exp k_2 - k_2 S \quad (15)$$

Thus:

$$k_2 = k_1 \left[ \frac{Y_1}{k(\bar{X})} - X_0 \right] \quad (16)$$

Initial and boundary conditions for Equations (3), (4) and (5) of this model are expressed, in terms of variable  $S$  as:

C.I.:

$$S_{(0,2)} = -\frac{1}{k_2} + \frac{1}{k_2} \exp k_2 \quad (17)$$

C.C.(1):

$$S_{(F_0^+,1)} = \frac{1}{k_2} \exp k_2 - \frac{1}{k_2} \left[ \exp \left( k_2 \left( 1 - W_{(F_0^+,1)} \right) \right) \right] \quad (18)$$

C.C.(2):

$$\frac{\partial S}{\partial Z} \Big|_{(F_0^+,0)} = 0 \quad (19)$$

Equation (12), with Equations (17), (18) and (19) as initial and boundary conditions was solved using the explicit finite differences method. Diffusion equation discretization can be accomplished by the following approach for its first and second order derivatives [2]:

$$\frac{\partial S}{\partial F_o^+} = \frac{S_i^{t+1} - S_i^t}{\Delta F_o^+} \quad (20)$$

$$\frac{\partial^2 S}{\partial Z^2} = \frac{S_{i+1}^t - 2S_i^t + S_{i-1}^t}{(\Delta Z)^2} \quad (21)$$

Combining Equations (20) and (21) with Equation (12) we have:

$$S_i^{t+1} = S_i^t + D \frac{\Delta F_o^+}{(\Delta Z)^2} [S_{i+1}^t - 2S_i^t + S_{i-1}^t] \quad (22)$$

For  $i = 1, 2, \dots, N - 1$ , and

$$D = \exp k_2 - k_2 S_i^t \quad (23)$$

The initial and boundary conditions expressed as finite differences for the exponential model are given by:

C.I.:

$$S_i^t = -\frac{1}{k_2} + \frac{1}{k_2} \exp k_2 \quad (24)$$

C.C. (1):

$$S_N^{t+1} = \frac{1}{k_2} \exp k_2 - \frac{1}{k_2} \left[ \exp \left( k_2 \left( 1 - \psi_{(F_o^+, 1)} \right) \right) \right] \quad (25)$$

C.C. (2):

$$S_N^{t+1} = \frac{1}{k_2} \exp k_2 - \frac{1}{k_2} \left[ \exp \left( k_2 \left( 1 - \psi_{(F_o^+, 1)} \right) \right) \right] \quad (26)$$

As a result of discretization, the system of simultaneous equations generated was solved by means of matrix direct multiplication outline. Once  $S_i$  values were obtained, concentration values in each position could be calculated by:

$$\psi_i = 1 - \frac{1}{k_2} \ln \left[ -k_2 \left( S_i - \frac{1}{k_2} \exp k_2 \right) \right] \quad (27)$$

Average concentration in each segment was determined by:

$$\psi = \frac{\sum_{i=1}^n \psi_i \Delta Z}{Z} \quad (28)$$

For estimation of  $X_s$ ,  $k_1$  and  $D_o$  parameters, the modified simplex method of Nelder and Mead [7] was used. Quality of model fitting was evaluated through average relative errors given by:

$$\% e_r = \frac{\sum_1^{N_p} \left[ \frac{|\psi_{pred} - \psi_{exp}|}{\psi_{exp}} \right]}{N_p} \quad (29)$$

where  $N_p$  is number of experimental points, and predicted and experimental concentrations are expressed dimensionless.

### 3 - RESULTS AND DISCUSSION

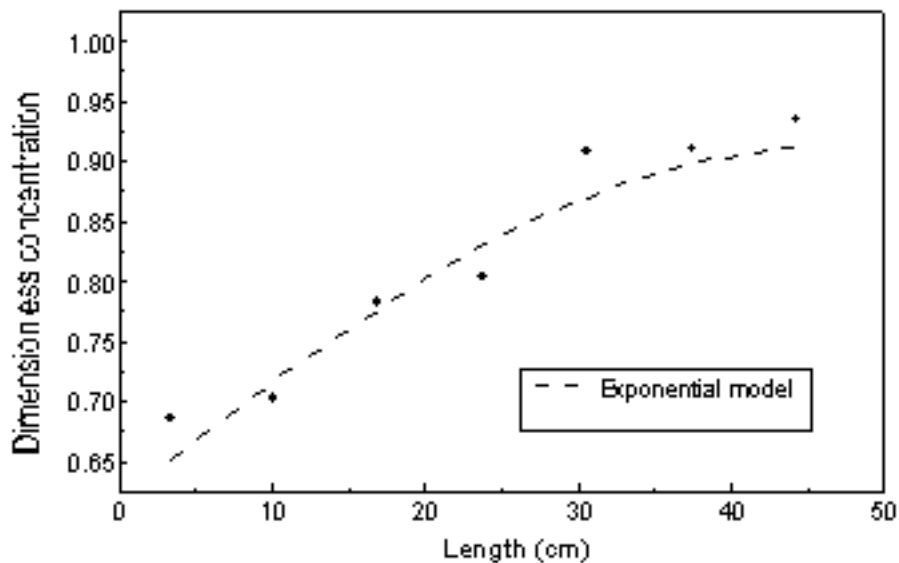
Initial solute concentration in solid phase,  $X_0$ , was taken to be oleoresin content as determined by Rosengartens hexane extraction method as described by Germer [4]. This value was  $25.0 \pm 0.3\%$  and is in agreement with results shown in the literature [4]. The composition of the clove oil is given in [Table 1](#).

**TABLE 1.** Composition of clove essential oil (mass %).

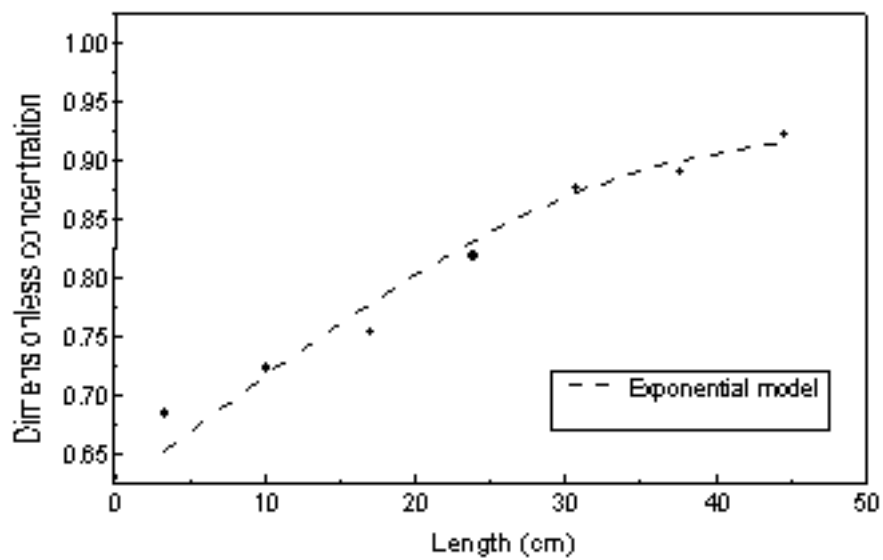
64.7 bar					
T °C	Eugenol	$\beta$ -caryophyllene	$\alpha$ -humulene	Eugenol acetate	Unidentified
10	50.14	19.77	2.10	27.60	0.39
12	58.62	19.81	1.60	19.50	0.47
69.7 bar					
10	48.41	19.86	2.49	28.19	1.05
12	49.34	19.91	2.23	27.64	0.88

For the solution of equations using the explicit method, 5 second increments, with 60 nodal points were used. Intervals for Fourier number were on the order of magnitude of  $10^{-5}$ . This size was chosen to avoid numerical oscillations that make the method unstable [8]. In this work the

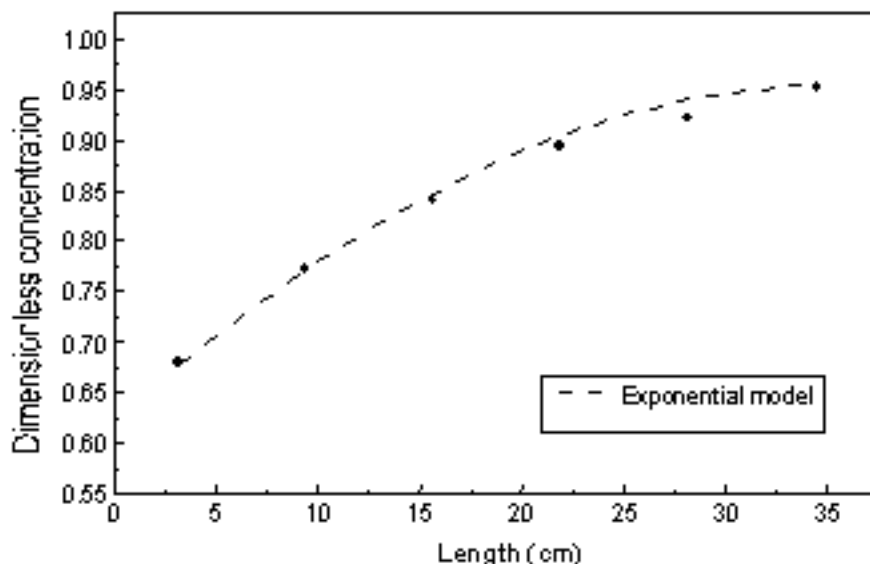
stability criterion was:  where D is given by Equation (23).



**FIGURE 3.** Experimental and calculated concentration profile at 69.7 bar and 10°C.



**FIGURE 4.** Experimental and calculated concentration profile at 64.7 bar and 12°C



**FIGURE 5.** Experimental and calculated concentration profile at 69.7 bar and 12°C.

[Table 2](#) shows estimated values of parameters  $k_1$ ,  $X_s$  and  $D_o$ . Mean relative deviations are smaller than 3.1%. [Figures 3, 4, 5](#) and 6 show the concentration profile for the pressure and temperature conditions studied. There is a good agreement between predicted curves and experimental data. At 10°C the effective diffusion coefficient did not change when pressure increased 5 bar. At 12°C, as pressure increased from 64.7 bar to 69.7 bar, the effective diffusion coefficient decreased as much 27%. At a constant pressure the following behavior was observed as temperature increased from 10 to 12°C: at 64.7 bar the effective diffusion coefficient was  $4.97 \times 10^{-10} \text{ m}^2/\text{s}$  and it was  $3.64 \times 10^{-10} \text{ m}^2/\text{s}$  at 69.7.

Several phenomena acted upon the system. An explanation of observed behavior can only be achieved if all combined effects are considered. For instance, the effective diffusion coefficient is associated with solute interaction in the solid phase, as well as in the fluid phase. Therefore, system thermodynamic limitations can, to a certain extent, interfere with effective diffusion coefficient determination. But this phenomena was not explicitly considered in the model. The possibility also exists that the operational conditions employed are near the crossover region, since results shown in the literature detected a minimum in the solubility at 16°C for pressures of 60.5 to 70 bar [11]. Another phenomena that should be considered is the modification of extract composition (see [Table 1](#)) observed at these pressures and temperatures. Composition was strongly affected by temperature at a pressure of 64.7 bar, but this was not observed at 69.7 bar. Therefore, eugenol conversion to eugenol acetate may be partially responsible for the observed behavior of the effective diffusion coefficient. An understanding of the role of the interaction of each solute with the solid matrix, as well as with the solvent, would probably help in explaining

the behavior of the effective diffusion coefficient calculated for the mixture.

**TABLE 2.** Estimated parameters for the exponential model

64.7 bar				69.7 bar				
Temp. (°C)	$k_1 \times 10^6$ g. m.l.o/g. oil	$X_s$ g. oil/g. m.l.o*	$D_o \times 10^{10}$ (m <sup>2</sup> /s)	% e <sub>r</sub>	$k_1 \times 10^6$ g. m.l.o/g. oil	$X_s$ g. oil/g. m.l.o	$D_o \times 10^{10}$ (m <sup>2</sup> /s)	% e <sub>r</sub>
10	1.93	0.155	4.97	3.05	1.93	0.155	4.97	2.74
12	1.93	0.155	4.97	1.83	1.32	0.155	3.64	0.81

\* m.l.o = oil free base

## 4 - CONCLUSIONS

The clove oil diffusion coefficient for compacted bed was predicted using the mass transfer equation for a finite solid.

An exponential relationship was used to describe diffusion coefficient dependence on oil concentration.

For this model, mean relative deviations between experimental and predicted data were smaller than 3.1%.

The effective diffusion coefficient and extract composition varied with both temperature and pressure, but further studies are required to explain the observed behavior.

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## 6 - NOMENCLATURE

D	dimensionless diffusivity
D(x)	variable diffusion coefficient (m <sup>2</sup> /s)
D <sub>ef</sub>	effective diffusion coefficient (m <sup>2</sup> /s)
D <sub>o</sub>	effective diffusion coefficient when X = X <sub>o</sub> (m <sup>2</sup> /s)
D <sub>+</sub>	variable diffusion coefficient in X = X <sub>o</sub> (m <sup>2</sup> /s)
F <sub>o</sub> <sup>+</sup>	Fourier number defined by D <sub>+</sub>
k <sub>1</sub>	constant defined by equation (13)
k <sub>2</sub>	constant defined by equation (16)
k( $\bar{X}$ )	equilibrium distribution coefficient
L	length
N	nodal points
X	concentration of oleoresins in the solid phase (kg (oil)/kg (oil free base) )
X <sub>o</sub>	initial concentration in clove
X <sub>s</sub>	concentration of oleoresins on the surface of solid
Y	concentration of oleoresins in the fluid (kg (oil)/kg (CO <sub>2</sub> ))
S	variable of transformation defined by equation (11)
t	time (s)
Z	dimensionless coordinate

$z$	axial coordinate (m)
$\psi$	dimensionless concentration

## 7 - ACKNOWLEDGMENT

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