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# SUPERCRITICAL EXTRACTION OF PUPUNHA (*Guilielma speciosa*) OIL IN A FIXED BED USING CARBON DIOXIDE

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**Abstract** - The pupunha (*Guilielma speciosa*) is the fruit of a palm tree typical of the Brazilian Northern region, whose stem is used as a source of heart of palm. The fruit, which is about 65% pulp, is a source of oil and carotenes. In the present work, an analysis of the kinetics of supercritical extraction of oil from the pupunha pulp is presented. Carbon dioxide was used as solvent. The extractions were carried out at 25 MPa and 323 K and 30 MPa and 318 K. The chemical composition of the extracts in terms of fatty acids was determined by gas chromatography. The amount of oleic acid, a saturated fatty acid, in the CO<sub>2</sub> extracts was larger than that in the extract obtained with hexane. The overall extraction curves were modeled using the single-parameter model proposed in the literature to describe the desorption of toluene from activated coal. *Keywords*: Supercritical extraction, *Guilielma speciosa*, pupunha, lipids, carotenes, extraction, extraction curve modeling.

**INTRODUCTION** 

The increasing concerns of consumers with the use of synthetic substances in the food industry, interest in research in the field of biomaterials processing, and the restrictive policies of pollution control adopted by governments worldwide have enormously contributed to the development of the so-called clean technologies. One of the most widespread clean technologies is supercritical fluid extraction (SCFE). Potential applications of SCFE to the field of natural products have being intensively investigated during the last two decades. Besides being harmless to the environment, the use of  $CO_2$  as a supercritical solvent permits the processing of thermosensitive material.

The nineties has been the decade of SCFE. Numerous technical and scientific papers were published in the area. Applications run from SCFE of lipid material from several vegetable materials to extraction of essential oils, oleoresins, and other odoriferous materials. For instance, SCFE was used to obtain vegetable oils from canola seeds (Lee et al., 1986), tucumã (Astrocaryum vulgare) (França et al., 1991), passion fruit seeds (Passiflora edulis) (Corrêa, 1994; Corrêa et al., 1994), bacuri (Platonia insignis) (Araújo, 1997), Theobroma grandiflorum (Araújo, 1997; Magno et al., 1999), palm (Elaes guineensis) (Corrêa et al., 1999), and ucuuba (Virola surinamensis) (Moraes et al., 1999). In all of these studies emphasis was given to the kinetics of extraction and to the elucidation of the chemical composition of the extracts. Other studies were devoted to the recuperation of precious substances from different substrata, such as the extraction of fatty acids from the shells of the bacuri fruit (Monteiro et al., 1997), the recovery of carotenes from the residual fibers of the palm (*Elaes guineensis*) industry (França, 1999; França and Meireles, 2000), and the extraction of lipids and vitamins from the shells and pulp of buriti (Mauritia flexuosa) (Ruster, 1997; França et al., 1999) and from the tucumã pulp (Christensen, 1997; Christensen et al., 1999; Christensen et al., 2000). All these studies reported the modeling of the mass transfer process and/or the chemical composition of the extracts.

The pupunha is the fruit of a palm tree from the Northern region of Brazil. The local population consumes the cooked fruit. The pupunha tree conceivably has a great economic potential since every part of the plant has its own specific application. For example, the roots are used as vermifuge, the trunk as wood for house construction, and the flowers as a spice. The heart of palm is obtained from the secondary stems and the fruit is a source of carotenes, vegetable oil, and fatty acids (Serruya et al., 1980; Bentes et al., 1981; Clement, 1991).

The objectives of this work were to study the SCFE of oil from the pupunha (*Guilielma speciosa*) pulp. The influences of process temperature and pressure on the yield and quality of the extracts were investigated. The overall extraction curves were modeled using the single parameter model proposed by Tan and Liou (1989). The constant for the extraction rate was modified to include the effects of extract solubility on the supercritical solvent. The appropriateness of the model was tested using data from the literature for the extraction of oil from tucumã seeds (França and Meireles, 1998).

## MATERIALS AND METHODS

## Materials

The pupunha fruit (Figure 1), from Ilha das Onças (Pará, Brazil) and Santa Maria (Pará, Brazil), was purchased at local stores in Belém (Pará, Brazil). The fruit had an average pulp content of 65%, with approximately 50% humidity, 30% seed, and 5% shell. Both the pulp and shells were used in the experiments. Carbon dioxide, 99.99% pure, was provided by GASPARÁ (Belém, Brazil).

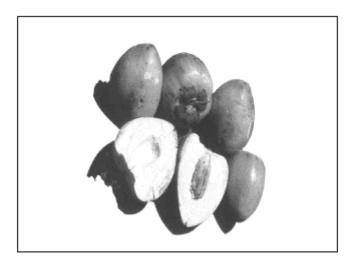


Figure 1: The pupunha fruit used in this work. The shell is yellowish green, the pulp is light yellow, and the seed is dark brown.

## Pretreatment and Characterization of the Raw Material

The fruit was separated into shells, pulp, and seeds. These parts were dried in an oven at 60°C during 36 hours. Thern the dried material was triturated in a rotor mill (TE090, Tecnal, Brazil). The ground material was packed in plastic bags and stored in a domestic refrigerator at approximately 5°C.

The size distribution of the triturated shells and pulp was determined with the aid of a Rot-up shaker and sieves of the Tyler series. Density was determined using the sand pycnometry technique (Buczek and Geldart, 1986). The raw material was characterized with respect to total lipids (method AOCS 920.39), total nitrogen (method AOCS 320.176), ash content, and humidity.

The dried shells and pulp were subjected to conventional (Soxhlet) extraction with hexane. SCFE was performed at 25MPa and 323 K and 30 MPa and 318 K. The assay at 25MPa was performed 6 month prior to that at 30MPa.

The SCFE Unit

The experiments were carried out in the supercritical extraction unit available in the Department of Chemical Engineering at the Federal University of Pará (Brazil). A detailed description of the unit can be found in Chassagnez-Méndez (1998) and França et al. (1999).

The separation unit has been modified and simplified, as illustrated in <u>Figure 2</u>, in order to meet the extraction requirements (extraction using small amounts of raw material) suggested by Reber (1997) and used by Rüster (1997) and França et al. (1999).

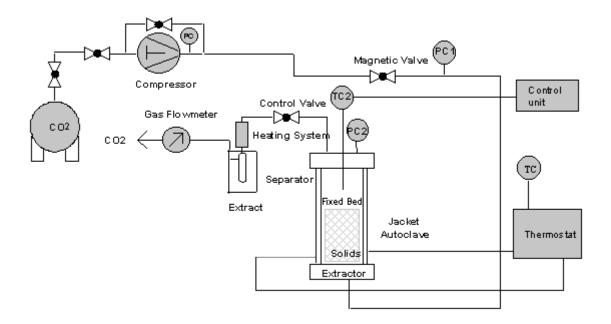


Figure 2: Flowsheet of the modified supercritical extraction unit (França et al., 1999).

## **Experimental Procedure**

The dried and triturated pupunha particles (0.020 kg) were packed inside the extraction cell (made of Stainless Steel with a height of  $4.4.10^{-2}$ m and a diameter of  $2.9.10^{-2}$ m). The extraction cell was adapted to the SCFE unit. Carbon dioxide was delivered at the required pressure by the membrane compressor and passed through a porous plate in order to assure a homogeneous flow through the fixed bed. After passing through the micrometering valve (control valve), the supercritical carbon dioxide containing the solubilized oil was expanded in the small stainless steel tube and the precipitated extract was collected in a glass tube placed inside the separator. The expanded carbon dioxide passed through a flow meter and was released into the atmosphere.

## **Chemical Analysis of the Extract**

The extracts were saponified with a 0.5 N solution of sodium hydroxide in methanol, followed by esterification using solutions of ammonium chloride, sulfuric acid, and

methanol in the proportion 1:1.5:30 (vol./vol.), according to the procedure described by Maia (1992). The composition of the extracts was determined using a gas chromatographer (Hewlett Packard, model 5890, series II) and an integrator (Hewlett Packard, model 3396, series II). The system was equipped with a fused silica capillary column (CP-Sil-8 CB). The carrier gas was helium and 1 m L of the sample was injected. The temperature was raised to 100°C at 6°C/min and from 100°C to 300°C at 5°C/min. A mixture of pure fatty acids was used as an external standard (Merck, Darmstad). The retention times for both the samples and the standards were converted to retention times relative to the palmitic acid due to analytical difficulties. The identification of the fatty acids was based on: (i) a comparison of the relative retention times for the sample with the graph for the fatty acids homologous series, as described by Araújo (1997). This procedure assumes that there is a linear relationship between the relative retention times for substances of a homologous series.

#### MATHEMATICAL MODELING

The extraction from solid substrata at low as well as at high pressures involves at least two phases, the solid and the fluid. The content of solute in each phase is expressed in terms of phase volume and/or phase solvent. Thus, to model the rate of interfacial mass transfer a partition coefficient is required. In general, the simplest way to deal with the problem is to model the phases separately (Brunner, 1994). Thus, the expression for the rate of interfacial mass transfer will contain the required partition coefficient.

The predominant mass transfer mechanisms can vary from system to system. For some substrata the resistance can be located in the solid phase, while for others in the fluid phase. It may also be possible that the resistance in both phases must be considered. One of the most frequently used approaches employs the overall mass balance for an element of the fixed bed.

Several models describing the mass transfer for SCFE from solid substrata have been cited in the literature (Brunner, 1994; Reverchon, 1997). Of these, the single-parameter model proposed by Tan and Liou (1989) was selected to describe SCFE for the pupunha-CO<sub>2</sub> system. This model represents the rate of interfacial mass transfer by a first-order kinetic model. The mass balance for an element of the bed is given by

Fluid phase:

$$\varepsilon \rho \frac{\partial Y}{\partial t} + U \rho \frac{\partial Y}{\partial z} = -(1 - \varepsilon) \rho_S \frac{\partial X}{\partial t}$$
(1)

Solid phase:

$$\frac{\partial X}{\partial t} = -kX \tag{2}$$

with the following initial and boundary conditions:

$$Y(z,0) = 0 \tag{3}$$

$$Y(0,t) = 0$$
 (4)

$$X(0) = X_0$$
 (5)

The dynamic behavior of the mass ratio of solute in the fluid phase at the bed outlet can be obtained by solving Eqs. 1 and 2 with the boundary conditions given by Eqs. 4 to 6, resulting in

$$Y_{H} = Y(H, t) = \frac{1 - \varepsilon}{\varepsilon} X_{0} \frac{\rho_{s}}{\rho} \left[ exp\left(\frac{k dH}{U}\right) - 1 \right] exp(-kt)$$
(6)

Integration of Eq. 6 from the beginning of extraction to time t gives the overall extraction curve:

$$\mathbf{m}_{\text{est}} = \int_0^t \mathbf{Y}_H \mathbf{Q}_{CO_2} \, \mathrm{dt} = \frac{\mathbf{A}}{\mathbf{k}} \left[ \mathbf{1} - \mathbf{e}^{\mathbf{k}\mathbf{B}} \right] \left( \mathbf{e}^{-\mathbf{k}\mathbf{t}} - 1 \right) \tag{7}$$

with

$$A = Q_{c0_2} \frac{1-\varepsilon}{\varepsilon} X_0 \frac{\rho_s}{\rho}$$
(8)

$$B = \frac{\varepsilon HS}{q} \rho \tag{9}$$

where Y is the mass ratio of solute in the fluid phase (kg/kg-CO<sub>2</sub>) and X is the mass of solute in the solid phase (kg/kg-inert solid), U is the superficial velocity (m/s), H is the bed length (m), k is the extraction constant (s<sup>-1</sup>), S is the cross-sectional area (m<sup>2</sup>) of the bed,  $^{Q}co_{2}$  is the solvent flow rate (kg/s), r<sub>s</sub> is the solid phase density (kg/m<sup>3</sup>), r is the fluid phase density (kg/m<sup>3</sup>), and e is the fixed bed porosity.

The extraction constant is given by (Tan and Liou, 1989)

$$k = k_0 \exp\left(\frac{-E}{RT}\right)$$
(10)

where  $k_0$  is a function of pressure and activation energy E is characteristic of the system.

To model the overall extraction curves, the extraction constant must be known. The results of Tan and Liou (1989) show that the desorption rate increased as temperature increased. For the SCFE of lipid from vegetable seeds, the extraction rate increased with the increase in pressure up to 30 MPa. The extraction rate also increased with the decrease in temperature for temperatures within the interval of 313 and 343 K (França et al., 1999; Corrêa et al., 1999). A similar behavior has been observed for solubility. Therefore, it is reasonable to expect the extraction constant to be a function of the solubility. Thus,

$$\mathbf{k} = \mathbf{k}_{\mathbf{r}}\mathbf{c} \tag{11}$$

where  $k_r$  is the proportionality constant for the extraction constant. Chrastill (1982) published a model that took into account the possibility of a chemical reaction between the solute and the solvent. The model was proposed to describe the solubility of lipid material in carbon dioxide and is given by

$$c = \rho^{n} \exp\left[\frac{a}{T} + b\right]$$
(12)

a = DH/R is the heat of reaction and it has a physical meaning similar to the activation energy given in Eq. 10. b is a model constant and n is the association number for the solvent. The Chrastill (1989) model was used by del Valle and Aguilera (1988) to correlate the solubility of vegetable oils in supercritical CO<sub>2</sub>. The authors used data for soybean, sunflower, and cottonseed oils at pressures of 15 to 68 MPa, 23 to 67, and 75 to 102 MPa, respectively. They also used data for corn oil obtained at 26.4 MPa. To improve the fit of the data, del Valle and Aguilera (1988) introduced a new parameter into the Chrastill model. The new parameter accounted for the variation in heat of vaporization as temperature varied. Their final equation was

$$\ln c = 40,361 - \frac{18708}{T} + \frac{2186840}{T^2} + 10,724\ln\rho$$
(13)

where r is in  $g/cm^3$  and c in g/L.

Using Eqs. 7, 11, and 13 and data for the overall extraction curves obtained at different temperatures, it is possible to estimate the value of the extraction constant.

#### **RESULTS AND DISCUSSION**

<u>Table 1</u> shows the characteristics of the raw material. The particle size distribution of the fixed bed had the following distribution: 49% of the particles had an average diameter of  $0.354.10^{-3}$  m, 32% of the particles had an average diameter of  $0.596.10^{-3}$  m, and 19% of the particles had an average diameter of  $0.210.10^{-3}$  m. The fixed bed porosity was 0.293.

	Density (kg/m <sup>3</sup> )	Humidity (%)	Lipids content (%)	Proteins (%)	Ash (%)
Բահր	960	7.16	12.25	8.13	2.17
Shells	800	3.91	8.84	15.82	4.05

Table 1: Characteristics of the raw material.

The experimental conditions and yields are shown in <u>Table 2</u>. <u>Table 3</u> shows the composition of the hexanic and SCF overall extracts. Oleic acid (C-18:1) is the most abundant constituent in both extracts.

## Table 2: Experimental conditions and yields for the SCFE from Guilielma speciosa.

Temperature Pressure		Mass of feed Q <sub>CO2</sub>		Total time	Yield*	
К	MPa	10 <sup>3</sup> kg	10 <sup>3</sup> kg/s	sec/60	%	
323	25	20	0.31	100	13	
318	30	20	0.28	78	9	

\*Mass of extract/mass of feed

Table 3: Fatty acids content of the total extract obtained
by hexane extraction and SCFE.

	Relative proportion (%)					
	Hex	ane	SCF extr	SCF extract (pulp)		
Fatty acids*	Shells	Pulp	25MP a	30 MP a		
C-6:0 (Caproic acid)			7.74			
C-10:0 (Capric acid)	1.76					
C-12:0 (Lauric acid)	4.42					
C-14:0 (Myristic acid)	6.99	9.55	5.34	8.71		
C-16:0 (Palmitic acid)	24.13	27.34	26.18	40.83		
C-18:0 (Stearic acid)	9.60	7.96	31.26	5.90		
C-18:1 (Oleic acid)	53.10	55.15	24.85	44.56		
C-20:0 (Arachidic acid)			4.63			

\* C-n:m: where n is the number of carbon atoms and m the number of double bonds

Table 4 shows the fatty acids profile for the assay carried out at 25 MPa and 323 K. The amount of fatty acids varies during the extraction process. The composition of the extracts varies considerably during extraction. An increase in the content of caproic and capric acids always coincides with a decrease in the amount of myristic acid. In the same manner, the observed decrease in stearic acid content was accompanied by an increase in the amount of oleic acid. These observations can be explained by two factors. First, due to the nature of the pupunha pulp it is possible that the fixed bed was not entirely homogeneous. The second and most probable explanation is that at the extraction temperature the medium had become reactive. Thus, it may be possible that the oleic acid was converted into stearic acid and myristic acid into caproic acid. Nevertheless, another series of experiments would need to be performed to confirm this hypothesis.

	Time of extraction (\$/60)								
	10	20	30	40	50	60	70	80	90
Fatty acids	Relative proportions (%)								
C-6:0	5.10	1.55	3.36	24.10	1.98	5.41	6.76	14.04	7.44
C-8:0	5.60								
C-14:0		9.60	8.85		8.99	6.48	5.88		6.48
C-16:0	31.25	28.65	28.02	26.52	28.80	24.07	19.95	27.97	19.85
C-18:0	31.61	56.41	6.62	49.38	56.48	7.40	5.81	57.99	6.04
C-18:1	24.67	3.59	53.15		3.75	56.64	39.87		42.0
C-20:0	1.77						21.73		18.19

Table 4: Fatty acids profile of the SCF extract obtained at 25MPa and 323 K.

#### **The Overall Extraction Curves**

The data of França and Meireles (1998) for SCFE of oil from tucumã seeds obtained at 30 MPa and temperatures of 313, 328, and 343 K were used to estimate the extraction constant. The least square fit of their data gave  $k_r = 7.10^{-5} \text{ m}^3/\text{kg.s.}$  The tucumã overall extraction curves were described well by the Tan and Liou (1989) single parameter model, as can be observed in Figure 3. Using the same value of  $k_r$ , the overall extraction curve for the SCFE from pupunha pulp was evaluated. As can be seen in Figure 4, the model underestimated the experimental data. This suggests that parameter k<sub>r</sub> also depends on the properties of the solid substrata. The data on tucumã extraction were selected because the characteristics of the solid matrices are similar. Nevertheless, tucumã pulp has a larger fiber content than pupunha pulp. In addition, the length and the diameter of the bed used by França and Meireles (1998) were different from those used for pupunha pulp extraction. The alternative to investigating the appropriateness of the model used to describe the overall extraction curves for SCFE from pupunha pulp was to treat the extraction constant of Eq. 7 and 11 as an empirical parameter. Thus, fitting the experimental data for the overall extraction curves for SCFE from pupunha pulp to the model gave  $k_r = 10^{-4} \text{ m}^3/\text{kg.s.}$  Table 5 shows the estimated model parameters and

Figure 4 compares experimental and calculated overall extraction curves. The results were much better than the previous ones. This corroborates the idea put forward that parameter  $k_r$  is a function of the properties of the solid substrata, as anticipated.

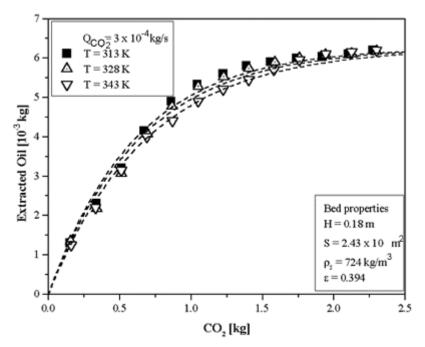


Figure 3: Experimental and calculated overall extraction curves for the data from França and Meireles (1998).

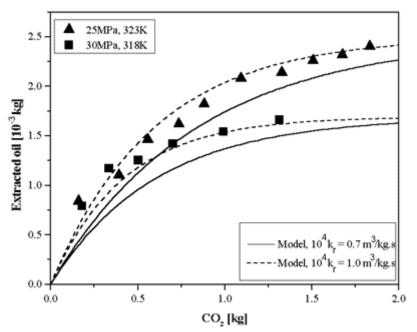


Figure 4: Experimental and calculated overall extraction curves for the SCFE from pupunha pulp.

Temperature K	Pressure MP a	ρ kg/m <sup>3</sup>	X <sub>0</sub>	Q <sub>co1</sub> 10 <sup>3</sup> kg/s	c kg/m <sup>3</sup>	10 <sup>4</sup> k s <sup>-1</sup>
323	25	850	0.126	0.31	5.26	5.33
318	30	890	0.085	028	6.73	6.67

 Table 5: Experimental conditions and estimated model parameters

 for SCFE from pupunha.

#### CONCLUSIONS

The difference in yield between the two overall extraction curves is possibly a result of the modifications suffered by the solid substrata during storage. It is well known that even at low temperatures solid matrices with high lipid contents are subject to oxidation reaction. Nonetheless, the two overall extraction curves have the expected behavior for SCFE from oily material. The mathematical model used was capable of describing the overall extraction curves for both tucumã and pupunha pulp. Comparison of the results showed that the extraction constant depends not only on solubility and/or temperature but also on the characteristics of the solid substrata.

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