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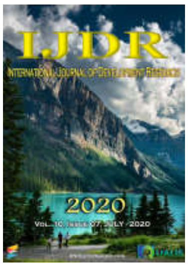

RESEARCH ARTICLE

OPEN ACCESS

EFFECTS OF CONVECTIVE DRYING IN FIXED BED ON CONTENT AND COMPOSITION OF ESSENTIAL OILS FROM *Croton cajucara* Benth. AND *Ocimum micranthum* Willd

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ABSTRACT

This study investigated how operating conditions in convective drying in fixed bed influences affects yield and composition of the essential oils of sacaca (*Croton cajucara* Benth) and alfavaca (*Ocimum micranthum* Willd). The essential oil yields were highest (0.48% w/w for sacaca and 4.66% w/w for alfavaca) with 100 g of moist leaves, 0.47 m/s of drying air velocity and 60 °C of drying air temperature. Low temperatures and low loads of wet material (2.19 mol/L) produced the highest content of linalool, a major compound of sacaca essential oil. In contrast, high temperatures and high loads of wet material (8.94 mol/L) produced the highest value of methyl eugenol, a major compound of Amazonian basil essential oil. These results show that linalool (present in sacaca essential oils) is more susceptible to drying operating conditions (drying air temperature and wet material loading) than methyl eugenol (present in alfavaca essential oils).

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INTRODUCTION

Essential oils are volatile and refractive liquids with a characteristic odor that may be present in several parts of the plant, including leaves, husks and fruits (Andrade and Gomes, 2000). Some plants contain essential oils that are of interest to the food and pharmaceutical industries, which may use them for food flavoring, or as ingredients for cosmetics, perfumes and drugs (Braga, 2002). Some volatile oils are used directly as medicine. Essential oil-rich plants may also be pulverized and used as seasoning (star anise, clove, nutmeg). *Croton cajucara* Benth. (family Euphorbiaceae), also known as sacaca, is a plant species from the Amazon whose essential oil contains linalool, which is used as a fixative for fragrances, and also has anti-inflammatory, analgesic, hypotensive, vasodilator, antinociceptive and antimicrobial activities (Araújo *et al*., 1971; Peana *et al*., 2002; Maciel *et al*., 2006). *Ocimum micranthum* Willd (Lamiaceae family), commonly known in the Amazon region and in Brazil as alfavaca, country alfavaca, wild alfavaca, chicken alfavaca, favaquinha and Amazonian basil, is an important source of essential oils that contains phenolic and aromatic antioxidant compounds of value to the food and pharmaceutical industries (Pandey *et al*., 2014). A predominant compound of alfavaca essential oil is methyl eugenol, used in the cosmetics industry for soap and shampoo production, and also as a flavoring agent for jelly, non-alcoholic drinks, chewing gum and ice creams (Santos, 2011). The drying process stops the enzymatic action that causes degradation of plant active compounds, and thus is a processing step in the production of essential oils from the majority of medicinal, aromatic and seasoning plant species (Corrêa Jr. *et al*., 2000; Hewrtwig, 1991). Examples are the extraction of essential oils from branches and leaves of long pepper (*Piper hispidinervum* C. DC.), where humidity superior to 30% of wet basis (w.b.) promotes emulsion, hindering the separation of oil and water and resulting in low oil yield (Maia and Silva, 1995); or from lemongrass leaves (*Cymbopogon* *citratus* (D.C.) Stapf), for which raising the drying air temperature increased essential oil yield by 21%, compared to fresh leaves. Therefore, this work aimed to establish operating conditions in a convective fixed bed drier that result in higher yields of essential oils from sacaca and alfavaca leaves, without altering their chemical composition.

MATERIALS AND METHODS

Alfavaca and sacaca leaves were dried, and their essential oils extracted, at the Materials Laboratory at Faculdade de Tecnologia, Universidade Federal do Amazonas, Manaus, AM, Brazil. The gas chromatography of essential oils was performed at the Natural Products Laboratory, Chemistry Department, Instituto de Ciências Exatas, Universidade Federal do Amazonas (Distrito Industrial de Manaus, Manaus, AM, Brazil).

Plant material: The study material consisted of green leaves from alfavaca and sacaca, derived from plantations in Embrapa Western Amazon, at km 29, AM 010 highway, Manaus, AM, Brazil. The study was performed between November 2014 and March 2015. Alfavaca seeds (collected from a natural population) were planted in 128-cell expanded polystyrene trays, and irrigated daily. Germination occurred after 7 days. On day 60, the seedlings were transplanted to fertilized beds and irrigated as necessary (Figures 1 and 2).

Figure 1. Alfavaca seedlings in the tray

Figure 2. Seedlings planted in beds

The leaves were collected at day 75 (in March 2015). Afterwards, they were separated from the branches, placed in plastic bags and transported to the Materials Laboratory for drying and extraction of essential oils. Sacaca leaves were collected on November 2014, from plants with 14 years of vegetative stage (Figure 3), in a plantation of the Medicinal Plant Sector at Embrapa Western Amazon. After collection, sacaca leaves were separated from the branches and placed in plastic bags that were sent to the Materials Laboratory for drying and extraction of essential oils.

Figure 3. Planting sacaca in Embrapa Western Amazon, Manaus, AM, Brazil

Drying method: A convective fixed bed dryer, consisting of an acrylic fixed bed drier with forced air convection, was used to dry sacaca and alfavaca leaves. The forced air system consists of a 4 hp radial compressor (model CR6, IBRAM, São Paulo, SP, Brazil), with a maximum flow of 4.2 L/min and minimum pressure 2700 mmH_2O, with a pipe attached to the air transport line to control air flow to the dryer. A 1320 W electrical resistance was used to heat the drying agent, and a data acquisition and recording system (model *FieldLogge*r I/O, NOVUS, Curitiba, PR, Brazil), with the NOVUS *FieldChart* v1.96 software, was adapted to this unit. The Fieldlogger records data on dry bulb temperature, relative humidity and pressure drop in the 2” pipe, using an L-shaped Pitot pipe (model TPL-03-100, KIMO, Curitiba, PR, Brazil), to monitor the flow of drying air towards the fixed bed. The mass of wet material (sacaca or alfavaca leaves) to be dried was weighed in a semi-analytical scale with 0.01 g precision (Model BK2000, Gehaka, São Paulo, SP, Brazil), and a humidity analyzer with a precision of 0.01 g (model MB35, OHAUS, São Paulo, SP, Brazil), was used to determine leaf humidity for both species.

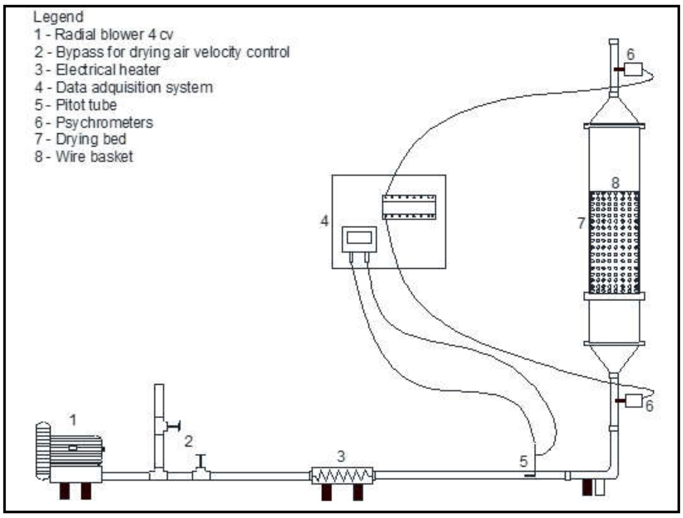


Figure 4. Drying system used in this study

The drying process started by adjusting the drying air temperature and humidity using resistances, monitored by humidity meters and thermocouples in the air feeding line and in the air exit line. Equilibrium was achieved after one hour, as evidenced by no significant variation detected in the temperature and humidity meters placed in the drying bed input and output air. Then, the fresh leaves were placed horizontally in a basket within the drying bed for 1h.

Essential oil extraction: Essential oils were extracted by hydrodistillation, using a distiller with a 2000 mL flat-bottom flask and a 2 kW heating mantle (AP137, Clevenger, São Paulo, SP, Brazil). Leaves were weighed on a semi-analytical scale (model BG2000, GEHAKA, São Paulo, SP, Brazil) with 0.001 g precision. Cooled water pumped from an ultra-thermostatic bath (model 214M2, QUIMIS, São Paulo, SP, Brazil) was used for condensation. The oily phase was removed with a Pasteur pipette and transferred to 2.0 mL transparent glass flasks, for storage in a refrigerator at 5 ^{o}C. The essential oil extraction equipment is depicted in Figure 5.

Figure 5. Essential oil extraction system. (a) Clevenger (b) Ultra thermostatic bath with pump

The essential oil yield, expressed in g, wet basis (fresh leaves), was calculated with equation 1:

$$Rd(\%bu) = \frac{m_{óleo}}{m_{ff}} \times 100 \qquad (3.1)$$

where:

m_{ff} – mass of fresh leaves, g;
$m_{óleo}$ – mass of essential oil, g.

Chromatographic analysis of essential oil: After calculating essential oil yield, content of the main components of essential oil was determined by gas chromatography for each situation (fresh and dry leaves). Linalool and nerolidol contents were determined for sacaca, and methyl eugenol and beta caryophyllene contents were determined for alfavaca. A gas chromatographer (model Trace 1310, Thermo, São Paulo, SP, Brazil) with a 25 m × 0.25 mm × 0.25 µm VF-1ms capillary column (Agilent, São Paulo, SP, Brazil) was employed. The oven had an initial temperature of 60 ºC and heat was ramped up at 3 °C/min until 160 ºC, followed by a gradient of 10 ºC/min until 250 ºC and final isotherm 2 min. The injector and detector temperatures were 220 °C and 250 °C, respectively. The injection flow of the dragging gas, helium, was l mL/min. The samples were solubilized in dichloromethane (1000 µL), injected at a concentration of 5 mg/mL with an injection volume of 1 µL and Split 1:2. Each analysis run lasted 44.33 minutes.

Statistical analysis: A statistical model was developed based on the response surface methodology using temperature and velocity of drying air, material loading and drying time as input variables. The goal was to obtain statistical models capable of properly predicting the final humidity, contents of methyl eugenol (in the essential oil from alfavaca) and linalool (in the essential oil from sacaca), and essential oil yield, in relation to the most significant variables of the drying process in fixed bed. Table 1 shows the variables and respective levels used to optimize leaf drying in fixed bed for the species under study.

Table 1. Coded and original values of the variables used to design the response surface methodology

Original variables (notation)	Coded variables	Units	Variables		
			-1	0	+1
Air temperature ($\boldsymbol{T_a}$)	X_1	℃	40	50	60
Material load ($\boldsymbol{W_0}$)	X_2	g	100	150	200
Air velocity ($\boldsymbol{v_a}$)	X_3	m/s	0.47	0.68	0.85

A fractional factorial design with a center point (2^{k-1}, where k is the number of variables) was used to evaluate the drying kinetics. The dimensionless contents of chemical components of sacaca and alfavaca essential oils and the dimensionless yield of essential oils were calculated by the ratio between average post-drying values and average pre-drying values (extraction assays were performed in duplicate). Ten experimental runs were performed randomly (8 experimental runs plus 2 in the center point), to minimize the error. Table 2 summarizes the original experimental matrix, containing the values for the response variables. The amounts of material loaded for this statistical analysis were chosen according to the drying bed capacity and the amount of material collected in Embrapa Western Amazon. Drying air temperature, drying air velocity and leaf loading values encompassed the minimum and maximum capacity of the blower, based on the characteristics of the fixed bed dryer.

Table 2. Experimental matrix

Ensaios	$\boldsymbol{X_1}$	$\boldsymbol{X_2}$	$\boldsymbol{X_3}$	$\boldsymbol{C_m}$	$\boldsymbol{C_l}$	$\boldsymbol{Rd_S}$	$\boldsymbol{Rd_A}$
1	+	-	-	C_{m1}	C_{l1}	Rd_{S1}	Rd_{A1}
2	-	+	-	C_{m2}	C_{l2}	Rd_{S2}	Rd_{A2}
3	-	-	+	C_{m3}	C_{l3}	Rd_{S3}	Rd_{A3}
4	+	+	+	C_{m4}	C_{l4}	Rd_{S4}	Rd_{A4}
5	0	0	0	C_{m5}	C_{l5}	Rd_{S5}	Rd_{A5}
6	+	-	-	C_{m6}	C_{l6}	Rd_{S6}	Rd_{A6}
7	-	+	-	C_{m7}	C_{l7}	Rd_{S7}	Rd_{A7}
8	-	-	+	C_{m8}	C_{l8}	Rd_{S8}	Rd_{A8}
9	+	+	+	C_{m9}	C_{l9}	Rd_{S9}	Rd_{A9}
10	0	0	0	C_{m10}	C_{l10}	Rd_{S10}	Rd_{A10}

where:

X_1, X_2, X_3, X_4 coded variables;
C_m – methyl eugenol content (mol/L);
C_l – dimensionless content of linalool (mol/L);
Rd – essential oil yield (% m/m);
X- final humidity content (% wb).

RESULTS AND DISCUSSION

Table 3 shows the results obtained for the experimental matrix proposed in this study.

Table 3. Results of the experimental matrix

Test	*v* (m/s)	T (°C)	W_0 (g)	C_m (mol/l)	C_i (mol/l)	Rd_s (%m/m)	Rd_A (%m/m)
1	+	-	-	5.3542	2.1941	0.4497	2.8944
2	-	+	-	4.9650	0.1232	0.1350	4.6589
3	-	-	+	4.7490	1.4099	0.4130	1.8020
4	+	+	+	8.9419	1.5531	0.1348	2.4829
5	0	0	0	5.4593	2.0297	0.1058	3.2514
6	+	-	-	4.8800	1.3084	0.3711	2.1834
7	-	+	-	5.2764	1.1744	0.3313	3.6838
8	-	-	+	4.9233	0.9929	0.4874	1.2091
9	+	+	+	5.1294	1.5107	0.2152	2.7488
10	0	0	0	5.9172	1.2721	0.2543	2.6487

Drying parameters affected essential oil yield from sacaca and alfavaca leaves differently using surface response method at 95% confidence level. Sacaca is a perennial species with a low essential oil yield, hindering large-scale production, while alfavaca produces plentiful leaves and a large essential oil yield. The greatest essential oil yields for both sacaca and alfavaca were achieved with low drying air velocities (0.47 m/s), low loadings of wet leaves (100 g) and low drying air temperatures (40 ºC). However, alfavaca essential oil yield was reached at high temperatures (60 °C), maintaining low material loads and air speed. These results are consistent with Smith *et al* (2007), which achieved higher essential oil yields at an air speed of 1.9 m/s and drying air temperature of 40 °C. These observations are shown in Figure 6. The significance of the calculated effects on the responses is easily visualized using Pareto diagrams (Box *et al*., 1978). Figures 8 and 9 show the Pareto diagrams obtained to analyze the significance of effects v (drying air velocity), T (drying air temperature) and W (material load) on the essential oil yield (%) for sacaca. Figures 7 and 8 (below) illustrate this behavior.

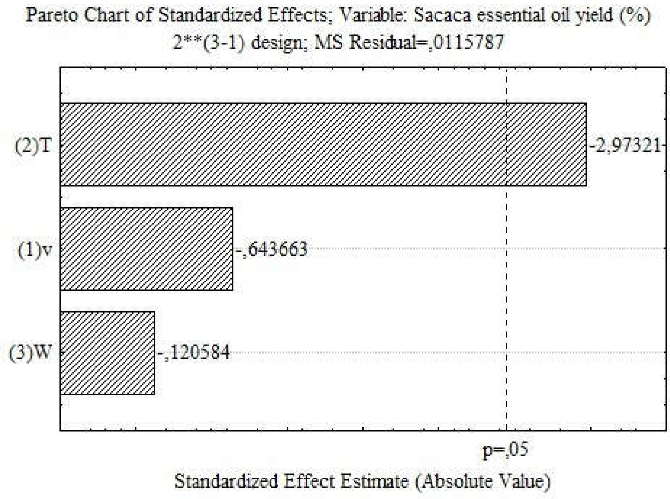


Figure 7. Effects of drying parameters on essential oil yield for sacaca

Increasing drying air temperature negatively affected essential oil yield for sacaca. With a temperature increase from 40 ºC to 60 ºC, essential oil yield decreased by 51.7% for sacaca. This drying air temperature favored entrainment of alfavaca essential oil through the rupture of the pockets that hold the essential oil.

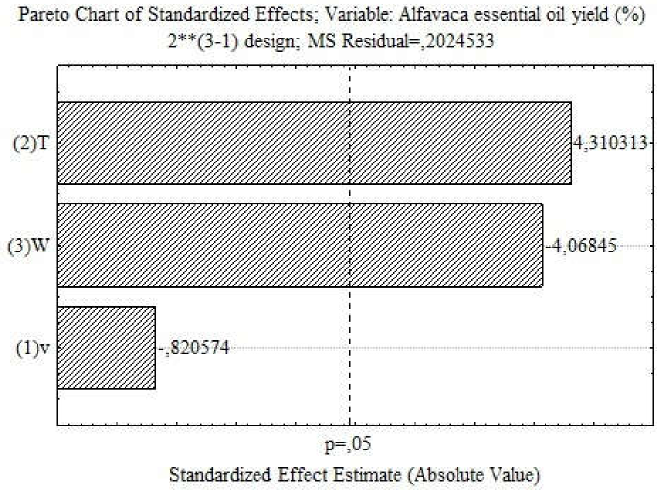
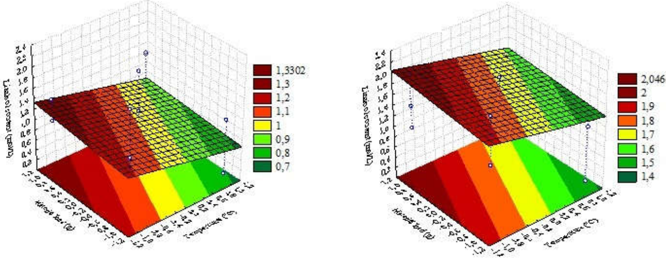


Figure 8. Effects of drying parameters on essential oil yield for alfavaca

The evaluation of the operating conditions of drying parameters on the content of the main components of sacaca and alfavaca essential oils (linalool and methyl eugenol, respectively) are shown in Figures 9-11. The increase of the drying air velocity, from 0.47 m/s to 0.85 m/s, at high temperatures and high loads of material, caused a 1.5–2.1 mol/L loss in linalool content, due to this compound having higher volatility under these drying conditions (Figure 9).



(a) v = 0.47 m/s (b) v = 0.85 m/s

Figure 9. Effects of drying air velocity on linalool content in sacaca essential oil

Increasing the leaf mass loaded but decreasing the temperature of the drying air increased linalool content in sacaca essential oil. Increasing the material load results in higher linalool content because the greater mass of material takes longer to heat, and thus there is less removal of the most volatile compounds. The effect of the higher temperature on content of linalool in the essential oil is due to the entrainment of other more volatile compounds (Venskutonis *et al*., 1996). For the major alfavaca essential oil component, methyl eugenol, increase in air velocity, from 0.47 m/s to 0.85 m/s, increased content from 6.0 mol/L to 7.0 mol/L for high loadings of wet material and high drying air temperatures (Figure 10). According to Venskutonis *et al.* (1996), this response is due to the high volatilities of other components.

Increasing alfavaca leaf mass raised the methyl eugenol content for high drying air speeds and temperatures. These conditions preserved the least volatile compounds, as time was too short to heat the leaves enough to cause volatilization of methyl eugenol (Venskutonis et al., 1996; Braga et al., 2004). Methyl eugenol content increased from 6.5 mol/L to 7.5 mol/L (Figure 11).

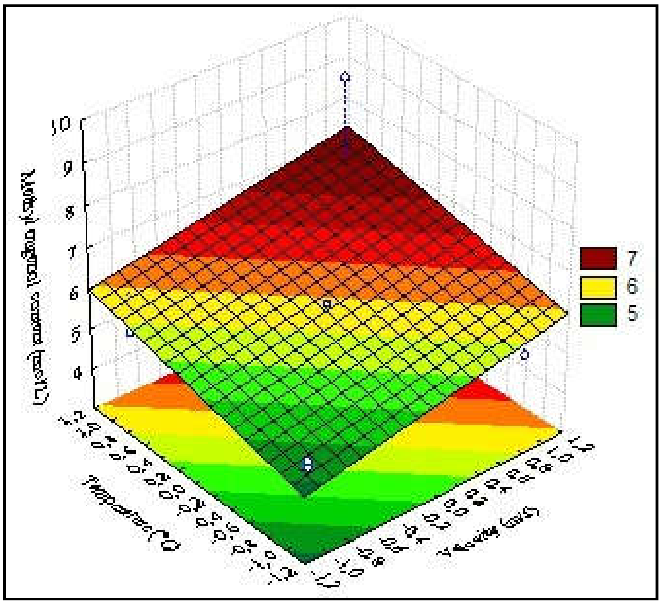


Figure 10. Effects of drying air velocity on methyl eugenol content

Increasing alfavaca leaf mass raised the methyl eugenol content for high drying air speeds and temperatures. These conditions preserved the least volatile compounds, as time was too short to heat the leaves enough to cause volatilization of methyl eugenol (Venskutonis et al., 1996; Braga et al., 2004). Methyl eugenol content increased from 6.5 mol/L to 7.5 mol/L (Figure 11).

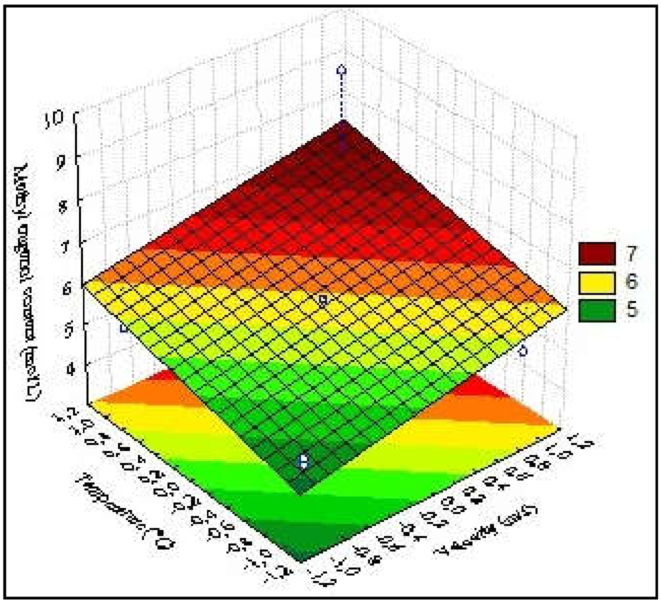


Figure 11. Effects of alfavaca leaf material loading on methyl eugenol content

Increasing the drying air temperature increased the methyl eugenol content from 6.2 mol/L to 7.4 mol/L), when using high leaf material loadings and high drying air velocities. This result is consistent with reports by Venskutonis et al (1996), Braga et al (2004) and Hanaa et al. (2012).

Conclusions

This work has shown the effect of drying alfavaca (*Ocimum micranthum* Willd) and sacaca (*Croton cajucara* Benth) leaves in a fixed bed dryer under specific operating conditions on the essential oil yield and major component content. The dryer used in this work provided high yields for alfavaca leaves but not for sacaca leaves. This behavior is directly linked to the moisture removal from the leaves and to the manner of storage of essential oils in the storage pockets of the leaves of each species. The statistical analysis identified the variables in the drying process of sacaca and alfavaca leaves that affected the essential oil results. The largest essential oil yields were achieved at low drying air speeds (0.47 m/s), high drying air temperatures (60 °C) and low leaf loads (100 g) for alfavaca. However, in these conditions the concentrations of major compounds were reduced, due precisely to the high temperature (60 °C) which, by increasing volatility, promotes the entrainment of essential oil (Venskutonis et al., 1996; Braga et al., 2004; Hanaa et al. 2012). For sacaca the largest essential oil yields were achieved at low drying air speeds (0.47 m/s), low drying air temperatures (40 °C) and high leaf loads (200 g). The highest content of the major sacaca essential oil component, linalool (2.2 mol/L) was reached at high drying air speeds, low air temperatures, and low leaf loads. Meanwhile, for alfavaca, the highest content of methyl eugenol (8.94 mol /L) was obtained also at high speeds of the drying air, but at high drying air temperatures and high loads of leaf material.

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