

# PATCHOULI [*Pogostemon cablin* (Blanco) Benth.] ESSENTIAL OIL: SFE YIELD AND HARVEST SEASONS

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The Supercritical Fluid Extraction (SFE) of Patchouli [*Pogostemon cablin* (Blanco) Benth.] essential oil has been studied. In this paper, the effects of harvest seasons about SFE yield were investigated. Patchouli is a tropical perennial herb that originates in Southeast Asia, but now grows mainly in Malaysia, Indonesia, India, Brazil and others. Patchouli oil is a viscous, pale to dark amber brown coloured liquid and is extensively used in perfumery. Its main components are sesquiterpenes (40-45%), sesquiterpenoid alcohols (30-35%) and others [3]. The patchouli leaves (raw material) was collected always in first hours of sunny days. Immediately, it was taken to the Process Control Laboratory, at EQA/UFSC, where it was dried at 30°C and 120 h. Then, the raw material was fractioned in a grinder and it was classified in a sieve shaker. One fixed bed extractor (length 50.0 cm, inner diameter 2.1 cm) was packed manually with 50g, approximately. The SFE was carried out at 100 bar and 32°C by 7 hours, with one hour of static period. The solvent used was CO<sub>2</sub> supercritical fluid. These conditions were performed according [1]. The results were analysed in terms of yield (g essential oil/g patchouli), varying the raw material harvest seasons (winter 2006 and 2007, fall 2007). The yield values of SFE carried out were in 2006 winter (3.13%), in 2007 winter (5.94%) and 2007 fall (5.06%). Mass transfer by diffusion of CO<sub>2</sub> on patchouli is a slowly step and govern the first minutes of process. The amount of patchouli and its pack inside extractor control the time of extraction and the diffusion. However, information on the influence time of photo-period and harvest seasons are important to evaluate the process and its yield [2]. The composition of patchouli essential oil was analyzed using GC-MS and FID systems which showed the presence of main components (sesquiterpenes and sesquiterpenoid alcohols).

## INTRODUCTION

Patchouli [*Pogostemon cablin* (Blanco) Benth.] is a kind of the family *Lamiaceae* or lip-flower (mint). The plant is a tropical perennial herb which originated in Southeast Asia and now grows mainly in Malaysia, Indonesia, Philippines, China, India, Seychelles and Brazil. Its oil is a viscous, pale to dark amber brown coloured liquid and is extensively used in perfumery, being its main components sesquiterpenes and sesquiterpenoid alcohols. It is used as a stimulant, relaxing, digestive, respiratory, nervous system, spleen, stomach, is not irritating and is not toxic [3].

The carbon dioxide is one of the most fluids used in supercritical extraction. It is not toxic and not flammable solvent. Used up moderate temperatures, allow extracting essential oils most noble and pure, compared to other methods of extraction, such as with organic solvents.

The extraction of one or more families of compounds of a natural matrix is the application most frequently requested and studied in cases of separation with supercritical fluids (SCF). The basic scheme of extraction consists of a pressure vessel loaded with the raw material to be extracted. Generally, the material is initially dried and grinded, in order to facilitate the process of extraction. It is loaded in a basket located within the extractor that allows rapid charge and discharge. The SCF flows through of the extractor outlet by a depressurization valve in which, due to the low pressure, the extracts are released in the gaseous medium and collected [4].

The distribution of rainfall and photo-period throughout the year helps in the development of the plant, so that each season of the year has been different compositions of the same essential oil. The influence of irrigation, organic fertilization and nitrogen levels applied to the plant on the yield of extraction of the oil (steam distillation) and the quantity of plants produced. Obtained significant increases in all variables studied (30, 82 and 128%, respectively), increasing also in the plants evaluated with differences in a year [2].

The diffusion of supercritical CO<sub>2</sub> in patchouli governs the first step of the process. It is a very slow step, being influenced by the amount of raw material and for its compaction inside the pressure vessel.

The objective of this study was to compare the yields of the supercritical extraction of patchouli essential oil and analyze the composition of oils obtained in two different seasons of the year: fall and winter. The results were analyzed by gas chromatography coupled to mass spectrometry (GC-MS) and by flame ionization detector (FID).

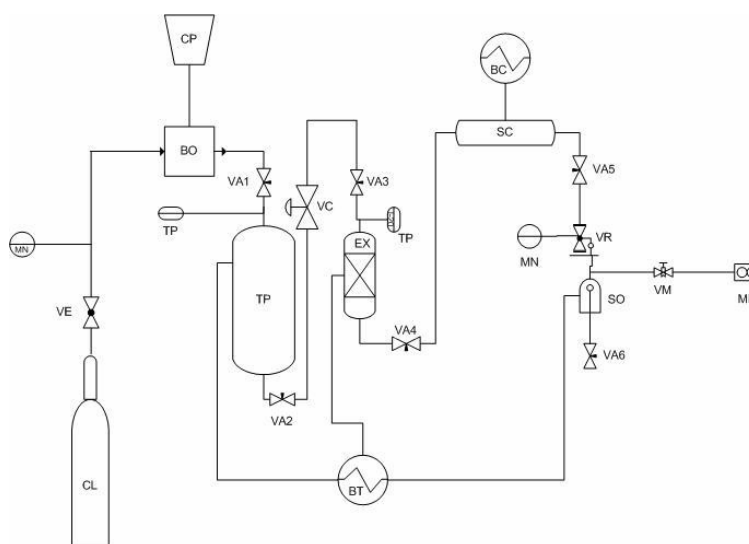
## MATERIAL AND METHODS

### Plant material

Tests were performed using dried leaves of patchouli, cultivated in Brazil and collected in 2006 september and 2006/2007 july (fall and winter). The raw material was dried at room temperature (approximately 20°C) and protected from direct light. The particle diameter for dried leaves, after size reduction, was estimated by using sieves and the mean diameter found was 0.555 mm. The moisture content was about 10% by weight on dry basis.

### Supercritical extraction apparatus and procedure

Fifty grams of the dried leaves were packed in a extractor of high-pressure, flow-up stream extraction apparatus. It is schematically shown in Figure 1. The extraction experiments were performed in a tubular fixed bed 0,175 L of capacity. Carbonyl dioxide was used as supercritical solvent. Extractions were carried out 32°C, at pressures of 10 MPa. Flow rate of supercritical CO<sub>2</sub> was 1.2 L min<sup>-1</sup> and extraction time for all experiments was 420 min with 60 minutes of static period. The solvent flows through a jacket vessel with controlled temperature. A back-pressure valve regulator adjusted the pressure in the extractor measured by a pressure transducer.



**Figure 1** – Pilot plant, schematic drawing. CL- CO<sub>2</sub> tank; CP-pump; BO- booster; TP-CO<sub>2</sub> tank; EX- extractor; SO- oil separator; SC- wax separator; VC- control valve; VR- back pressure valve; VA- needle valves; VE- ball valve; VM- regulation valve; MF- flow meter; BT-heat exchanger (hot water); BC- heat exchanger (cold water); TP- flow rate measurement; MN- manometer.

## Chemical analysis

The extracts were analyzed by gas chromatography-mass spectrometry (CG-MS) and flame ionization detector (FID). The identification was performed comparing their time retentions by Kovatz indexes with those of authentic compounds and from database collected from literature.

## RESULTS AND DISCUSSION

We studied two seasons of the year: fall and winter. The extractions performed in the winter had an interval of one year and those performed in the fall in the same year.

The objective of this study is to assess the difference in yield of extractions performed between the different seasons, taking into account the rainfall and solar intensity, and in the same way, comparing different seasons of the year.

Figure 2 shows the yield of supercritical extractions of essential oil of patchouli for plants harvested in the winter, with a year of difference.

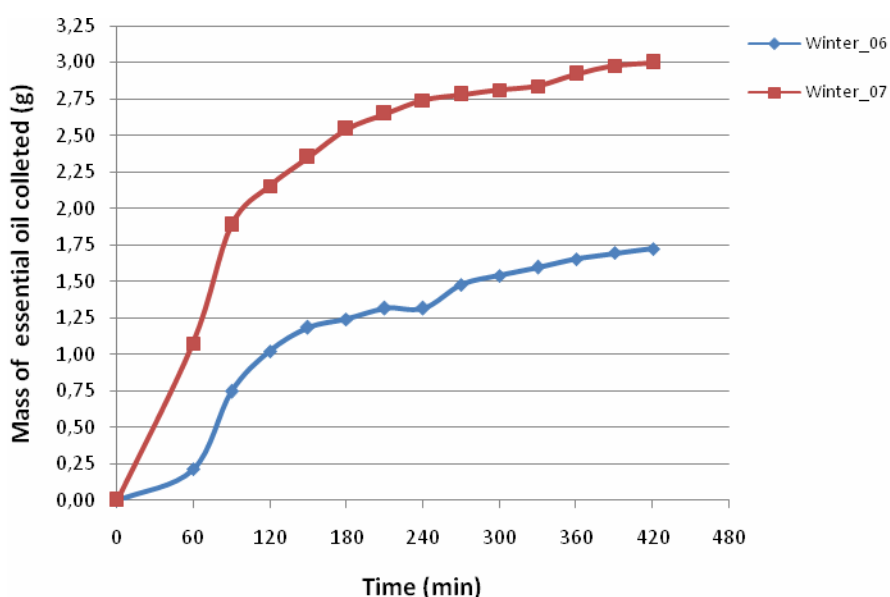
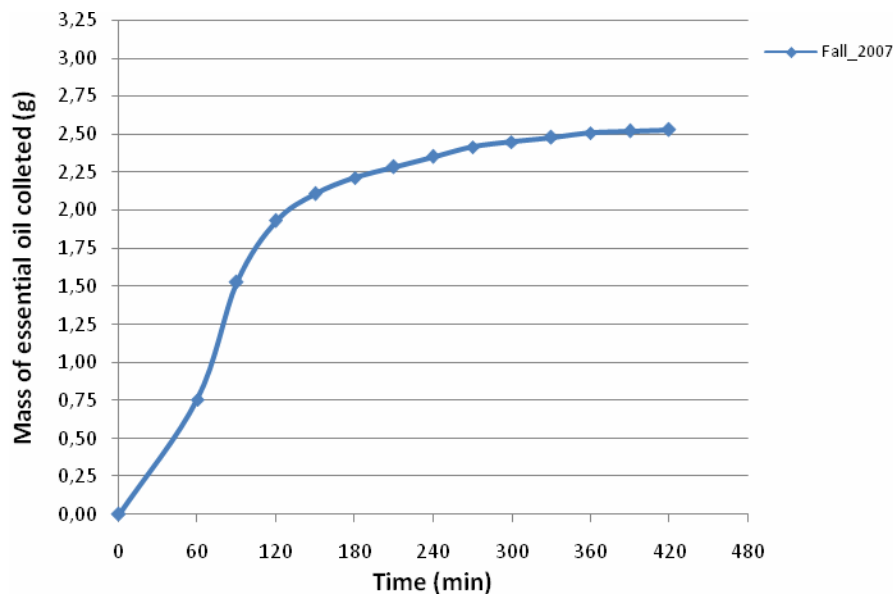


Figure 2 – Extraction curves for plants collected in winter.

The extraction regarding plants harvested in the 2007 winter produced the highest yield of process (5.94%, against 3.13% of the plants harvested in the 2006 winter). The plants began to receive irrigation at the end of 2006. In 2007 summer there was intense presence of sun and good distribution of rainfall, that can be one of indicatives for the yield increase.

The harvest period should be considered and, additionally, the distribution of rainfall and photo-period throughout the year helps in the development of the plant. The growth of patchouli is suppressed when there is lack of moisture for the plant, which leads to decrease the amount of essential oil to be extracted [2].

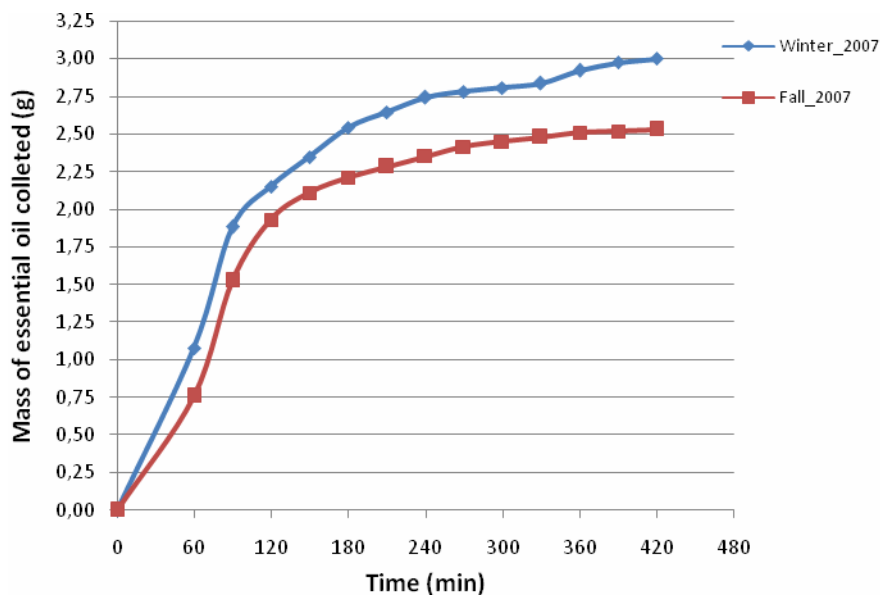
Extractions were performed with plants harvested in the fall, as shown in Figure 3.



**Figure3 – Extraction curves for plants collected in fall 2007.**

The extraction performed with plants collected in 2007 april obtained a high yield (5.06%). The level of irrigation depends on the amount of rain and sun period at the time of harvest, because a period of drought induces the use of irrigation to keep the quality of the plants. Other climate conditions can be affect the plant growth like the day length and maximal and minimum temperature.

A comparison of extractions carried out in winter and 2007 fall is showed in Figure 4.



**Figure 4 – Comparison between extractions carried out in fall and winter 2007**

Can be observed that winter is the optimal season to harvest and extract the patchouli essential oil. Because the climate conditions said previously and other parameters of process like size particle that need more studies. However, the curves behaviour is similar due Brazil climate.

Table 1 outlines a comparison between the seasons studied in terms of yield of the process.

**Table 1 - Comparisons data between the crops in different seasons.**

Season	Crop Date	Yield % (g essential oil/ g patchouli)
Winter	2006, September	3,13
Fall	2007, April	5,06
Winter	2007, July	5,94

GC-MS and FID analyses showed the presence of the main components responsible for the characteristic note of the essential oil of patchouli. *Patchoulol*,  *$\alpha$ -bulnesene*,  *$\alpha$ -patchoulene*,  *$\alpha$ -guaiene* appeared in both the seasons studied. Tables 2 and 3 show the amount and the time of retention of key compounds in the different seasons of the year studied.

**Table 2 – Key compounds of extraction carried out in 2007 winter.**

Peak	Retention time (min)	Compound	Percentage %
1	28,376	<i><math>\alpha</math>-guaiene</i>	17,009
2	29,365	<i><math>\alpha</math>-patchoulene</i>	5,066
3	31,095	<i><math>\alpha</math>-bulnesene</i>	18,007
4	37,577	<i>patchoulol</i>	28,400

**Table 3 – Key compounds of extraction carried out in 2007 fall.**

Peak	Retention time (min)	Compound	Percentage %
1	28,387	<i><math>\alpha</math>-guaiene</i>	17,408
2	29,360	<i><math>\alpha</math>-patchoulene</i>	5,390
3	31,101	<i><math>\alpha</math>-bulnesene</i>	19,394
4	37,574	<i>patchoulol</i>	25,892

Together with other components of the essential oil of patchouli, these compounds form the synergy of essential oil, giving it its characteristic scent. Analyses showed some similarity between the chemical composition of essential oils, but with differences in amount, which justifies the work of [2].

## CONCLUSION

The time of the year in which they extract the greatest amount of essential oil of patchouli is in the winter, with so climatic factors involved. However, further study on the levels of irrigation, condition of the plants exposure to the sun and other agronomic aspects are necessary. But, since Brazil a tropical country, there are no significant differences about the seasons, representing a good place to cultivate these plants and others.

The profiles of concentration of essential oils of patchouli analyzed showed significant data to know the ideal time of year to harvest the plants and extract the essential oil, indicating a change in the amount of each compound. The supercritical fluid extraction permits to extract the key compounds from raw material by the modulation of pressure and temperature. It is good to isolation of some compounds like the  *$\alpha$ -bulnesene*, important inhibitor on platelet-activating factor (PAF), combating diseases such as arteriosclerosis and thrombosis [5]. With the data harvest and a study about the ideal extraction time, the SFE will can be a technique of fractionation of which compound.

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