
STUDY OF ESSENTIAL OIL FROM GUACO LEAVES SUBMITTED TO DIFFERENT DRYING AIR TEMPERATURE

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ABSTRACT

This study aimed to evaluate the influence of drying air temperature on the content and chemical composition of essential oil extracted from guaco. Six drying treatments were used: ambient air and heated air at 40, 50, 60, 70 and 80° C. The essential oil contents, after drying, were compared with the values obtained from the fresh plant (control). The essential oil extraction was performed by hydrodistillation. From the results obtained, it could be concluded that the temperature of the drying air at 50° C provided the best results for the extraction of essential oil in guaco leaves, despite the fact that the chromatographic profile of the oil showed alterations in comparison with that obtained from the fresh plant.

Keywords: dryer, medicinal plants, *Mikania glomerata*.

RESUMO

ESTUDO DO ÓLEO ESSENCIAL DAS FOLHAS DE GUACO SUBMETIDAS A DIFERENTES TEMPERATURAS DO AR DE SECAGEM

Este estudo teve como objetivo avaliar a influência da temperatura do ar de secagem sobre o teor e composição química do óleo essencial extraído de guaco. Seis tratamentos de secagem foram utilizados: ar ambiente e ar aquecido a 40, 50, 60, 70 e 80° C. O conteúdo de óleo essencial, após secagem, foi comparado com os valores obtidos a partir da planta fresca (testemunha). A extração do óleo essencial foi realizada por hidrodestilação. A partir dos resultados obtidos, pode-se concluir que a temperatura do ar de secagem a 50° C proporcionaram os melhores resultados para a extração do óleo essencial em folhas de guaco, apesar de que os perfis cromatográficos do óleo mostraram alterações em comparação com o obtido da planta fresca.

Palavras-chave: *Mikania glomerata*, plantas medicinais, secador.

Recebido para publicação em 16/12/2009. Aprovado em 20/01/2010

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INTRODUCTION

Guaco (*Mikania glomerata* Sprengel), belonging to the compositae family, is a native plant of South America. It is a perennial, climbing plant with smooth wooden brown cylindrical branches, and green leaves with flat margins. Flowers grow in either round or oblong clusters along the stem and achene fruit are also found. Flowering does not occur in all regions of Brazil but is most common in the south. The plant is mainly used for medical purposes, especially as bronchodilators and is useful for treating respiratory tract diseases (flu, cough, snoring, bronchitis and asthma). It is also employed in rheumatism, neuralgia, febrifuge, depurative and cicatrizant (LOW et al., 1999).

Buggle et al. (1999) carried out the drying of lemon grass (*Cymbopogon citratus*) in an oven heated to 30, 50, 70 and 90 °C, until reaching constant weight, to evaluate the quantity and quality of essential oil. For the essential oil content, the best results were obtained when drying at 50 °C (1.43%), although the treatment at 30 °C (1.34%) showed little significant difference with the previous, but it was not indicated, because it favored fungus growth. Treatment at 70 and 90 °C (1.19 and 1.06% respectively) showed significant reduction in the essential oil content compared to the others treatments. However, variations of the Citral contents (main component) were not evaluated statistically. The authors concluded that the changes were small: 95.2, 90.6, 91.8 and 94.6% for the drying treatments of 30, 50, 70 and 90 °C, respectively.

Radünz et al. (2002a) used 5 temperatures (ambient air and heated air at 40, 50, 60 and 70 °C) for the drying of *Lippia sidoides* Cham, compared with the fresh plant, to evaluate the essential oil content. For the sample dried with environmental air a significant reduction of 8% in the essential oil content was observed, while drying at 40, 50, 60 and 70°C showed no significant differences between it and the fresh plant.

According to Radünz et al. (2002b), for the chemical analysis of the same treatments described above, no significant qualitative changes in the thymol percentage (main constituent) or to ρ -cymene compared to the fresh plant were found. However, significant increases in caryophyllene values were observed in which the drying air was heated to 50, 60 and 70 °C.

In a study carried out to determine the drying

type and fragmentation of *Cymbopogon citratus* leaves, to optimize the essential oil content 6 drying treatments were established including: 2 drying types (oven with forced ventilation at 40 °C and room temperature with dehumidifiers) and 3 sizes of dried leaves fragments (pulverized, 1 cm fragments and 20 cm long fragments). The authors concluded that the higher essential oil content was obtained with the samples dried at room temperature with dehumidifiers, with no significant differences for the leaves sizes. The most abundant component in the essential oil was citral, which also showed the highest concentrations in the dry leaves in the dehumidifiers (COSTA, 2005).

Radünz et al. (2006) used common-mint (*Mentha x villosa* Huds), drying it on a fixed-bed dryer with ambient air and air heated to 40, 50, 60, 70 and 80 °C and evaluated the essential oil content extracted after drying with that extracted from the fresh plant. Based on the results, it was concluded that the higher content was obtained when the drying process was done with drying air at 50 °C.

Barbosa et al. (2006) submitted *Lippia alba* leaves to 6 drying treatments, being: ambient air (25 °C) and air heated to 40, 50, 60, 70 and 80 °C, and comparing with fresh leaves (control). They found that the citral level presented a significant increase when the leaves were submitted to drying, independent of treatment, compared to the fresh plant. It was considered that this increase could be attributed to the oxidation of geraniol during drying, converting it into geranial. It was also observed that the nerol content did not differ significantly between drying treatments, but statistically showed a significant decrease when compared with the fresh plant. This decrease was attributed to the oxidation of nerol during drying, which was transformed into neral. Considering that citral is the main chemical constituent of interest in the oil from this plant, it was concluded that drying for marketing purposes can be carried out using heated air from 40 to 80 °C.

To evaluate the influence of the drying air temperature on *Ocimum selloi* Benth essential oil content and composition, David et al. (2006) observed that the essential oil content did not show significant variation. The main components were elimicin (69.8%), **trans-caryophyllene (6.0%)**, germacrene D (3.7%) and bicyclogermacrene (3.5%), and they found that the temperature increase caused reduction of the components; concluding that a drying air temperature of 40 °C is the most

appropriate for the species.

Soares et al. (2007) studied the influence of 4 drying air temperatures (40, 50, 60 and 70 °C), in thin layers, and 2 air velocities (0.9 and 1.9 m s⁻¹) on the essential oil content and brazilian linalool (*Ocimum basilicum* L). The higher essential oil contents were obtained in the drying process with an air temperature at 40 °C and air velocity of 1.9 m s⁻¹. The highest linalool contents were obtained with drying air temperature from 50 to 60 °C and an air velocity of 1.9 m s⁻¹. They concluded that the essential oil chemical composition of *Ocimum basilicum* L was affected by both temperature and air velocity during drying.

Considering the facts above, the objective of this work was to determine drying's effect on guaco with natural air and heated air at 40, 50, 60, 70 and 80 °C, on essential oil content and on its chemical components.

MATERIALS AND METHODS

The study was conducted at the Universidade Federal de Viçosa (UFV) - Brazil. To conduct the experiment, a randomized block statistical design was used, with three repetitions. In each block, all drying treatments were tested as well as the extraction of essential oil, aiming to minimize the effect of storage on guaco after drying. The treatments consisted of six drying air temperatures, including drying with natural air (ambient) and air heated to 40, 50, 60, 70 and 80 °C. Drying with natural air was done in the same manner of dryer of the other treatments, but with the heating system off.

The statistical evaluations of the essential oil content, obtained from the dried guaco and the fresh leaves (control), as well the constituents in the essential oil, were carried out using the analysis of variance, and when necessary, the multiple averages comparison test - Duncan to 5% probability – using the program for statistical analysis, SAEG (2007).

The plants were harvested between 7:00 and 8:30 am and the material was forwarded immediately to the laboratory for selection, moisture content determination and subsequent drying. Selection included the removal of plant parts that: were in the decomposition stage (advanced ripening), contained pests or diseases, as well as other plants or parts of those which were present then the remaining sample was homogenized.

The moisture content was determined by the

gravimetric method, methodology recommended by the Asae Standards (ASAE, 2000), using 25 g, with 3 repetitions, in an oven with a temperature of 103 ± 2 °C for 24 hours.

A fixed-bed dryer with upward air flow was used, equipped with a liquefied petroleum gas burner for heating the drying air, as described by Radünz (2004).

The whole leaves were dried using only 2 of the dryer trays, without completely filling the tray to give a total weigh of 0.5 kg per treatment. The reason for not completely filling the trays was due to the amount of plant samples available.

The control of drying air temperature was done with an automatic controller, with a variation of ± 2 °C, as described by Jesuz et al. (2001). Temperature data was taken with the use of thermocouples, previously calibrated and placed in pre-set points of the dryer and coupled to an automatic data acquisition system (ADAS) that registered their values in a microcomputer. The drying air velocity was tracked by an anemometer.

After drying, the samples were packed in polyethylene bags (40 µm) and stored in refrigerated chamber at 5 °C, until being submitted to extraction of the chemical components.

The essential oil was extracted by hydrodistillation using a Clevenger device, adapted to a round-bottomed two liter flask as described by Skrubis (1982) and Ming et al. (1996), with heating maintained at the minimum temperature required to boil. The flask was loaded with 100 g of fresh sample or the equivalent of 100 g for the previously homogenized dry samples. Distilled water was then added with a volume sufficient to cover the material, beginning the hydrodistillation process. Extraction time was 6 hours, which was previously calibrated in preliminary tests, and three repetitions were performed for each treatment. At 1 hour intervals after the beginning of process, hydrolat samples (mixture of water and oil) were taken and all were grouped in a single sample.

After the hydrolat was obtained, it was separated with pentane (10:1) in a 125 ml separation funnel and the procedure was repeated 3 times. The organic fraction (pentane and essential oil) obtained was transferred to a 125 ml Erlenmeyer flask and treated with excessive anhydrous magnesium sulfate. After a few minutes at rest in order to remove the remaining water, the solution was filtered directly to a flask of 125 ml and concentrated in a rotary evaporator at 38 °C and

the oil obtained was transferred into a 5 mL bottle to be properly weighed. The flask was washed 3 times with small amount of solvent, and an aliquot was added to the sample in the bottle. The sample was then subjected to a water-bath at 38 °C for about 1 hour to evaporate the residual solvent.

Quantification of essential oil was performed by weighing the samples on an analytical balance with an accuracy of 0.0001 grams. After weighing, the bottles were closed, sealed with parafilm and wrapped with aluminum paper to protect from light and then stored at 5 °C until the time of the chromatographic analysis. The essential oil contents, obtained from drying treatments, were compared with those from the fresh sample, and calculated according to Venskutonis (1997), based on dry matter.

Identification of the essential oil components was performed using gas chromatography associated with mass spectrometry (GC-MS), with the help of the library present in this equipment. The chromatograph used was a Shimadzu, GC-17A gas chromatograph and a GCMS-5050A QP mass spectrometer and with DB 5 type column. Therefore, for the quantification of essential oil components the equipment model used was the gas chromatographer Shimadzu GC-17A coupled to a flame ionization detector (GC-FID). The methodology used to identify the components of the essential oils is that described by Adams (1995). The sample volume injected was 1 µL at a concentration of 10.000 ppm and using previously distilled hexane as a solvent. For the identification of the essential oil components, in addition to the mass spectra suggested by the library present in the equipment, the Kovats index was calculated (ADAMS, 1995).

To quantify each constituent in the essential oil, integration of the areas generated calculated by the program itself in this computer connected to the GC-FID, with results expressed as percentage proportions of area. The component levels of the essential oil, obtained from drying treatments were compared with those from the fresh sample.

RESULTS AND DISCUSSIONS

The average temperature of ambient drying air was 21.5 °C and the mean drying air velocity was 0.473 m s⁻¹. The average moisture content of fresh leaves was 82.34% and the final moisture content of the guaco leaves after drying was 13.9, 9.63, 8.70, 6.70, 7.55 and 7.75%, respectively, for drying with natural air and heated to 40, 50, 60, 70 and 80 °C.

The essential oil content obtained from fresh guaco leaves cultivated at the UFV was 0.49% db (mass/mass). Considering the fresh plant's moisture content of 82.34% wb, and the mean specific mass of essential oils, which according to Simões & Spitzer (2003) are between 0.69 and 1.118 g mL⁻¹, the approximate value to compare with Oliveira et al. (1999), is 0.078% wb (mass/volume). Meanwhile, Oliveira et al. (1999) obtained 0.26% wb (mass/volume). This variation can be attributed to the harvest time, because normally medicinal species have the highest concentration of active principles during certain periods of the year or development stage. Oliveira et al. (1999) harvested during the flowering period, different from the technique used at the Universidade Federal de Viçosa. The highest concentrations of essential oils can also be attributed to genetic factors, climatic factors such as temperature, photoperiod, soil type as well as extraction techniques, among other factors.

As shown in Table 1, the harvest season characterized by 3 blocks, significantly influenced the essential oil content extracted from the guaco leaves. Normally, the medicinal species have the highest concentration of active principles during certain periods of the year, and this increase over the collection dates may be due to the approximation of the optimal harvest period.

Table 2 shows the results of the statistical analysis of the essential oil content from guaco obtained for the drying treatments. It was observed that the dry processing with heated air heated positively influenced the essential oil content of guaco, contrary to leaves dried with ambient air. According to Oliveira et al. (1999), guaco's essential oil is produced mainly inside the secretory pockets or channels as a mixture with resin. Increase in the extractive efficiency of essential oil from the leaves submitted to drying with heated air can be a function of the location of its essential oil inside specific cells which have special structures with lignified cell walls and are located in the leaves parenchymatous tissue, not in fragile structures, such as glandular hairs, trichomes and epidermais glands.

The highest essential oil content was achieved using drying air heated to 50 °C, in accordance with Buggle et al. (1999) whom, despite having used lemon grass (*Cymbopogon citratus* (DC.) Stapf.), found the highest essential oil extraction efficiency when temperature was 50 °C. The authors believe that the use of high temperatures promotes the

decrease of essential oil due to volatilization, and low temperatures are not appropriate because they favor the emergence of fungi, and that the same may have occurred with the guaco.

The results may also be in accordance with Rocha *et al.* (2000) who obtained the highest essential oil content of citronela (*Cymbopogon winterianus* Jowitt) when drying at 60 °C. They concluded that this temperature may possibly have interfered in permeability or mechanical physical resistance of the plant tissues, helping to retain the volatile compounds in their original structures. In guaco, since oil is located in specific oil cells which have special structures with lignified cell wall, the drying air temperature of 50°C may have facilitated the essential oil extraction, without causing its volatilization during the drying process and. Greater temperatures may have therefore caused volatilization resulting in a direct reduction of essential oil content extracted from the leaves.

The lowest essential oil content obtained from the dried guaco was with ambient air and may be due to the long drying time, favoring fungi development and enzymatic activity and promoting the oxidative process.

The main components of the guaco's essential oil are shown in Table 3. The values found show small differences in relation to the constituents described by Oliveira (1999). These variations are probably according to those described by Johnston & Spitzer (2003), who affirmed that the chemical composition of a volatile oil, extracted from the same organ of the same plant species can vary significantly according to the harvest time, development stage, climate and soil.

The two main components of essential oil from guaco are germacrene D and bicyclogermacrene, probably because they present the highest volatilization temperatures and suffered no volatilization during the drying process. These two components showed relative increase in their contents with the increase in drying air temperature compared with the fresh plant, such that their highest contents were obtained using air heated to 80 °C.

β -caryophyllene presented no significant difference between drying treatment, however results were better than those obtained with the fresh leaves.

α -Pinene, mirceno and β -pinene presented their highest contents in the fresh plant, showing reduction in their concentrations with the increase of drying temperature, indicating possible losses due to volatilization during drying.

CONCLUSIONS

Under the conditions which the study was conducted, it was concluded that:

- The drying temperature of 50 °C provided the best results for essential oil content in guaco leaves.
- The chromatographic profile of the essential oil presented changes due to the drying treatment, when compared with the fresh plant;

ACKNOWLEDGMENTS

Sincere thanks are due to FAPEMIG, CAPES and CNPq, for their support during this work.

Table 1. Essential oil content in guaco leaves obtained, according to the harvest season

Harvest season	Essential oil content (d.b)
20 e 21 de may	0.6900 a
03 e 04 de may	0.5814 b
21 e 22 de april	0.4829 c

CV= 7.44%

Table 2. Essential oil content in guaco leaves subjected to different drying air temperatures, compared with the fresh plant (control)

Drying air temperature (°C)	Essential oil content (d.b)
50	0.7367 a
70	0.6500 b
40	0.6400 bc
60	0.6000 bc
80	0.5600 cd
Sem secagem	0.4933 d
Ar ambiente	0.4133 e

CV= 7.44%

Table 3. Statistical analysis results of guaco's essential oil constituents compared with the fresh plant, expressed as a percentage proportion of area

Table 3.1- Germacrene D		Table 3.2- Bicyclogermacrene		Table 3.3- β - caryophyllene	
Treatment	Mean	Treatment	Mean	Treatment	Mean
80 °C	42.69 a	80 °C	19.38 a	70 °C	10.74 a
60 °C	41.72 ab	70 °C	18.90 a	80 °C	10.67 a
70 °C	41.21 ab	60 °C	18.68 a	60 °C	10.11 ab
50 °C	39.86 bc	40 °C	18.39 a	ambient	9.50 ab
40 °C	38.60 c	50 °C	18.21 a	50 °C	9.47 ab
control	32.51 d	control	17.85 a	40 °C	9.44 ab
ambient	15.34 e	ambient	9.65 b	control	8.75 b
CV= 3.84		CV= 7.15		CV= 8.02	

Table 3.4- α -pineno		Table 3.5- Mirceno		Table 3.6- β -pineno	
Treatment	Mean	Treatment	Mean	Treatment	Mean
control	5.23 a	control	3.80 a	control	3.54 a
40 °C	4.31 ab	50 °C	3.08 ab	40 °C	2.21 b
50 °C	3.59 bc	40 °C	2.98 abc	50 °C	1.90 c
60 °C	3.00 cd	60 °C	2.57 bc	60 °C	1.59 d
ambient	2.67 cd	80 °C	2.29 bc	ambient	1.53 de
80 °C	2.23 d	70 °C	2.05 c	70 °C	1.34 de
70 °C	2.19 d	ambient	1.07 d	80 °C	1.29 e
CV= 17.64		CV= 19.88		CV= 7.50	

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