NOTA TÉCNICA:

EFFECT OF DRYING AIR TEMPERATURE ON THE QUALITY OF ESSENTIAL OIL FROM *MIKANIA GLOMERATA*

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ABSTRACT

This study aimed to evaluate the influence of drying air temperature on the content of essential oil extracted from guaco. The drying tests were performed in an experimental fixed-bed dryer, which contains 4 perforated trays with upward air flow. Treatments consisted of five temperatures for the drying air (ambient air and air heated to 40, 55, 70 and 85ºC), with three repetition, using 850 g of fresh guaco leaves. Guaco leaves were dried until reaching a final moisture content of approximately 10% w.b. The drying air velocity was 0.5 m s^{-1}. Essential oil extraction was performed by hydrodestilation, using Clevenger equipment. Results of the essential oil content showed that the drying air temperatures from 55 to 85ºC did not statistically influence the essential oil content. The chromatographic profile of the essential oil presented changes as a function of the drying treatment when compared with the *in natura* plant (control).

Keywords: dryer, guaco, medicinal plants

RESUMO

EFEITO DA TEMPERATURA DO AR DE SECAGEM SOBRE A QUALIDADE DO ÓLEO ESSENCIAL DE *MIKANIA GLOMERATA*

Este estudo objetivou avaliar a influência da temperatura do ar de secagem sobre o teor de óleo essencial extraído de guaco. Os testes de secagem foram realizados em um secador de leito fixo, o qual contém 4 bandejas perfuradas com fluxo de ar ascendente. Os tratamentos consistiram de cinco temperaturas do ar de secagem (ar ambiente e ar aquecido a 40, 55, 70 e 85ºC), com três repetições, utilizando-se 850 g de folhas frescas de guaco. As folhas de guaco foram secas até atingir um teor de água final de, aproximadamente, 10% b.u. A velocidade do ar de secagem foi de 0,5 m s^{-1}. A extração do óleo essencial foi realizada por hidrodestilação, usando o equipamento Clevenger. Os resultados do teor de óleo essencial mostraram que as temperaturas do ar de secagem de 55 até 85ºC não influenciaram estatisticamente a quantidade de óleo essencial extraído. O perfil cromatográfico do óleo essencial apresentou mudanças em função dos tratamentos de secagem, quando comparados com a planta in natura (testemunha).

Palavras-chave: secador, guaco, plantas medicinais.

Recebido para publicação em 01/09/2011. Aprovado em 30/12/2013.

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INTRODUCTION

Currently, global interest in phytopharmaceutical products has increased, even among pharmaceutical companies, and some new policies focused on herbal products are emerging, promoting the use, standardization and development of reliable analytical methods of quality control to support their safety and efficacy. The commercialization of medicinal plants and phytotherapy products is expanding around the world due to factors such as the high cost of industrialized medicines, the lack of access to chemotherapy products by a large portion of the population and the tendency of consumers to preferentially use organic products (LOW *et al*., 1999).

Mikania glomerata Sprengel (Compositae) is popularly known in Brazil as "guaco". 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. Flowering does not occur in all regions of Brazil but is most common in the south (LOW *et al*., 1999). It is used in folk medicine for the treatment of fever, rheumatism, and illnesses of the respiratory tract (VILEGAS *et al*., 1997). Pharmacological studies have confirmed the anti-inflammatory properties of the crude extract of the guaco, while chemical studies point to coumarins and ent-kaurenoic acid as the main constituents of this species (VIEIRA *et al*., 2002).

Kinetics of the drying process can define the final quality properties of the dried material. Artificial drying has been one of the most important processes in pre-processing of agricultural products, seeking to achieve the phytotherapy product requirements of the pharmaceutical industry, which does not have structure to use fresh plants in the quantities required for industrial production (LORENZI; MATOS, 2002).

The post-harvesting process of medicinal plants has great importance in the production chain, because of its direct influence on the quality and quantity of active principles in the product sold (SILVA; CASALI, 2000). For this reason adequate dryers are needed, which utilize temperature, velocity and humidity values for drying air that result in a rapid reduction in the moisture content without affecting the quality of the active principles of medicinal plants. The drying process may also contribute to regular supply and facilitate the marketing of plants, because it facilitates transport and storage (CASTRO; FERREIRA, 2001).

Changes in the concentrations of volatile compounds during drying depend on several factors, such as the drying method and drying air temperature. Sefidkon *et al*. (2006) studied the effects of three different drying methods (sun-drying, shade-drying and oven-drying at 45°C) on content and chemical composition of the essential oil of *Satureja hortensis*. They concluded that drying of *S. hortensis* in an oven at 45°C is most suitable and is recommended for fast drying and high oil content, as well as for a high percentage of carvacrol.

Lippia alba leaves, studied by Barbosa *et al.* (2006), were submitted to 6 drying treatments (ambient air and air heated to 40, 50, 60, 70, and 80°C), and its essential oil was compared with that obtained from fresh leaves (control). They found that the citral level presented a significant increase when the leaves were submitted to drying, independent of the treatment, compared to the fresh plant. Considering that citral is the main chemical constituent of interest in the essential oil from this plant, it was concluded that drying for marketing purposes can be carried out using air heated from 40 to 80°C. Radünz *et al*. (2006) evaluated the influence of the same drying air treatments above (ambient air and air heated to 40, 50, 60, 70, and 80°C) on the essential oil content of *Mentha x villosa* Huds. They concluded that the highest content of essential oil of *M. x villosa* Huds was obtained at 50°C when compared with the fresh plant.

Ennajar *et al*. (2010) used sun-drying, shade-drying and oven-drying at 45ºC to study the effects on yield and chemical composition of *Juniperus phoenicea* L. essential oils. The authors concluded that drying of berries of *J. phoenicea* in an oven is more suitable and is recommended for obtaining higher essential oil yields; for higher percentages of some special components, however, such as α-pinene and δ-3-carene, shade-drying was more suitable.

As described above, the drying process great importance in improving the quantity and quality of essential oil from aromatic and medicinal plants. This study sought to evaluate the influence of drying air temperature on the quality of essential oils extracted from *M. glomerata*.

MATERIAL AND METHODS

Fresh guaco plants (*M. glomerata*) were grown in the experimental area of the Crop Science Department at the Universidade Federal de Viçosa. The city of Viçosa is located 648 m above sea level, at latitude 20°45'14" south and longitude 42º52'55" west. *M. glomerata* plants were properly identified. The *M. glomerata* plants were approximately 4 years old and were cultivated with organic fertilizer. Before drying, samples were selected and homogenized by removing of plant parts that were in decomposition stage (advanced ripening) and others containing pests or diseases. Samples were then immediately sent to the laboratory for moisture content determination and cooled in a refrigerated chamber at 5°C for subsequent drying.

The moisture content was determined using the gravimetric method recommended by ASAE Standards (2000) for forage and similar plants. Samples of 25 g were applied, in triplicate, to an oven with forced air circulation at 103 ± 2°C for 24 h.

Drying tests were carrying out using a fixed-bed dryer, which had 4 perforated trays and upward air flow. The treatments consisted of 5 drying air temperatures (ambient, 40, 55, 70, and 85°C), with 3 repetitions, using 850 g of fresh leaves in the top tray. The reason for not completely filling all the trays was due to the amount of plant samples available. *M. glomerata* was dried to the moisture content equivalent of 10% w.b. The drying air velocity was 0.5 m s^{-1}, measured with an Impac anemometer, model IP-720.

The weight of the dried guaco leaves, M_f, was calculated using equation 1:

$$M_f = M_i\left(\frac{100 - W_i}{100 - W_f}\right) \quad (1)$$

where,

Mf is the final mass after dried, Kg;
M_i is the initial mass of product to be dried, Kg;
W_i is the initial moisture content, % w.b.; and
W_f is the final moisture content, % w.b.

The initial moisture content was previously measured by the gravimetric method. Trays with the samples were weighed on a digital scale every 10 min during the first hour, every 20 min for the second hour and every 30 min for the remaining time of the drying process. The drying treatment was interrupted when the final mass (M_f) was achieved.

Control of drying air temperature was performed using an automatic controller, with variation of ± 2°C, as described by JESUS *et al*. (2001). Temperature data was monitored with the use of thermocouples, previously calibrated and placed at pre-determined points (outside the dryer, in the bottom tray, in the exhaust output and on the top tray) of the dryer. An automatic data acquisition system (ADAS) was used to register the temperature values in a microcomputer. Relative humidity was monitored in the bottom tray and in the exhaust air using previously calibrated T type thermocouples. The values of temperature and relative humidity were measured at 10 minute intervals.

After drying, the samples were packaged in polyethylene bags (40 μm) and stored in a refrigerated chamber at 5ºC, until being submitted to essential oil extraction.

Quantitative analyses were performed at the laboratory of analysis and synthesis of Agrochemicals –LASA at the Universidade Federal de Viçosa. Essential oil was extracted by hydrodistillation with clevenger equipment, adapted with a round-bottomed two liter flask as described by Skrubis (1982) and Ming *et al*. (1996). Heating was maintained at the minimum temperature required for boiling. Flasks was loaded with samples of 20 and 90 g of dried and fresh guaco leaves, respectively. The extraction time was 3 hours, which was previously calibrated in preliminary tests, and three repetitions were performed for each treatment. After the hydrolat (mixture of water plus oil) was obtained, it was separated with pentane (3 x 30 mL) in a separation funnel. The organic fraction obtained was treated with excess anhydrous magnesium sulfate . After a few minutes at rest, the solution was filtered and concentrated in a rotary evaporator at 40°C and the essential oil obtained was transferred into a 5 mL bottle. Quantification of essential oils was performed by weighing the samples on an analytical balance with an accuracy of 0.0001

grams. The essential oil contents, obtained from the drying treatments, were compared with those from the fresh sample, and calculated according to Venskutonis (1997), based on dry matter.

The main component of the essential oil of guaco was identified using a gas chromatograph system coupled to a mass spectrometer (Shimadzu GC-EM, GC-14A/QP-5000) and equipped with a capillary column DB-5 (30 m x 0.25 mm (ID) x 0.25 μm film). Helium was used as the carrier gas at a flow rate of 1.0 mL/min. The temperature of the injector was 220 °C and the temperature of the detector was 240 °C. The initial temperature in the oven was maintained at 60 °C for 2 min, and was increased at a rate of 3 ºC per minute until reaching 240 °C. This temperature was maintained for over 30 min. Only ions at charge mass (m/z) ratios between 29 and 600 were detected by the mass spectrometer.

The sample volume injected was 1 μL, at a concentration of 10000 ppm, with hexane as a solvent. Identification of components was conducted by comparing mass spectrometer readings obtained from the equipment database and the Kovats retention index for each component, as shown in Equation 2 (ADAMS, 1995).

$$IK = 100NC + 100\left(\frac{\text{Log } t'_{RX} - \text{Log } t'_{RZ}}{\text{Log } t'_{R(Z+1)} - \text{Log } t'_{RZ}}\right) \quad (2)$$

where,

IK: Kovats index;
NC: number of carbons from the hydrocarbon immediately before the evaluated component;
t′RX: retention time of the evaluated component;
t′RZ: retention time of the hydrocarbon immediately before the evaluated component; and
t′R(Z+1): retention time of the hydrocarbon immediately after the evaluated component.

In order to obtain the hydrocarbon standard curve to calculate the Kovats index, a solution of hydrocarbons was prepared, varying from hexane to tetracosane. Two milligrams of each hydrocarbon were weighed in the same flask in order to prepare the solution. The final mass was solubilized in 2 mL of hexane, producing a 1000 ppm solution in relation to each hydrocarbon. This solution was analyzed using a gas chromatograph, coupled with a mass spectrometer, with the same operational conditions used for the essential oil samples.

To obtain the quantity of the main components, a Shimadzu GC-17A gas chromatograph equipped with a flame ionization detector (FID) and a smelt silica capillar column with a DB-5 (30 m x 0.25 mm (ID) x 0.25 μm film) was employed. Nitrogen was used as the purging gas with a flow rate of 1.33 mL/min. The initial temperature of the column was kept at 60°C for 2 min, and programmed to increase at 3°C per minute, until reaching the maximum temperature of 240°C, where it was maintained for 61 min for analysis. The split ratio was 1:10 and the solvent cut period was 5 min. Injection and detection temperatures were fixed at 250 °C. The sample injection volume was 1 μL, at a concentration of 2000 ppm, using hexane as a solvent. Components were quantified based on comparison of the compound retention times, which were similar in both techniques. The normalization method was used, where the value of the total peak areas is considered 100% and the percentage of each component was calculated using the area of each peak.

To conduct the experiment, a completely randomized design was used, with three repetitions. Statistical evaluations of the content and components of the essential oil, obtained from the dried guaco and the *in natura* plant (control), were carried out using the analysis of variance, and when necessary, the multiple averages comparison test - Duncan with 5% probability – using the program for statistical analysis, SAEG (2007).

RESULTS AND DISCUSSION

Figure 1 shows the results of the essential oil content of *M. glomerata* obtained for the drying treatments with ambient air and air heated to 40, 55, 70, and 85ºC, compared with values for the *in nature* plant (control).

A trend of essential oil reduction is observed in all drying treatments compared to the fresh plant (control). These results agree with those found by Figiel *et al*. (2010) and Szumny *et al*. (2010) when studied the influence of the drying methods on volatile compounds of *Origanum vulgare* and *Rosmarinus officinalis*, respectively. The drying methods tested were convective (CD) at 60°C and vacuum-microwave (VMD), as well as a combination of convective pre-drying and VM finish-drying (CPD–VMFD). The drying method had significant effects on the quality of the final

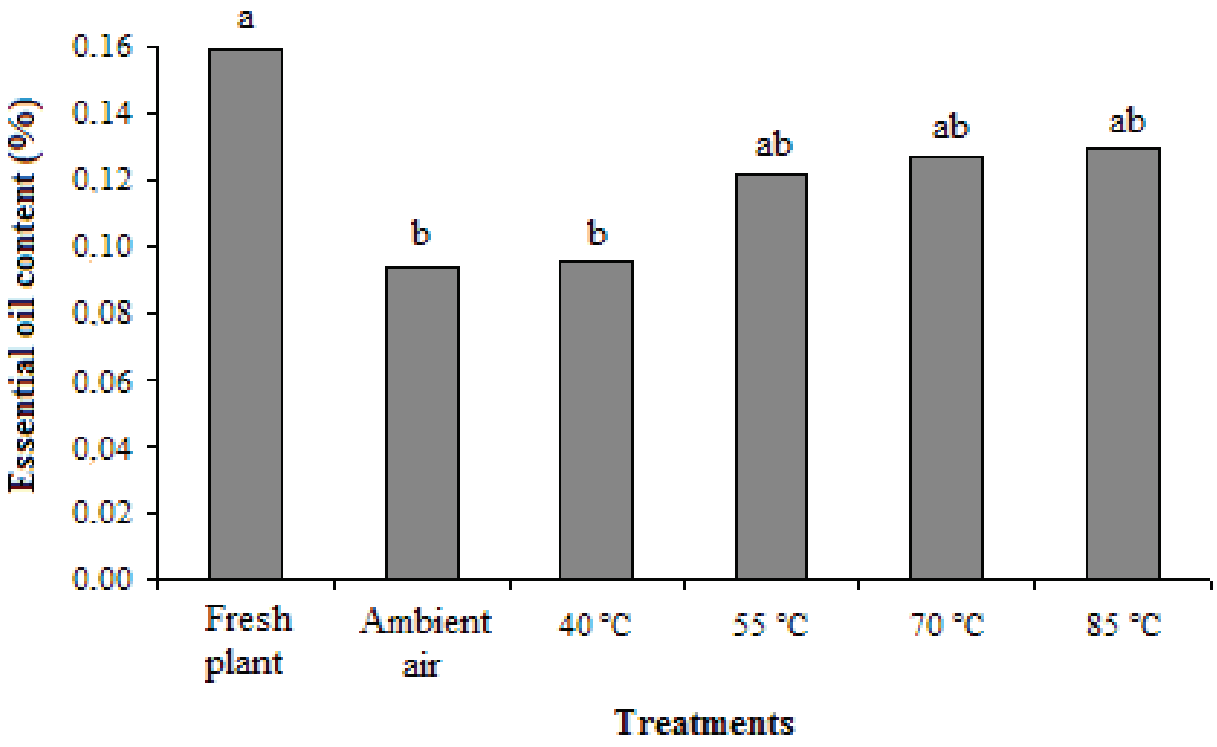

Figure 1. Essential oil content, on a dry basis, extracted from guaco leaves subjected to the different drying treatments (CV = 8.46%).

dried samples. Use of hot air in any part of the drying process of fresh *O. vulgare* and *R. officinalis* caused important losses of essential oil compounds and consequently a significant reduction of the quality of dried product compared to the fresh plant.

The drying treatment with ambient air and air heated to 40°C were statistically different from the fresh plant (control), negatively affecting the essential oil content extracted from guaco. This may be due to the long drying time in the treatments, favoring fungi development and enzymatic activity and promoting the oxidative process. However, treatments with drying air heated to 55, 70 and 85°C were statistically equal to the control (Figure 2). According to Oliveira *et al*. (1999), essential oil of guaco is produced mainly inside the glandule secretors or channels as a mixture with resin. Increase in the extractive efficiency of essential oil from leaves submitted to drying with heated air may 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 parenchymatous tissue of the leaves, and not in fragile structures such as glandular hairs, trichomes and epidermais glands.

Results may also be in accordance with Rocha *et al*. (2000) who obtained the highest essential oil content of *Cymbopogon winterianus* Jowitt when dried with high temperature (60°C). They concluded that the high 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, because oil is located in specific oil cells which have special structures with lignified cell wall, the drying air temperatures of 55, 70, 85°C may have facilitated essential oil extraction, without causing its volatilization during the drying process.

The main components of guaco essential oil are shown in Table 1. The values found show small differences in relation to the constituents described by Oliveira (1999). These variations are probably in accordance with those described by Johnston and 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.

Table 1. Statistical analysis results of germacrene and bicyclogemacrene compared with the *in natura* plant (control), expressed as a percentage of area.

Germacrene D		Bicyclogermacrene	
Treatment	Mean	Treatment	Mean
85 ºC	42.69 a	85 ºC	19.38 a
70 ºC	41.21 ab	70 ºC	18.90 a
55 ºC	39.86 bc	40 ºC	18.39 a
40 ºC	38.60 c	55 ºC	18.21 a
in natura (control)	32.51 d	in natura (control)	17.85 a
ambient	15.34 e	ambient	9.65 b
CV= 3.84		CV= 7.15	

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 presented a relative increase in their contents with the increase in drying air temperature compared with the fresh plant, such that the highest contents were obtained using air heated to 85°C.

CONCLUSIONS

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

- The drying air temperatures of 55, 70, and 85ºC provide the best results of essential oil content extracted from guaco leaves.The drying air temperatures of 70 and 85ºC provide the best results of gemacrene content extracted from guaco leaves.
- Only drying with ambient air reduced the bicyclogermacrene content from *M. glomerata* when compared with the other treatments

ACKNOWLEDGMENTS

The authors thank FAPEMIG and CNPq for the financial support.

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