EFFECT OF DRYING TEMPERATURE ON YIELD AND PHYTOCHEMICAL QUALITY OF ESSENTIAL OIL EXTRACTED FROM *SCHINUS TEREBINTHIFOLIUS*

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ABSTRACT – Among the post-harvest stages processing of medicinal and aromatic plants, drying stands out as a critical and essential step for the bioactive compounds conservation. Schinus terebinthifolius is a very important species due to its medicinal properties and food industry uses. This work aimed to evaluate the drying temperature effect on S. terebinthifolius essential oil yield and phytochemical quality. The ripe fruits were harvested and dried in a forced air oven. The essential oils were extracted in Clevenger apparatus. Chemical constituents were identified by gas chromatography coupled to the mass spectrophotometer. There was no significant variation in the essential oils yield and phytochemical quality independent of drying temperature. Considering that the drying temperatures did not influence the yield and the phytochemical quality of the essential oil, these results may contribute to the optimization of the post-harvest drying process of S. terebinthifolius fruits.

Keywords: pink pepper, post-harvest, volatile compounds.

1. INTRODUCTION

Schinus terebinthifolius Raddi is a tree species, perennial, native to Brazil, commonly known as pink pepper, red pepper and peppermint (Carmello-Guerreiro & Paoli, 1999; Carvalho et al., 2013). In terms of its geographic distribution, its occurrence is registered in the Southeast and South regions of the country, mainly in the Cerrado, Atlantic Forest and Pampa (Reflora, 2020). In Brazil, a considerable part of its production is still from extractive exploration and has a great socioeconomic importance, as it is one of the main sources of income for family farmers (Gomes et al., 2013; Ventura et al., 2018). The city of São Mateus, state of Espírito Santo, stands out as the largest producer and exporter of pink pepper in the world (Gilbert & Favoreto, 2011; Ventura et al., 2018). In the industry, *S. terebinthifolius* Raddi is underused, mainly due to the lack of uniformity of the commercialized fruits and the quality control of the processing conditions (Ventura et al., 2018). Much of the production is destined to the international market, especially for culinary use, due to its sweet taste, discreet pungency and pleasant aroma (Carvalho, 2003; Bertoldi et al., 2006; Ventura et al., 2018).

Its medicinal importance is associated with activities such as antiulcerogenic (Carvalho et al., 2013), anti-inflammatory (Formagio et al., 2011), antibacterial (Dourado, 2012; Machado & Valentini, 2014; Gomes et al., 2020); antioxidant (Bertoldi, 2006; Píccolo et al., 2018), antitumor (Matsuo et al., 2011) and antifungal (Freires et al., 2011). For this reason, *S. terebinthifolius* was included

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among the 71 medicinal species listed in the National List of Plants Medicinal Products of Interest to the Unified Health System - RENISUS (Brasil, 2009).

Although Brazil has a rich biodiversity, it is known that there are still barriers in the productive chain of medicinal plants, among them are extractivism, irregular supply, safety and quality of the plant raw material (Barata, 2005; Ventura et al., 2018). Therefore, the development and application of suitable technologies for production and postharvest for *S. terebinthifolius* is extremely important for the conservation of its active compounds and, consequently, to guarantee the therapeutic effectiveness of this medicinal species and its flavor and aroma to food industry. In this context, in 2008, the National Program for Medicinal Plants and Phytotherapic was implemented in Brazil, in which the guidelines for the development of the production chain for medicinal plants and phytotherapic are described (Brasil, 2008; Rodrigues, 2008).

Essential oils are among the secondary metabolites extracted from medicinal plants, including S. terebinthifolius. Its use is increasing due its broad application, like in the production of medicines, cosmetics, perfumes, food and agriculture (Gilbert & Favoreto, 2011). Several factors can influence the composition and the content of the chemical constituents of essential oils, especially the cultivation (genotype, stage of plant development, solar radiation, altitude and water and nutrient availability, genetic factors, season, time of harvest, type of soil as well as the method and temperature of drying the plant material and storage (Ventura et al., 2018). Among these factors, the drying temperature constitutes a critical point of control of the post-harvest processing of medicinal and aromatic species considering that it can significantly affect the yield and phytochemical quality of essential oils.

The objective was to evaluate the effect of drying temperature on the yield and phytochemical quality of essential oil extracted from ripe fruits of *S. terebinthifolius* as a way to contribute to the adequacy of postharvest processing of pink pepper.

2. MATERIAL AND METHODS

2.1. Geographic coordinates, soil characteristics, fertilization and irrigation

The research was carried out at the Piranga Valley Experimental Field, in Oratórios-MG. The geographical coordinates of the municipality are: latitude 20° 30"S, 43° 00" W and altitude 500 m, with the following meteorological variables: average annual maximum temperature of 21.8 °C and annual minimum of 19.5 °C and precipitation average annual rainfall of 1,250 mm. The predominant climatic type in the place is the Cwa, humid tropical, the Aw, semi-humid of hot summers, being the original vegetation constituted of subcaducifolia tropical forest, according to Köppen's classification (Cunha et al., 2000). The soil was classified as sandy loam-clay, with the following chemical characteristics: pH (water 1: 2.5) = 6.2; P (Mehlich) = 49.8 mg dm⁻³; P (remainder) = 40 mg L⁻¹; K = 220 mg dm⁻³; Ca = 2.6 cmol dm⁻³; Mg = 1.3 cmol dm⁻³; Al = 0 cmol dm⁻³; H + Al = 2.8 cmol dm⁻³; SB = 4.5 cmol dm⁻³; t = 4.5 cmol dm⁻³; m = 0%; T = 7.3 cmol dm⁻³; V = 61%; Organic matter = 2.63 dag kg⁻¹.

Fertilization was carried out, according to the soil analysis, using cattle manure, with the following chemical characteristics (%): N (1.4), P (0.39), K (0.88), Ca (1.54), Mg (0.27), S (0.23), CO (10.45); C / N (7.46). The drip irrigation system was used and for the control of spontaneous plants, manual weeding was performed when necessary.

2.2. Botanical identification

Samples of species were sent to EPAMIG's PAMG Herbarium, where the botanical identification was confirmed. Exsiccates were assembled (herborization), according to usual botanical standards and incorporated into the collection of the PAMG / EPAMIG Herbarium: *Schinus terebinthifolius* Raddi (PAMG voucher 58180)

2.3. Plant material - Seedling production, planting, cultivation and harvesting

Seedlings of *S. terebinthifolius* Raddi were purchased from a nursery in the Viçosa-MG region. The planting and cultivation in an organic system were carried out at the Piranga Valley Experimental Field of the Minas Gerais Agricultural Research Corporation (EPAMIG), in Oratórios-MG, in an area of 16 x 32 m, using the 4 x 4 m spacing between plants and lines. For extraction of essential oil, analysis of yield and phytochemical quality branches were harvested with ripe fruits from 18 useful plants in the central part of the production area. The harvest was carried out during the autumn of 2019, in the month of June, in the morning.

2.4. Drying of fruits

Immediately after harvesting, the ripe fruits of *S. terebinthifolius* were selected and submitted to drying (40,



50 and 60 °C) in an oven with forced air circulation until reaching constant weight. Subsequently, samples of 100 g of fruits were separated and packaged in polyethylene bags, sealed and packed in kraft paper packaging until the moment of extraction of essential oils.

2.5. Extraction of essential oil, calculation of yield and storage

In the extraction of essential oil from the riped fruits, the Clevenger apparatus was used, adapted to a round-bottomed flask with a capacity of 2000 mL. In each extraction, 250 mL of distilled water and 100 g of ripe fruits crushed in a knife mill were added to the flask, beginning the hydro distillation process. The extraction was carried out by dragging the essential oil through water vapor, for 2 hours and 30 minutes. After extraction, the oil yield was calculated by the difference between the final weight of the bottle containing the oil and the initial weight of the bottle, without oil, obtaining the yield for 100 g of fruits. The oil samples were stored at 4 °C in an amber glass bottle with a screw cap, for later chromatographic analysis.

2.6. Analysis and identification of volatile chemical constituents

The identification and content of volatile constituents were carried out in an Agilent gas chromatograph, model HP-6890, equipped with an Agilent mass selective detector, model HP-5975 and an HP-5MS capillary column (30m x 0.25mm x 0.25µm). The splitless injection mode was used, in the following temperature conditions: injector at 220 °C, column at 60 °C, with heating ramp of 3 °C · min⁻¹ and final temperature of 240 °C and detector at 250 °C. Helium was used as carrier gas at a flow rate of 1 mL · min⁻¹. A sample of essential oil was dissolved in ethyl acetate (20 mg \cdot mL⁻¹) for analysis. The identification of the analytes was carried out by comparing the retention indices (IR) obtained by the injection of hydrocarbon standards (C-8 to C-24), with the equipment's database (NIST-11 library) and with data from literature (Adams, 2007).

2.7. Statistical analysis of oil yield

The Tukey test at 5% probability was used to compare the average yields of essential oil at different drying temperatures, with the aid of the SAEG statistical analysis program (Ribeiro Jr., 2001).

3. RESULTS AND DISCUSSION

3.1. Yield of essential oil extracted from S. terebinthifolius ripe fruits

There was no significant difference (P>0.01) in the yield of essential oils regardless of the drying temperature used (Figure 1). Yields were 4.61% after drying the fruits at 40 °C, 4.63% at 50 °C and 4.91% at 60 °C, results similar to those described by Lorenzi & Matos (2002) who found of 5.0% of essential oil consisting of mono and sesquiterpenes in fruits. However, the oil yield in fruits submitted to drying at 50 °C was higher than the yield found by Governici (2019), which was 2.83% in fruits produced in the state of Espírito Santo (ES), submitted to drying at the same temperature. This difference can be attributed to edaphoclimatic factors, considering the difference in fruit origin. Other factors can also influence the yield and composition of essential oils, such as the phenological status of the crop, temperature, solar radiation, altitude of the crop location, water availability, nutrients, in addition to the pre-treatment of the raw material and the extraction process (Gobbo-Neto & Lopes, 2007; Maggieri et al., 2015). Governici (2019) also observed a reduction in the content of essential oil extracted from fruits at 70 °C, which can be explained by the increase in the temperature of the drying air that can cause the rupture of the oil storage structures and consequent the volatilization of some compounds and reduced essential oil yield (Ebadi et al., 2015; Ahmed et al., 2018).

Our results of essential oil yield extracted from ripe and dried fruits 40 °C were also superior to those of Maggieri et al. (2015) who observed a yield of 4.09% of essential oil when extracted from *S. terebinthifolius* fruits "in natura" and from 3.74% in dried fruits in an oven with forced air circulation at 40 °C.

Some authors found that the yield of essential oil extracted from whole fruits was not affected by the temperature, but by the crushing of the fruits. In this case, there is an increase in oil yield 26 times higher than the extraction of whole fruits submitted to drying at 50 °C and 23 times higher than 60 °C and 18 times higher than 70 °C (Governici, 2019). This difference may be associated, probably, with the rupture of the storage structures of the essential oil of the fruits, and a consequent increase in oil yield. However, Schimitberger et al. (2018) did not observe differences in yield of essential oil extracted from whole fruits and probably crushed, due to the method used for crushing that can interfere with the disruption of oil storage structures, since the seeds of *S. terebinthifolius* fruits present



well developed secretory channels and are protected by the bark (Machado & Carmello-Guerreiro, 2001).

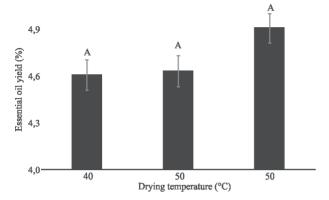


Figure 1 - Yield of essential oil extracted from ripe fruits of pink pepper submitted to drying (40, 50 and 60 °C).

Chemical constituents identified in the essential oil of pink pepper fruits subjected to drying at 40, 50 and 60 °C. The chemical constituents identified in the essential oil extracted from ripe pink pepper fruits and their concentrations are shown in Table 1. It is noteworthy that the major compounds (beta-pinene and alpha-pinene) were not altered with the drying temperature of the ripe fruits. Similar results was found by Sales (2013) and Acacio (2019), regarding to the major compounds and their contents in pink pepper fruits harvested in the Espírito Santo region. Some authors have identified only one of these constituents as the majority, Formagio et al. (2011) found beta-pinene as a majority and Macedo (2018) found alphapinene. However, other authors have identified different major compounds in ripe pink pepper fruits: Dourado (2012) identified mycrene, Barbosa et al. (2007) identified α -cadinol and elemol, Singh et al. (1998) and Santana et al. (2012) identified alpha-fandandrene, germacrene D, limonene and p-cymene.

In the essential oil of ripe pink pepper fruits, there is generally a predominance of monoterpenes and sesquiterpenes (Lorenzi & Matos, 2002, Formagio et al., 2011; COLE et al., 2014). However, essential oils from leaves, flowers and fruits of *S. terebinthifolius* from different locations can demonstrate some chemical variations in their composition (Ibrahim, 2004), which can be attributed to edaphoclimatic conditions, genetic variation, solar radiation, irrigation, fertilization, harvest time, post-harvest processing, among other factors (Gobbo-Neto & Lopes, 2007; Figueiredo et al., 2009; Alverenga et al., 2012).

TR (min)	IR	Chemical constituent	Drying temperature		
			40 °C	50 °C	60 °C
5.21	925	alpha-thujene	0.39	0	0
5.41	933	alpha-pinene	30.83	29.37	29.37
5.76	946	camphene	0.6	0.58	0.6
6.41	972	savineno	3.35	3.02	3.24
6.56	978	beta-pinene	39.22	37.79	38.24
6.86	989	beta-mycrene	1.48	2.92	0
7.29	1004	alpha-felandrene	0.74	0	0
8.08	1027	beta-felandrene	6.14	5.92	5.89
23.66	1416	trans-karyophylene	7.83	9.81	9.69
26.15	1478	germacrene D	8.18	11.88	24.02
26.75	1493	bicyclogermacrene	1.91	2.3	2.37
27.82	1521	delta-cadinene	0.73	0.98	0.93

Table 1 - Chemical constituents identified and their content (%) in essential oils extracted from ripe fruits of *S. terebinthifolium* submitted to drying at 40, 50 and 60 °C

TR - Rentention time; IR - Retention index.

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Maggieri et al. (2015) observed in the essential oil extracted from pink pepper fruits "in natura" a monoterpene content of 83.42% and in dried fruits a content of 76.01%. According to the authors, significant losses of monoterpenes may occur during the drying process of *S. terebinthifolius* fruits associated with their greater volatility.

Essential oil extracted from pink pepper can also vary in color according to post-harvest processing. Essential oils extracted from whole fruits showed a yellowish color while the oil extracted from crushed fruits of pink pepper has a translucent color. The oil obtained from the ripe fruits used in our work was translucent, as well as observed in fruits processed in the same way in studies conducted by Sefidkon et al. (2007); Memarzadeh et al. (2015); Schimitberger et al. (2018); and Ahmed et al. (2018).

4. CONCLUSION

The results of this study may contribute to optimize the post-harvest processing of ripe pink pepper fruits (*Schinus terebinthifolius*), considering that the yield and phytochemical quality of essential oil extracted from ripe fruits were not affected by the drying temperatures of 40, 50 and 60 °C.

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