Industrial Crops & Products 112 (2018) 389–395

Contents lists available at ScienceDirect

Industrial Crops & Products

journal homepage: www.elsevier.com/locate/indcrop

Changes in the essential oil content and composition of *Thymus daenensis* Celak. under different drying methods

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ARTICLE INFO

Keywrods:
Thymus daenensis Celak
Vacuum oven
Microwave
Essential oil
Thymol

ABSTRACT

Some active ingredients of the medicinal plant are changed during a drying process. This study was aimed for the effect comparison of the various drying methods on quantity and quality of *Thymus daenensis* essential oil. The factorial experiment treatments included pre-drying under sun as the first factor and various drying methods (fresh plant, sun drying, shade drying, oven drying at 35, 45, and 55 °C, vacuum drying at 35, 45, and 55 °C, and microwave drying at 100, 500, and 1000 W) as the second factor. The results showed that various drying methods and their interaction with the pre-drying had a significant effect on the essential oil content and its components. In present study an adverse relation was found between the essential oil content and thymol or carvacrol amount in the dried plant. The highest essential oil amount was observed in the oven and vacuum oven-drying at 35 °C without the pre-drying operation. While, the greatest content of thymol as the predominant oxygenated monoterpenes was acquired in the vacuum oven-drying at 55 °C. Generally, the oven and vacuum oven-drying at 35 °C without the pre-drying are recommended for *Thymus daenensis* drying due to possessing the highest quantity of essential oil and appropriate quality.

1. Introduction

Thyme (*Thymus daenensis* Celak.) is an herbaceous, perennial, and bushy herb which belong to Thymus genus of Lamiaceae family. The Persian and local names of *Thymus daenensis* is “Avishan-e-denaee” (Mozaffarian, 1996). Thymus genus includes about 350 species that are rich in essential oils. The major components in *Thymus daenensis* essential oil are thymol and carvacrol. These compounds have antioxidant activity (Dorman and Deans, 2004; Miguel et al., 2004; Sokmen et al., 2004; Youdim et al., 2002). It has been reported that the essential oil obtained from leaves and flowers have antispasmodic, carminative, anti-rheumatic, anti-sciatica, and strong antiseptic (Pirbalouti et al., 2013). As well as, this oil was used for producing mouthwash and cough syrups in the pharmaceutical industry.

Drying is one of the oldest techniques for storage food, meats, and herbs. This process involves water removal by evaporation into a certain threshold to prevent the activities of enzymes, microorganisms, and yeasts to increase the shelf life (Prusinowska and Śmigielski, 2015). Thus, biochemical reactions attributed to post-harvest decay due to lack of enough water impaired and herbs protected from degradation. Moreover, weight and volume of herbs reduced by a drying process, therefore the costs of packaging, storage and transportation will be minimal (Chakraborty and Dey, 2016).

In current, the variety of natural, artificial, and combined methods were used for plant matter drying. Natural methods are drying by sun or shade, while artificial drying procedures include warm air drying or modern technologies such as drying under vacuum and microwave radiation (Ekechukwu, 1999a, 1999b). Natural drying is an important method for drying crops because of lower costs. The natural drying has disadvantages such as not being able to move a large amount of plant matter and achieve the stable standards of quality (Soysal and Öztekin, 2001). Drying with hot air also due to lessen costs is often used for industrial production of dried herb. Albeit, the low energy efficiency and time-consuming operations are notable disadvantages for the warm air drying (Soysal and Öztekin, 2001).

Microwave radiation is used for drying plants. Short drying time is including important benefits to this approach. In addition, microwave energy helps to maintain the color of dried herbs and improving the plant active ingredient. Microwave radiation spread to plant matters quickly and impressive, thus energy consuming reduced due to rapid

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https://doi.org/10.1016/j.indcrop.2017.12.012
Received 13 June 2017; Received in revised form 2 December 2017; Accepted 4 December 2017

drying (Diaz et al., 2003).Unlike other drying methods that the heat penetrated from a surface to depth, in microwave method the heat was produced within the plant material and then distributed to the outside (Blose, 2001; Alibas, 2007). Heindl and Müller (2002) reported that valerian roots dried by microwave radiation possessed the high quality and low microbial contamination.

Drying under vacuum is another method, which conducted out in sub-atmospheric pressures. This is a very effective procedure for dehydration of sensitive products because a high degree of drying was obtained without the need to increase the temperature. Thus, this possibility is provided that susceptible materials to high temperature were dried by vacuum drying technique in short time. Moreover, vacuum drying is the best method for material which exposed to air oxygen rapidly oxidized (Jaya and Das, 2003; Mitra et al., 2011).

The active ingredients composition in the fresh medicinal plant may be changed by enzymatic processes during the drying process (Jambor and Czosnowska, 2002). Also, some studies have been reported that drying method has a significant impact on the essential oil content and constitutes (Basver, 1993; Deans and Svoboda, 1992). However, drying methods effect on essential oil yield and its components vary depending on using temperature, drying time, and plant species (Yazdani et al., 2006). Sefidkon et al. (2006) reported that the highest essential oil content in *Satureja hortensis* L. was respectively obtained in drying by oven 45 °C, the sun, and shade. Ahmadi et al. (2007) expressed that various drying methods include the sun, shade, oven 30 and 40 °C did not have a significant effect on quantity and quality of *Rosa damascena* Mill. essential oil. However, the maximum amount of citronellol and geraniol (the most important components for improving Rosa essential oil) was observed in plants dried by the shade.

Thus, an assessment regarding the effect of various drying methods on medicinal plants essential oil changes is an important issue. This study was aimed for the effect comparison of natural and artificial drying methods on quantity and quality of *Thymus daenensis* essential oil. Moreover, different temperatures effect in artificial methods on the essential oil was evaluated.

2. Materials and methods

This study was performed to evaluate the effect of different drying methods on quality and quantity of *Thymus daenensis* essential oil in the Institute of Medicinal Plants, ACECR. A factorial experiment was conducted based on the randomized complete block design (RCBD) with 3 replications. The first factor was pre-drying included exposing to the sun for 4 h and without the pre-drying that the samples dried immediately after harvest under related drying methods. The samples in the drying delay were exposed to the sun for 4 h; thereafter, placed under related drying methods. The second factor was various drying methods, which were included control (freshly harvested plant), sun drying, shade drying, oven drying at 35, 45, and 55 °C, vacuum drying at 35, 45, and 55 °C, and microwave drying at 100, 500, and 1000 W.

2.1. Sample preparation

The same and identical cuttings were collected from a *Thymus daenensis* plant. The cuttings were embedded in growth media that included sand, coco peat, and perlite (3:1:1) for rooting. Approximately 45 days later, the rooted cuttings were sown in the farm at October 2015. All agronomy practices include irrigation, fertilization; weed management, etc. conducted out on the base of plant requirement. Fresh plants in the flowering stage were harvested as randomly. Samples were collected at 10 June 2016 in early hours of a sunny morning (8–9 am). The samples were divided into two groups. A group exposed under the sun for 4 h in the farm. Another group immediately transported to the laboratory. Then, the samples in each group were dried by the various methods.

2.2. The sun and shade drying

For the sun drying method, a white clean cloth was flattened on the open area and then the plant samples were spread on the cloth beneath the sun. In sun-drying conditions, the pre-drying operation was to place the plant sample after harvest for 4 h in the shade and then in the sun. Also, the samples without the pre-drying operation were dried immediately after harvest at sun.

Also in the shade drying method, the samples were spread inside the room without direct sunlight penetration at room temperature. In both methods, the drying process was continued until the moisture content reached to 10%.

2.3. Oven drying

The sample was spread in a thin layer on the tray. Oven temperature was regulated on operating temperatures including 35, 45 and 55 °C. The samples were removed from the oven after their moisture reached to 10 percent. Then they were ready for essential oil extraction.

2.4. Microwave drying

A microwave oven (Samsung, model MC35J8055CK) with 2250 W power output at 2450 MHz, which equipped to a swivel tray plus digital setting for power and time was used for the sample drying. The plant material was poured into a glass container and then placed inside the microwave cavity. Radiations power were regulated on 100, 500 and 1000 W. These operations have been continued as long as the moisture content was reduced to 10%.

2.5. Vacuum drying

The samples in vacuum drying method were dried in a vacuum-dryer (Memmert Inc. VO 500 model, Germany) with technical features of 230 V, 60 Hz, and 2400 W. The vacuum oven temperature was sensitive to temperatures ranging from 5 to 200 °C. The various temperatures included 35, 45, and 55 °C under vacuum condition (600 mbar) was adjusted for every 5 h.

2.6. Essential oils (EO) analysis

Each dried sample was ground to a fine powder using a Moulinex food processor and was passed through a 20 mesh sieve to remove large pieces of debris. Oils were extracted by hydrodistillation for 3 h, from 50 g the ground tissue in 1 l of water contained in a 2 l flask using Clevenger-type apparatus, according to producers outlined British Pharmacopoeia. The oils were dried over anhydrous sodium sulphate and kept at 4 °C until it was analyzed. The experiment was repeated three times and its mean was reported as EO percent on the dry plant.

GC/MS analysis was performed on an Agilent instrument coupled with a 5973 Mass system equipped with flame ionization detector (FID) and a BPX5 capillary column (30 m × 0.25 mm; 0.25 μm film thicknesses). Temperature program includes oven temperature held for 2 min at 60 °C and was enhanced to 150 °C with 3 °C per min rate. Then, temperature enhancement was programmed up to 270 °C as 5 °C per min rate and this temperature held for 15 min. Other operating conditions include: carrier gas was He with a flow rate of 1.1 ml min^{-1}; injector and detector temperatures were 300 °C, and split ratio, 1:50. Mass spectra were taken at 70 eV. The mass spectra and retention indices of essential oil components were identified by comparison to published literature and presented the MS computer library (Adams, 2001).

2.7. Statistical analysis

All the data were analyzed by SAS software to GLM procedure. The