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The effect of drying time on the yield and the chemical composition of essential oil and dissolved oil in hydrolat from aerial parts of Moroccan *Thymbra capitata* (L.) Cav.

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Abstract: The present research aimed to study the effect of drying time on the yield and chemical composition of essential oil (EO) and dissolved oil in hydrolat (HY) from aerial parts of Moroccan *Thymbra capitata* (L.) Cav. Drying of plant material was carried out naturally in the shade of a draughty place at room temperature (25–27°C). A series of 10 plant samples were subjected to hydrodistillation using a Clevenger-type apparatus. The results indicated that the yield of EO increased with drying time to reach the highest value on the 8th drying day (2.7%), while the yield of HY has not undergone an apparent variation (0.2% – 0.6%). Based on the GC-MS analyses, EO was composed mainly of the phenolic monoterpene carvacrol (80.10%–92.27%) along with its biogenetic precursors' monoterpene hydrocarbons in a 1.02%–4.81% range *p*-cymene and 0.24% -1.86 % γ-terpinene. Other essential components occurring in minor quantity were sesquiterpene hydrocarbon α-humulene (2.58% – 4.67%) and oxygenated monoterpene linalool (0.80% –2.06%). At the same time, HY was constituted mainly of carvacrol (94.67–98.42%) along with α-humulene at much lower concentrations (0.31%–0.86%) and the oxygenated derivative acetovanillone acetate (0.2%–1.80%). On the other hand, the highest concentration of carvacrol in EO was reached on the 5th day of the drying plant process (92.27%), while the HY recovered on the 7th day has shown carvacrol in its highest concentration (98.42%).

Keywords: Essential oil; hydrolat extract; *Thymbra capitata* (L.) Cav; drying; GC-MS.

1. Introduction

Aromatic and medicinal plants are a source of abundant secondary metabolites such as essential oils, phenolic compounds, and flavonoids, which show different biological effects [1-4]. Several genera of aromatic and Medicinal plants are included in the *Lamiaceae* family. The thymus is one of these genera represented by more than 200 species everywhere in the world [5]. In Morocco, this genus is represented by 21 species, which 12 are endemic [6]. *The thymus* is a taxonomically complex group of aromatic plants utilized for medicinal uses or as spices almost everywhere in the world. It is much frequent in the Mediterranean region [7-8]. *The thymus* is a plant of enormous economic importance, especially in the Mediterranean basin and North America [9]. Essential oils of *Thymus* have proven its value as a source of bioactive compounds with several biological activities such as antispasmodic [10], antibacterial [11], antifungal [12-13], anti-tabagism [14], antioxidant [11], and antimicrobial [15].

T. capitata (L.) Cav. (formerly *Thymus capitatus* (L.) Hoffmanns. & Link) as a *Thymus* species, is a Mediterranean endemic plant [16]. In Morocco, it grows only in the perimeter of Tetouan city (northern Morocco) at a temperate bioclimate [6]. This species is locally known under the vernacular name of "Zaetra" in Moroccan dialect and "Azoukeni" in Tamazight. It commonly used as a food preservative for meat and fish [17]. Furthermore, several studies have reported that *T. capitata* (L.) Cav essential oil possesses different biological and pharmacological activities, such as antibacterial [18], antimicrobial [19], antioxidant [20-22], antifungal [23], anti-inflammatory [1], parasiticide [21], and antispasmodic [24].

Drying, like dehydration, is a process that eliminates moisture from the plant. The elimination of water from the plant inhibits possible decomposition of phytochemicals and microbial contamination. Most of the drying methods such as air drying, vacuum drying, and oven drying, apply heat on the plant to eliminate the moisture. The increasing drying temperature reduces the drying time by promoting the drying rate. Nevertheless, the employ of high temperature is frequently compromised by the degradation of plant quality [25]. The primary consideration in plants drying is the conservation of

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the phytochemicals, which are often heating sensitive.

Consequently, in the present work, the drying of the studied plant was carried out at room temperature (25–27°C). *Thymus* is so perishable, and drying has a beneficial effect, which enhances its storage life for further use. Thus, drying is a conservation method used to ensure the microbial safety of aromatic and medicinal plants. The loss of volatile compounds in aromatic and spice plants depends mainly on drying procedures. Indeed, these compounds, especially terpenes, are the most sensitive ones in the drying process [26]. The drying time also has a significant impact on the qualitative and quantitative composition of aromatic plants' essential oils.

Thus, this work aimed to study the effect of drying time on the yield and the chemical composition of essential oil and hydrolat from aerial parts of Moroccan *T. capitata* (L.) Cav and the economic benefit of this process.

2. Results and Discussion

2.1. Dehydration kinetics of *T. capitata* (L.) Cav

The dehydration kinetics of plant material was studied. As shown in Figure 1, a sharp decrease in plant matter weight was noted until the 6th day of the plant drying process. Afterward, no weight loss was observed, confirming the moisture removal from the plant matter.

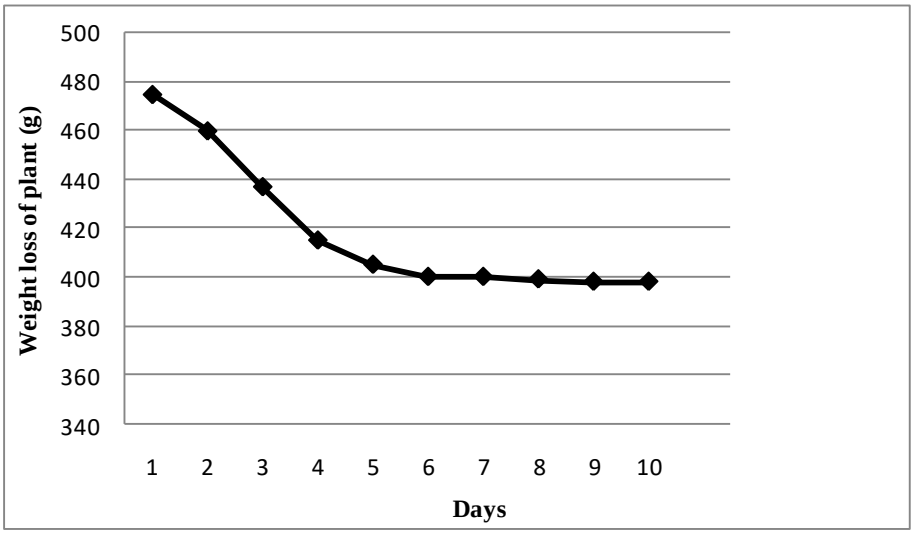

Figure 1. Variation in the weight loss of *T. capitata* (L.) Cav plant during drying time (Days). Data are the mean of three determinations

2.2. Evolution of essential oil and dissolved oil in hydrolat yields

As shown in Figure 2, *T. capitata* (L.) Cav EO yield was strongly influenced by drying time. Indeed, its value increased to reach a maximum rate of 2.7% on the 8th day of plant drying and decreased after that. This result is following a study carried out by Bourkhiss et al. (2009) [27]; these researchers found that *Tetraclinis articulata (Vahl) Masters* essential oil yield increased with drying time to range its maximal value on the 9th day of plant drying and after that decreased. Goudjil et al. (2015) [28] also confirmed the same results by studying the drying effect on the *Laurus nobilis Lauraceae* essential oil content.

On the other hand, Zrira et al. (1995) [29] reported that during drying of E. camaldulensis leaves under the shade. The essential oil yield increased by 54% (maximum reached the 16th day). According to them, this result is due to enzymatic activity and that the essential oil biosynthesis persists after the harvesting of the plant material because of water stress. Conversely to the EO yield, which has undergone a significant variation, the HY yield showed a lower variation (0.2%–0.6%) (Figure 2).

2.3. Evolution of essential oil and hydrolat chemical composition

As shown in Table 1, the GC-MS analyses of *T. capitata* (L.) Cav EO have identified ten volatile compounds representing 97.28 to 99.50% of total oil. It mostly consisted of phenolic monoterpene carvacrol during the process of plant drying (80.10%–92.27%) confirming that *T. capitata* (L.) Cav is a carvacrol chemotype, according to literature data for this species in the world [30-33]. Biogenetic precursors of the phenols were present in a 1.02%–4.81% range *p*-cymene and 0.24%–1.86% γ-terpinene along with α–humulene 2.58%–4.67% as the only sesquiterpene constituent identified in this oil. Thus, linalool, as an oxygenated monoterpene was identified at relatively much lower levels (0.80%–2.06%). Among others constituents detected in EO, many monoterpene hydrocarbons were found at very lower concentrations: α-pinene (0.36%–0.71%), sabinene (0.42%–0.62%), β-phellandrene (0.45–0.90%), β–ocimene (0.28%–0.78%) and terpinolene (0.52%–0.86%).

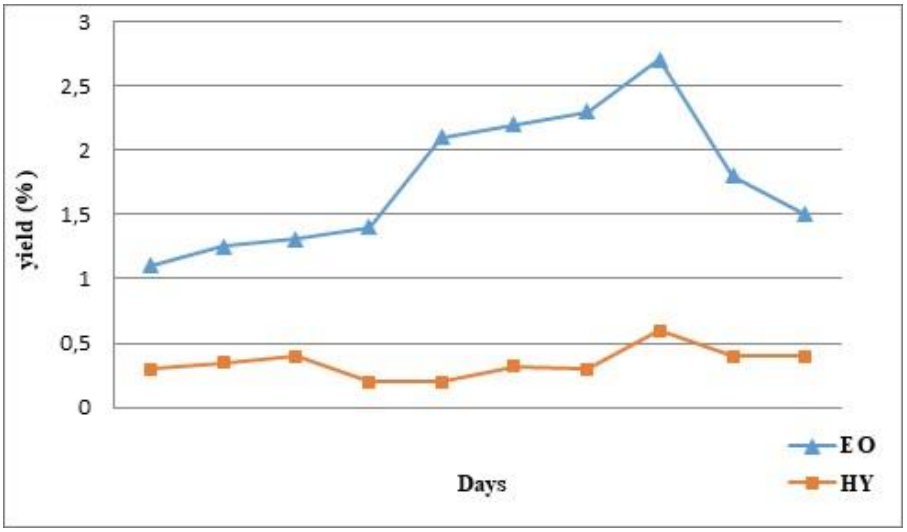

Figure 2. Evolution of *T. capitata* (L.) Cav essential oil and dissolved oil in hydrolat yield versus drying time (Days). Data are the mean of three determinations

The results summarized in Table 1 indicated likewise that drying time influenced the chemical composition of T. capitata (L.) significantly Cav, especially carvacrol, *p*-cymene, and *γ*-terpinene. Thus, the concentration of carvacrol changed, unlike that of *p*-cymene, which may explain the biogenetic relationship between these constituents. Indeed, the metabolic pathway for the carvacrol formation begins with the unsaturation, followed by the hydroxylation to C-2 aromatic ring [32] (Figure 3). This shows the critical role played by the *γ*-terpinene in the flavoring process and by *p*-cymene as a precursor for oxygenates compounds [34-35]. On the other hand, our results are in harmony with those of Silou et al. (2002) [36] and Bourkhiss et al. (2009) [27] who reported that drying time influences the chemical composition of *Eucalyptus citriodora* and *Tetraclinis articulata (Vahl) Masters* essential oil respectively, and especially the main components.

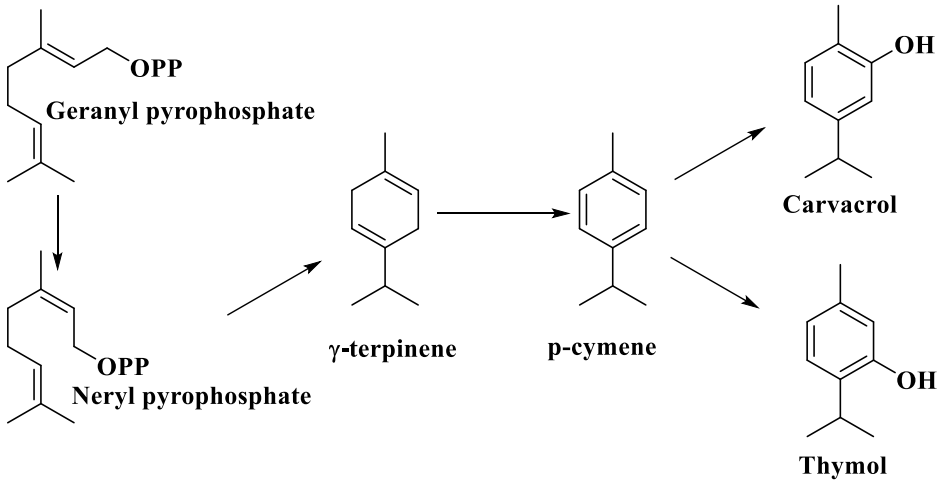

Figure 3. General biosynthesis pathways of aromatic monoterpenes carvacrol and thymol

A total of seven components were identified, amounting to 98.44%–100% of the *T. capitata* (L.) Cav hydrolat (HY) during the plant drying process. This extract was constituted mainly of carvacrol (94.67%–98.42%) along with *α*–humulene at much lower concentrations (0.31%–0.86%). It was likewise characterized by the absence or almost absence of monoterpene hydrocarbons owing to their lipophilic character. Generally, the oxygenated constituents are found in large quantities in hydrolat because of their hydrophilic character. In contrast, the lipophilic terpene compounds are absent or almost absent, which is by other studies [32,33,37]. On the other hand, acetovanillone acetate, as an oxygenated derivative not detected in EO, was present in HY (0.2%–1.80%).

Based on the data obtained in this work (Table 1), the highest proportion of carvacrol (92.27%) was observed in EO on the 5th day of drying plant, while this compound recorded utmost concentration in HY on the 7th day (98.42%).

Table 1. Chemical composition of EO and HY from T. capitata (L.) Cav during plant.

RT	RI	LRI	Compounds	Concentration %																	
				Essential oils (EO)										Hydrolats (HY)							
				EO_1	**EO_2**	**EO_3**	**EO_4**	**EO_5**	**EO_6**	**EO_7**	**EO_8**	**EO_9**	**EO_{10}**	**HY_1**	**HY_2**	**HY_3**	**HY_4**	**HY_5**	**HY_6**	**HY_7**	**HY_8**	**HY_9**	**HY_{10}**
Monoterpene hydrocarbons																					
7.28	931	939	α-Pinene	tr	tr	tr	0,42	tr	0.36	0,71	tr	0.48	0.45	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr
8.43	969	975	Sabinene	tr	0.52	Tr	0,62	0.58	0.61	0,47	0,42	0,45	0.48	tr	tr	tr	0,39	tr	tr	tr	0,19	tr	tr
9.15	1023	1025	*p* -Cymene	3.08	3.12	3.21	4,81	1,02	7.02	5,44	3,36	3,02	3.11	0.31	0.33	0,34	1,15	0 ,44	0,30	0,37	0,67	0,55	0.57
9.50	1031	1031	*β*-Phellandrene	0.46	0.43	0.48	0.45	tr	0.78	0.90	0.58	0.51	0.53	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr
9.74	1034	1037	*β*–Ocimene	0.33	0.35	0.39	0.28	tr	0.78	0.42	0.26	0.41	0.43	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr
10.70	1056	1062	*γ*-Terpinene	1.55	1.53	1.51	1.27	0.24	1.70	1.86	1.59	1.55	1.52	tr	tr	tr	0.38	tr	tr	tr	0.18	tr	tr
10.90	1083	1088	Terpinolene	0.51	0.55	0.52	0.53	tr	0.86	0.80	0.61	0.55	0.52	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr
Oxygenated monoterpenes																					
10.95	1101	1098	Linalool	1.25	1.21	1.21	2.04	1.84	2.06	1.55	0.91	0.80	0.83	0.55	0.61	0.67	1.28	0.56	0.41	0.53	0.60	0.43	0.38
17.49	1300	1298	Carvacrol	86.78	86.74	86.66	85.23	92.27	80.10	83.56	87.01	87.50	87.41	97.51	97.47	97.48	94.67	97.23	97.14	98.42	96.73	96.85	96.83
Sesquiterpene hydrocarbons																					
20.69	1451	1454	*α*–Humulene	2.85	2.89	2.92	3.60	3.55	4.67	3.54	2.62	2.58	2.61	0.47	0.37	0.36	0.86	0.31	0.35	0.34	0.48	0.41	0.46
Oxygenated derivatives																					
21.26	1570	1573	Acetovanillone acetate	tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	1.12	1.20	1.14	1.26	1.45	1.80	0.34	0.27	0.20	1.25
Monoterpene hydrocarbons				5.93	6.50	6.11	8.38	1.84	12.11	10.60	6.82	6.97	7.04	0.31	0.33	0.34	1.92	0.44	0.30	0.37	1.04	0.55	0.57
Oxygenated monoterpenes				88.03	87.95	87.87	87.27	94.11	82.16	85.11	87.92	88.32	88.24	98.06	98.08	98.15	95.95	97.79	97.55	98.95	97.33	97.28	97.21
Sesquiterpene hydrocarbons				2.85	2.89	2.92	3.60	3.55	4.67	3.54	2.62	2.52	2.61	0.47	0.37	0.36	0.86	0.31	0.35	0.34	0.48	0.41	0.46
Oxygenated derivatives				tr	tr	tr	tr	tr	tr	tr	tr	tr	tr	1.12	1.20	1.14	1.26	1.45	1.80	0.34	0.27	0.20	1.25
Total %				96.81	97.34	96.90	99.25	99.50	98.94	99.25	97.36	97.81	97.89	99.96	99.98	99.99	99.99	99.99	100	100	99.12	98.44	99.49

tr: Trace (≥0.17)
RI: Retention indices as determined on DB-5MS column using homologous series of n-alkanes
LRI: Literature retention indices on DB-5MS column
RT: Retention times

3. Conclusion

Our findings have shown that the highest yield in the hydro distillate *T. capitata* (L.) Cav EO was observed on the 8th day of the plant drying process (2.7%). Besides, the highest rate of carvacrol in EO was reached on the 5th day of the plant drying process (92.27%), while HY recovered on the 7th day shown carvacrol in its highest concentration (98.42%) which makes this oil as quasi-pure product. On the economic plan, it is better to dry this plant for 8 days to reach maximum yield, while it is drying for 5 days provides a maximum concentration of the phenolic compound carvacrol as the active component of this plant.

4. Acknowledgments

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5. Experimental

5.1. Plant material

The aerial parts of *T. capitata* (L.) Cav. (11kg) was collected at the flowering stage in July 2015 from the Tetouan area in northern Morocco (Latitude: 35°34'42" N, Longitude: 5°22'06" W; at 121m above sea level). The identification of this plant was confirmed by Professor Mohamed Kadiri (botanist in Biology Department, Faculty of Sciences, Tetouan, Morocco). The voucher specimen (INP 1235) was deposited in the herbarium of the national institute of medicinal and aromatic plants of Taounate in the Sidi Mohamed Ben Abdellah University Fes Morocco. Ten (10) samples were air-dried at room temperature under shade until the weight was stable.

5.2. Isolation of essential oil and dissolved oil in hydrolat

After plant harvesting, a series of 10 samples of 500g were weighed and subjected to hydrodistillation for 3h (dried with an interval of 24h) using a Clevenger-type apparatus advocated by the European Pharmacopoeia that allows the recycling of the aqueous phase of the distillate using a cohobage system. Essential oils then dissociate spontaneously of hydrolats for their immiscibility. The dissolved oil in hydrolat was obtained by liquid-liquid extraction of hydrolat from each hydrodistillation with dichloromethane (CH_2Cl_2) [38]. The organic phase was evaporated under reduced pressure by a rotary evaporator giving a yellowish extract. Three replicates were performed for each sample, and the recovered oils each day were combined and named from EO_1 to EO_{10}. The same for corresponding hydrolats witch named from HY_1 to HY_{10}. Finally, all oils were dried with Na_2SO_4 and stored in the tightly closed dark vial at 4°C until analysis.

5.3. Chromatographic analysis

The analysis of the essential oils and hydrolat extracts was performed on a GC–MS (Agilent Technologies, J&W Scientific Products, Palo Alto, CA, USA), equipped with an Agilent Technologies capillary DB-5MS column (30 m length; 0.25 mm i.d.; 0.25 mm film thickness), and coupled to a mass selective detector (MSD5975B, ionization voltage 70 eV; all Agilent, Santa Clara, CA). The carrier gas was Helium and was used at 1 mL min^{-1} flow rate. The oven temperature program was as follows: 1 min at 100°C ramped from 100 to 260°C at 4°C min^{-1} and 10 min at 260°C. The chromatograph was equipped with a split/splitless injector used in the split mode. The split ratio was 1:100. The identification of components was assigned by matching their mass spectra with Wiley and NIST library data and standards of the main components. After that, Kovats retention indices calculated by linear interpolation relative to retention times of C_8C_{22} *n*-alkanes [39] were compared with reference libraries or literature data [40] and with those of authentic compounds by their co-injection under the same chromatographic conditions mentioned above. Quantification was done by the standard external method using calibration curves generated by running GC analysis of authentic representative compounds. All compounds concentrations less than 0.17% are considered as traces.

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