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Extraction Essential Oil from *Thymus vulgaris* L. Leaves Using Microwave Reactor: Optimization and Kinetic Study

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Abstract

In this work, the extraction of natural oil from closed Thymus vulgaris L. (thyme) leaves was examined utilizing microwave hydro-refining. Microwave hydro-refining strategy was read up for the extraction of Thymus vulgaris L. oil. Besides, the impact of the size of the material (unblemished and hacked leaves), the impact of the proportion of the heaviness of unrefined substance to the volume of dissolvable (1:10, 1:20 and 1:30g/ml) and the impact of microwave power (500 and 700W) to the yield of thyme oil and energy through the extraction interaction. Thus, at that point, a microwave hydro-refining model in light of the presumption of a second-request system was created to foresee the rate steady of extraction.In this exploration, the best conditions were at 1hr as an extraction time for 700w microwave power, dissolvable to strong proportion of 1g plant leaves/30ml dissolvable for the hacked plant. Chromatography-Mass Spectrometry (GC-MS) was utilized as a portrayal procedure to examine and recognize the got thyme oil to its structure compounds.

Keywords

Thyme oil, Microwave hydrodistillation, Extraction, Kinetics. ***Thymus vulgaris***

Introduction

The extracted essential oils can be characterized as perplexing combinations of various compunds like unpredictable, fragrant, and hydrophobic mixtures, which can be found in different pieces of sweet-smelling plants, for example seeds, sprouts, blossoms, shoots and leaves. (Khalil, Ashour, Fikry, Singab, and Salama, 2018)(K M Abed and Naife, 2018). These mixtures are auxiliary plant metabolites which cause them a decent wellspring of a few bioactive mixtures that to have antioxidative and antimicrobial properties (Calo, Crandall, O'Bryan, and Ricke, 2015)(Tongnuanchan and Benjakul, 2014)(K M Abed, Noori, and Darwesh, 2014). Medicinal oils contain a few mixtures and metabolites which would hinder the wide scope of microorganisms like microbes, molds and yeasts (Nazzaro, Fratianni, De Martino, Coppola, and De Feo, 2013). Because of these properties (the cell reinforcement and antimicrobial properties), coriander seeds rejuvenating balm plays a part in forestalling food waste and food conservation (Mandal and Mandal, 2015). Natural ointments are not just successful in the treatment of irresistible infections yet additionally decrease the many secondary effects that are frequently brought about by engineered antimicrobial mixtures (Zardini et al., 2012).

Thyme (Thymus vulgaris L.), having a place with the Lamiaceae family, is notable as quite possibly the main zest plants having incredible restorative properties. It is usually utilized in protection and food seasoning (Ojeda-Sana, van Baren, Elechosa, Juárez, and Moreno, 2013).

Moreover, thyme EO have very strong antibacterial, anti-mutagenic, anti-carcinogenic, antifungal, anti-inflammatory, antioxidant,, , , and insecticidal properties, and that due to its variety important components (Usai et al., 2011), which make it an active substance that can be used in different sectors such as alternative medicine, cosmetic, pharmaceutical, and agricultural industries, additionally, as constituents of insecticides and disinfectants and in natural therapies or (Gavarić et al., 2015).

The genus thymus contains 215 species which the Mediterranean area could be expressed as a centre of the genus(Stahl-Biskup & Saez, 2002).

Figure 1. Thyme (*Thymus vulgaris*) plants in Haditha city (IRAQ).

Thyme oil (distilled from its leaves) is a common in the world's top ten essential oils due to its important uses (Stahl-Biskup & Saez, 2002). In this research, Thyme leaves were collected from Al- Anbar Province (specifically Hit and Haditha cities)- IRAQ (Fig. 1). Compunds such as thymol, p-cymene, γ terpinene, β-caryophyllene have been recognized and mentioned by many studies which are considered as the main constituents of thyme essential oil..

There were numerous strategies to separate the medicinal ointments from plants parts, like hydrodistillation(HD), steam refining, steam and water refining, maceration, empyreumatic (or horrendous) refining, and articulation (Stahl-Biskup and Saez, 2002). From the writing, it very well may be seen that HD is the most well-known method for removing the medicinal balm and has been broadly utilized in past examinations (Golmakani and Rezaei, 2008)(Stahl-Biskup and Saez, 2002). Nonetheless, modern techniques, for example, the Microwave-Assisted Hydrodistillation (MAHD) strategy were explored to upgrade the Hydro refining strategy. MAHD can upgrade the nature of the concentrates, altogether diminish the extraction time, most likely work on the yield of the extraction (Golmakani and Rezaei, 2008). Different benefits of MAHD are more successful warming, lower energy utilization, decreased warm slopes (Farhat, Ginies, Romdhane, and Chemat, 2009).

The Microwave-Assisted Hydrodistillation is a high level hydrodistillation method that uses a microwave in the extraction interaction (Golmakani and Rezaei, 2008).

The motivation behind this examination is to concentrate on the course of thyme oil extraction from thyme leaves utilizing a microwave hydrodistillation strategy. This examination concentrated on the impact of a few factors like the treatment of plant materials (hacked and flawless leaves) in addition to the impact of the proportion of unrefined substance being removed with a dissolvable to yield thyme oil. Other than it additionally concentrated on the impact of microwave power on the yield of thyme oil and energy through the extraction interaction. So far, at that point, techniques for deciding the underlying extraction rate and the enactment energy of rejuvenating oil extraction, in light of a second-request extraction procedure are created. Active boundaries are at last reasoned to anticipate how much natural balm extricated, then, at that point, the aftereffects of these computations were contrasted and talked about with streamlining the extraction interaction. This study was important prior to creating and carrying out a total modern interaction**.**

Materials and methods

Plant material

New leaves of thyme filling in a public region in Hit city were arbitrarily gathered in March 2020. Gathered leaves were washed, by refined water to eliminate dust, and dried in the dark area for around 15 days. the leaves then, at that point, were hacked utilizing a business grade blender (Generic BPA 3HP 2200W) and put away at room temperature until required.

Strategy of Microwave Hydrodistillation

To use microwave hydrodistillation, utilize a home microwave. The components of the microwave are 46.1 cm × 28.0 cm × 37.3 cm. A microwave being adjusted via penetrating an opening in the top. The level base carafe with the limit of 1000 mL is set inside the stove and is associated Clevenger mechanical assembly part during the opening. Afterthat and at that point, the opening is shut with PTFE to forestall any deficiency of hotness as displayed in fig. (2). In the thyme oil extraction utilizing microwave hydrodistillation, refined water and thyme leaves (unblemished or hacked) were placed in level base cups in a succession relying upon a proportion of thyme passes on to the water of 1:10, 1:20 and 1:30 g/ml. The extraction was done for 90 min under various degrees of viable microwave power (500 and 700 W) temperatures changed from 373 to 390 K for the microwave power 500 W and from 373 to 400 K for the microwave power 700 W. The temperature degree is estimated via utilizing a fiber optic temperature sensor embedded in a microwave cavity (Conde, Falqu, and Domínguez, 2016). The various densities and their immiscibility necessitated which water and thyme oil being isolated from one another in a graduated cylinder and the abundance water being refluxed to the extraction to keep the strong to dissolvable proportion. The thyme oil was gathered and dried under anhydrous sodium sulfate and put away at 4 C.

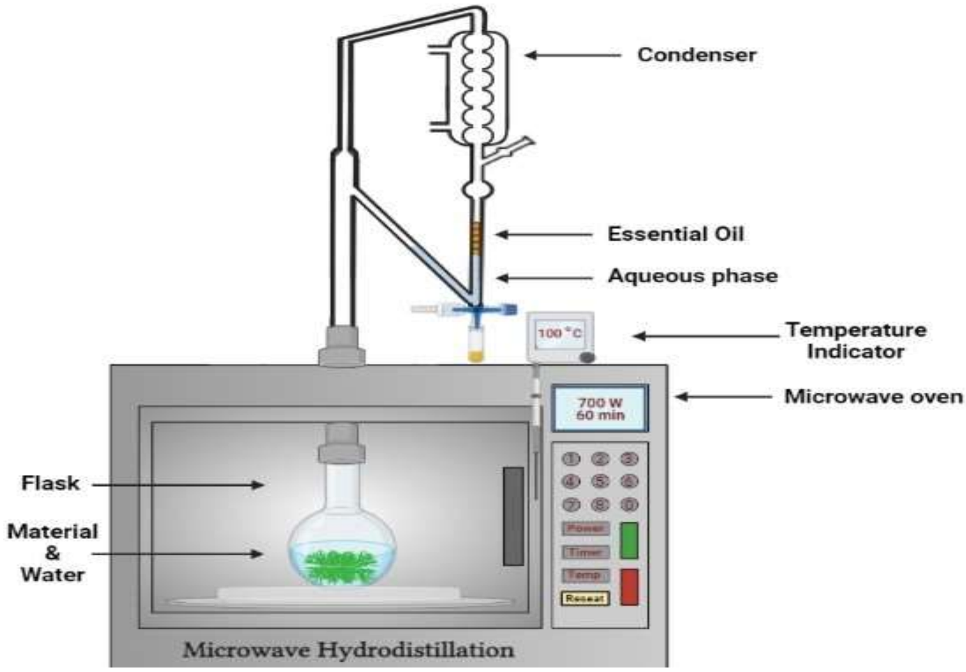


Figure 2. Schematic representation of the microwave hydrodistillation apparatus used in this study.

Kinetic model

Different approaches of strong fluid extraction process being depicted (Heri Septya Kusuma and Mahfud, 2017) and (Ramanathan and Thangarasu, 2019) shows, in the investigation of the systems and energy in the extraction cycle of medicinal ointment from Pogostemon cablin Benth and Aegle Marmelos Correa separately. model in light of second-request extraction energy being more a reasonable model for a medicinal ointment extraction process. That is being conceivable to assemble the motor models of rejuvenating oil extraction utilizing a microwave hydrodistillation strategy and the extraction request and rate steady still needed not entirely settled by tests.

General Conditions

The second-request energy condition for the pace of extraction by (H S Kusuma and Mahfud, 2018). The second-request system model implies that the extraction happens in two synchronous cycles. How much separated oil increments quickly with time initially and afterward diminishes gradually with time until arrive at balance (Heri Septya Kusuma and Mahfud, 2016) ; (Ramanathan and Thangarasu, 2019); (Series and Science, 2020). The pace of disintegration for the medicinal balm contained in the strong to an answer can be portrayed by Eq. (1)

$$\frac{dC_t}{dt} = \text{K}\,(C_s - C_t) \qquad \text{(eq. 1)}$$

Where:

K, stands for second-order extraction rate constant (L/g. min); C_s stands for the concentration of thyme oil at saturation in (g/ L) and C_t is the concentration of thyme oil at any time t (min).

The law of second-order mass transfer model integrated via regarding the initial boundary conditions like time = 0 to t, C_t = 0 to Ct as:

$$\frac{1}{C_s - C_t} - \frac{1}{C_s} = \text{Kt} \qquad \text{(eq. 2)}$$

$$C_t = C_s - \frac{C_s}{1 + C_S Kt} \qquad \text{(eq. 3)}$$

Eq. (3) could be changed back into linear form as follows:

$$\frac{\text{t}}{C_t} = \frac{\text{t}}{C_s} + \frac{1}{K\,C_S^2} \qquad \text{(eq. 4)}$$

k would be identified via plotting a graph t versus *t/C_t*. And the initial extraction rate, h, as Ct/t while approaches 0, coud be erpresed as follows:

$$\text{h} = K\,C_S^2 \qquad \text{(eq. 5)}$$

Then, the focus of thyme oil at any moment could be explained in the following:

$$C_t = \frac{t}{\frac{t}{C_s} + \frac{1}{h}} \quad \text{(eq. 6)}$$

Activation energy

Activation energy is the base measure of energy is needed to response happen. Arrhenius condition is utilized to ascertain the actuation of the energy of oil extraction concerning temperature. The impact of temperature on the rate steady can be resolved utilizing following condition:

K=K0 exp(- Ea/RT) (eq. 7)

while k stands for the extraction rate steady (L/g. min), k0 the temperature-free element (L/g.min), Ea the enactment energy for the extraction (kJ/mol), R the gas steady (8.314 J/mol. K) and T is the outright suspension temperature (K). As displayed in the Arrhenius law, once k0 and k being common for an extraction.

The linearized type of the Arrhenius condition is viewed as in Eq. (8)

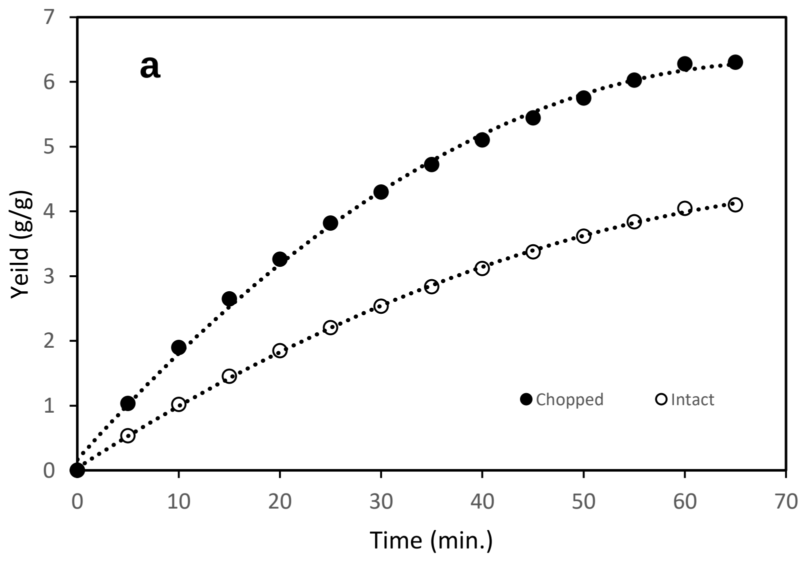
lnK=lnK_0+(- Ea)/R(1/T) (eq. 8)

the enactment energy not entirely settled from the slop by plot (ln k) against (1/T), and the Arrhenius steady not set in stone from the block (Thamsanqa, Basitere, and Vincent, 2021)

Result and Discussion

Impact of thyme leaves size

The impact of molecule size on the extraction yield was considered as unblemished and hacked leaves. In light of this exploration should be visible that the yield of thyme oil got from the slashed leaves is higher when contrasted and thyme oil acquired from the unblemished leaves. Concerning point, diminishing the molecule size of passes on prompts a higher surface region, making extraction more productive. The actual design of the materials is of basic significance, as the extraction proficiency is connected with the capacity of the water fume to diffuse inside the materials. Therefore, the extraction states of similar gathering of rejuvenating balms might contrast starting with one material then onto the next (Heri Septya Kusuma and Mahfud, 2017). By and large, the decrease in extraction yield with expanding molecule size demonstrates that oil was not shipped through the solid cell dividers, and just surface oil was eliminated. Coincidentally, much of the time the impact of molecule size was inspected in the writing by fractionation of the squashed natural substance followed by extraction of the small parts of various size goes independently (Maksimovic, Ivanovic, and Skala, 2012), (Khalid M Abed, Kurji, and Abdul-Majeed, 2015) and (Radivojac et al., 2021) as displayed in figure 3:



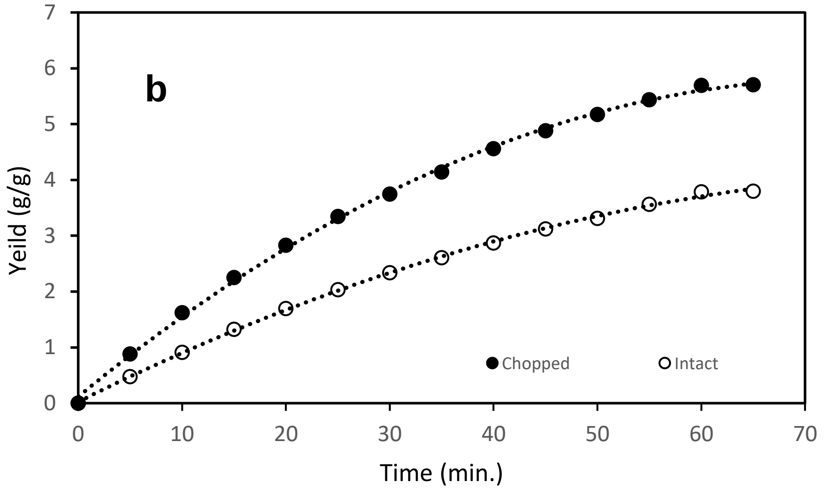
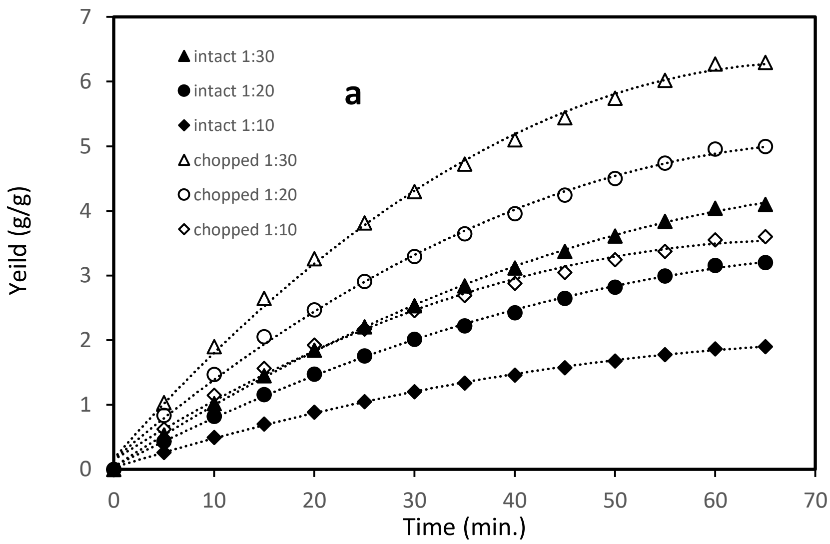


Figure 3. effect of thyme leaves size on the extraction yield at (a) 700W and (b) 500 W.

Impact of solid to water ratio

The impact of the fluid strong proportion on the extraction yields of thyme oil not set in stone. The extraction cycle was completed over a scope of fluid strong proportions (1:10 - 1:30 g:ml) to evaluate the impact of this boundary the outcomes acquired are represented in Figure 4. On account of a lower strong to water proportion, where how much water is less, the oil yield is proclaiming on the grounds that the plant material may have overheated or roasted. For high water content, the hotness could be squandered in warming the water which may have decreased the proficiency of the cycle (Heri Septya Kusuma and Mahfud, 2017) likewise, esters are constituents of medicinal ointments and they will more often than not respond with water to shape acids and to frame alcohols also, and that occurs in the presence of water and with high temperatures. Thusly, the measures of corrosive and liquor will be huge assuming how much water is huge, and that will bring about a diminished yield of medicinal ointment (Hamid, Kurji, and Abed, 2021). A particular constraint of dissolvable to strong proportion is reached, where up as far as possible, an expansion in the oil fixation not entirely set in stone. Great mass exchange is inclined toward in light of the fact that a focus angle between the fluid stage and strong particles are probably going to be more noteworthy (K.Abed, B.kurji, 2018).



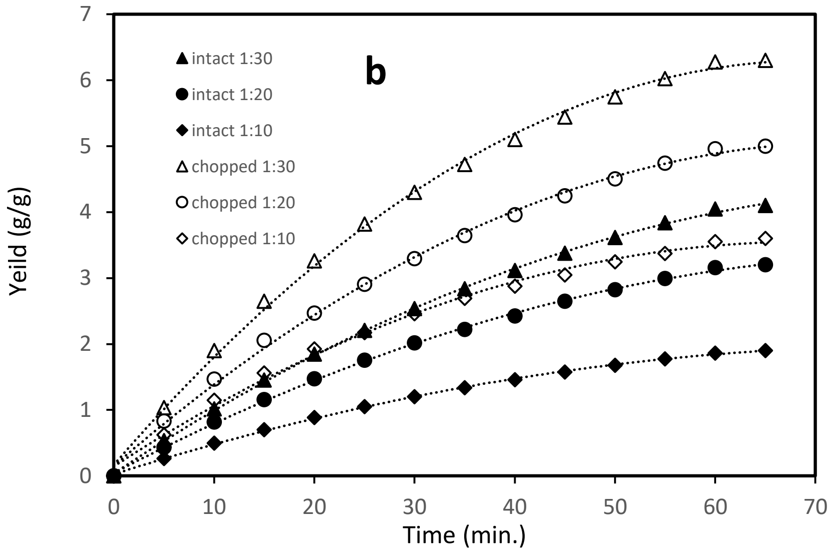


Figure 4. effect of the liquid-solid ratio on the extraction yields of thyme oil at (a) 700W and (b) 500 W.

Impact of microwave power

The extraction interaction was done north of 500 and 700W. Figure 5 shows thyme oil yield increments with microwave power. Microwave power and temperature are connected, on the grounds that the powerful activity can raise the temperature over the edge of boiling over of the dissolvable and produce an expansion in the extraction yield results. Microwave power goes about as a main thrust to separate the construction of plant cell films with the goal that the natural ointment can be diffused out and disintegrated in a dissolvable (Heri Septya Kusuma and Mahfud, 2017) and (Ramanathan and Thangarasu, 2019). Consequently, expanding the microwave power will for the most part further develop the extraction yield and result in a more limited extraction time.

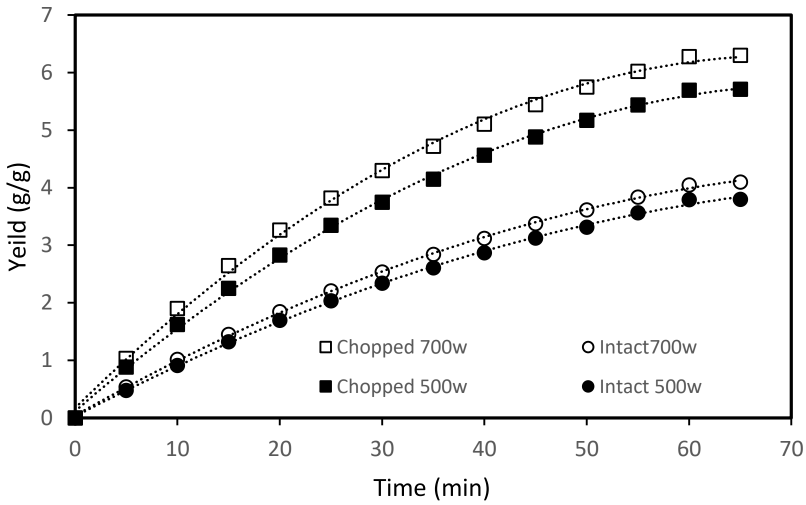
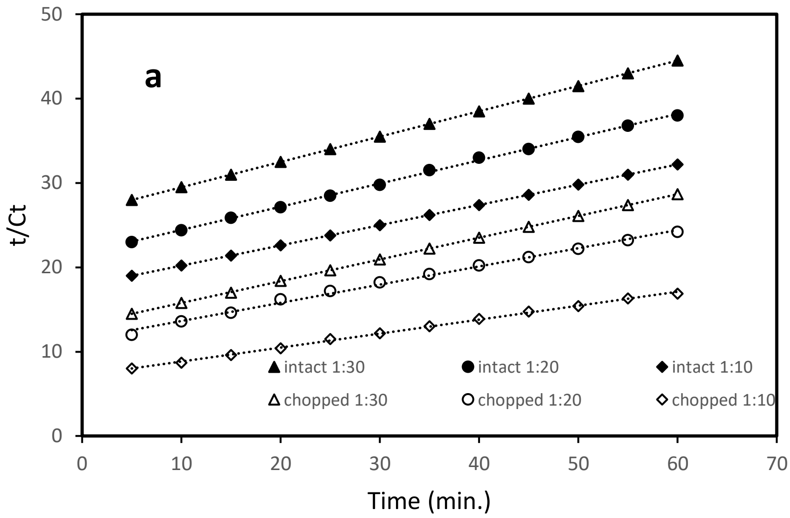


Figure 5. effect of the microwave power on the extraction yields of thyme oil at 1g:30ml

Kinetics of Thymus vulgaris L Oil

Thyme medicinal oil extraction process happens in two progressive ways: (I) disintegration and scouring cycle could be made via the new dissolvable (as a main thrust); (ii) then, at that point, outer dispersion of peppermint oil into the concentrate creating a lot more slow stage. This displays peculiarities run of the mill of a second request motor model. The plotting of t/Ct versus time clarified that peculiarities (Heri Septya Kusuma, 2015), (Heri Septya Kusuma and Mahfud, 2017) and (Khalid M Abed, Kurji, Rashid, and Abdulmajeed, 2019). Figure 6 shows the consequences of this investigation for two the microwave power considered, Moreover, as displayed in Table 1, the extraction limit in the microwave power 700 W was consistently better than that the microwave power 500 W. It was likewise seen that the underlying extraction rate was quicker with the microwave power 700 W than with 500 W. Notwithstanding, the rate steady was higher on account of the microwave power 700 W than in that of the microwave power 500 W, for all sizes of the plant material and strong to water proportion, showing that cell entrance and dissemination are better in the microwave power 700 W.



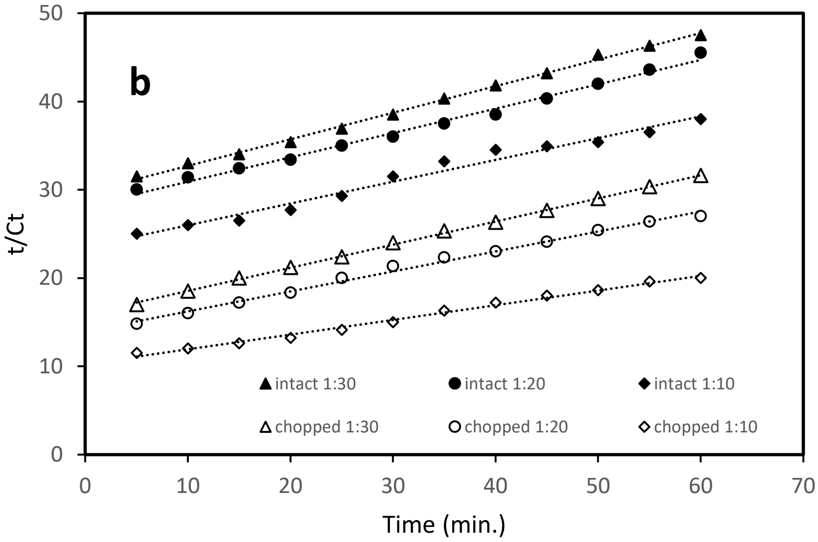


Figure 6. Second-order extraction kinetics of essential oil in microwave power ; (a) 700W and (b) 500 W at the various size of leaves and solid to water ratio.

Table 1. Kinetic of a second-order model of thyme oil extracted from thyme leaves by MAHD at different parameters.

Leaves weight to volume of water ratio (g:ml)	Microwave power	Size of thyme leaves	Cs (g L^{-1})	K (Lg^{-1} min^{-1}) $*10^{-4}$	h (gL^{-1} min^{-1}) $*10^{-2}$	R^2
1:10	500 W	Chopped	5.995	27.130	9.750	0.992
		Intact	4.050	25.921	4.252	0.9789
	700 W	Chopped	6.038	38.219	13.936	0.9985
		Intact	4.166	32.360	5.617	1
1:20	500 W	Chopped	4.413	36.784	7.163	0.9936
		Intact	3.629	26.935	3.548	0.9924
	700 W	Chopped	4.631	40.591	8.707	0.9954
		Intact	3.639	34.820	4.610	0.9992
1:30	500 W	Chopped	3.809	43.304	6.284	0.9992
		Intact	3.323	30.490	3.367	0.9974
	700 W	Chopped	3.875	50.420	7.574	1
		Intact	3.333	33.960	3.772	1

Gas Chromatography-Mass Spectroscopy

Procedure and specifications

As mentioned above, GC-MS (Agilent (7820A) USA) was used to analyze and identify the extracted *Thyme vulgaris* essential oil. The obtained oil, from the Microwave Hydrodistillation method, was injected in the GC-MS to result in approximately 33 compounds, account for 96.9% of the total essential oil. As is illustrated in Table 2, the main constituents of Thyme oil are thymol (43.78%), endo-Borneol (9.87%), α- Terpineol (4.47%), p-cymene (1.8%) and γ-terpinene (6.79%). In comparison with different researchers, the level of these synthetic organizations are somewhat unique and that because of the different climatic states of the world which change in the different topographical locale of the world. Amount may likewise change because of the wellbeing and assortment of the plant, age of the plant at the hour of assortment, technique for drying and strategy for extraction of the oil from tests.

Table 2. Chemical composition of the essential oil from Thyme leaves

Compound	Content %
α- Pinene	0.61± 00.0
Camphene	1.03±
2-Carene	0.64±
p-Cymene	1.8±
γ-Terpinene	6.79±
1,6-Octen-3-ol	0.87±
Endo-Borneol	9.87±
Terpinen-4-ol	0.78±
α- Terpineol	4.47±
Thymol	43.78±
Caryophyllene	4.09±
Humulene	0.41±
Beta-Bisabolene	1.86±
Ar-tumerone	2.26±
Heptadecane, 1-bromo-	1.97±
Octacosyl trifluoroacetate	1.42±
Sulfurous acid …..ester	2.37±
Tetraentacontane …….	2.05±
2-Piperidinone, N-[4-bromo-n-butyl	1.08±
Octatriacontyl pentafluoropropionate	2.16±

Add the abbreviations same as above ….

Conclusion

To sum up, in this paper, Thyme medicinal oil was acquired by utilizing the Microwave Hydrodistillation strategy. The airborne pieces of thyme passes on utilized in two sorts to extricate the oil: hacked and unblemished, which were oppressed in a Clevenger-type device utilizing refined water was as a dissolvable. Many trials have been examined to concentrate on the impacts of three distinct boundaries: the impact of the leaves size of the material as flawless and cleaved leaves, the impact of unrefined substance weight to the volume of dissolvable proportion (1:10, 1:20 and 1:30g/ml) and the impact of microwave power (500 and 700W. In this review, the ideal acquired conditions were at 1hr as the extraction time for 700w microwave power, dissolvable to strong proportion of 1g plant leaves/30ml dissolvable for the hacked plant. The energy of the separated thyme EO was considered in light of a second-request model. Thus, it tends to be inferred that the component of the EO extraction continues in two stages: a quick disintegration of peppermint oil followed by sluggish outer dissemination of solute from the plant leaves.

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