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# ASPECTS REGARDING THE EXTRACTION OF MENTHOL ESSENTIAL OILS IN HIGH FREQUENCY ELECTROMAGNETIC FIELD

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**Key words: Microwave extraction, Essential oil, Mentha crispa, Mentha piperita.**

**The extraction of essential plant oils through conventional methods has been used for over a century, but the use of high frequency electromagnetic field energy has been experimented as an alternative distillation method that: would reduce water consumption, reliance on non-renewable energy and very important energy sources impact on the environment. In this paper is studied the extraction microwave method of the essential oil content from two species of peppermint (mentha crispa *vs* mentha piperita), the differences between the two types of species and the yield. A by-product of the extraction of essential oils in the microwave field observed during the experiments is the creation of aerosols. During the experiment the ethyl alcohol ($C_2H_5$ -OH) was used as extraction solvent. In order to obtain a high efficiency and to avoid the occurrence of phenomena that would affect the integrity of the variable-power microwave generator, the system will operate in pulses, method that allows the achievement of frequencies that facilitate breakage of chemical bonds of the components.**

## 1. INTRODUCTION

Medicinal plants have been used since ancient times as medicines to treat a wide range of conditions. In our days, modern methods allow scientists to use plants in order to cure many diseases. It was demonstrated that menthol produces a mild anaesthetic of the gastric mucosa, which is why the mint preparations have antiemetic action, stimulates secretion and elimination of the bile due to the presence of flavonoid compounds [1, 2]. Mint presents also anti-fermentative and disinfecting properties mainly due to tannins, as well as spasmolytic properties [3]. Menthol applied locally has antiseptic, analgesic and decongestant effect on the airway. Also the mint oil is one of the most popular essential oil, being mainly used for menthol as a flavour for pharmaceutical products and oral preparations [4]. There are some studies that show that the mint menthol has anti-cancer properties.

Based on morphological and genetic features, mint plants have recently been classified into 18 species, 11 hybrids and hundreds of subspecies, types and varieties, mint plants being characterized by high morphological variation and natural hybridization between species [5–7]. The plants are perennial, grow fast and tolerate a wide range of agro-climatic conditions with distribution on all continents.

The economic importance of the mint is obvious because essential oil extracted from dry or fresh vegetable material is used daily as part of the pharmaceutical, food, cosmetic, tobacco and pesticide manufacturing [7] products. Mint plants are scented by carvone that are a member of a family of chemicals called terpenoids, found naturally in many essential oils.

An increase in world trade in essential oils extracted from mint is expected to continue to expand in the future as a result of the growing number and consumer preferences and the very broad spectrum of uses of these compounds [8]. From this reason, in 2014, the world mint production reached 5630 Mt and continue to rise, without many statistic reports published on mint production and use of mint as prime material [9, 10].

The latest reports have estimated world production of menthol at 9 400 Mt [11]. The main exporting countries are India and China. Most of the production (9 400 Mt) comes from the extraction of oil from dry material, majority of this oil coming from India [12].

The main objective followed in these experiments is to highlight the possibilities of extraction of volatile oils from the two mentha crispa / mentha piperita species by using high frequency electromagnetic field, and to point out the differences between the two types of species, the yield, a qualitative comparison and the comparisons of main process parameters. For the extraction of essential oils, during experiments dry leaves from those two species were used.

Advantages of the microwave heating allowed the researchers to design, test and implement new processes for industrial use [13, 14], with main application for different materials processing in high frequency electromagnetic field [15, 17].

Technical literature presents different calculation methods, computer solvers and different experimental equipments used for mathematical and physical analysis of material processing in high frequency electromagnetic field. This complex process are discussed in [18–22]

We intent to establish a complex system that allows the microwave extraction of volatile oil from two species of mint: mentha crispa and mentha piperita – to be able to use it as an active aid in the design and optimization of the microwaves devices and processes used for the extraction of volatile oil from different samples.

## 2. EXPERIMENTAL PROCEDURE

The microwave systems use high frequency electromagnetic waves to transmit energy at a frequency of 2450 MHz. The waves from this frequency range are absorbed by various environments such as water, fats, sugars, and other molecules. In order to increase the efficiency, the variable-power microwave generator will operate in pulses, method that allows to achieve the right frequency that facilitate breakage of chemical bonds of the components with respect to the quality regulations for the extracted oil [23–25].

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In the specific case of ethyl alcohol ($C_2H_5 - OH$) which is an electrical dipole, positively charged at one end and negatively charged on the other, once with the induction of the electric field, the molecules will line up inside the vessel. Through this movement and the collision with other molecules will produce heat that allow the breakage of existing linkages in the substrate. The alcohol used as solvent during extraction also presents the advantage of having a lower boiling point and this reduces the risk of destruction of the sample through overheating [26, 27].

When microwaves excite molecules, that will produce a rotational and translational movement, resulting heat dissipation. However, high frequency electromagnetic waves will unevenly penetrate the material used for undergoing experiments [28]. It is known that extracted mint oil is soluble in both water and ethyl alcohol, but the recovery yield is higher for the ethyl alcohol, which has the advantage of having the boiling point lower, and there is no risk of destruction of the sample.

Microwave energy is an advantageous alternative to the vast majority of thermal applications due to efficient volumetric heat generation [28]. Volumetric heating as opposed to heat transfer from the outer surface to the inside is more efficient and uniform. Fast heat control and transfer are the biggest advantages of high frequency electromagnetic waves compared to conventional methods. During experiments, the ability to instantly shut off the heat source makes product quality difference.

The experiments were carried out on two different types mentha crispa and mentha piperita by using the ethyl alcohol ($C_2H_5 - OH$) as solvent. The microwave system used for the performed experiments is provided with a variable microwave power 0÷3 kW, and is able to collect the alcohol vapours ($C_2H_5 - OH$) emitted from the undergoing sample used for experiments (Fig. 1).

Experimental samples consisting in 150 g dried mint leaves, exposed to a maximum applied microwave power that has reached 1 kW value, and the maximum time of a sample subjected to the experiments was 20 minutes. The purpose of the experiments was to extract the volatile oil from the two species and to compare the two types of extracted oils as well as the extraction yield in the microwave field and the use as a solvent of the $C_2H_5 - OH$ ethyl alcohol (Fig. 2).

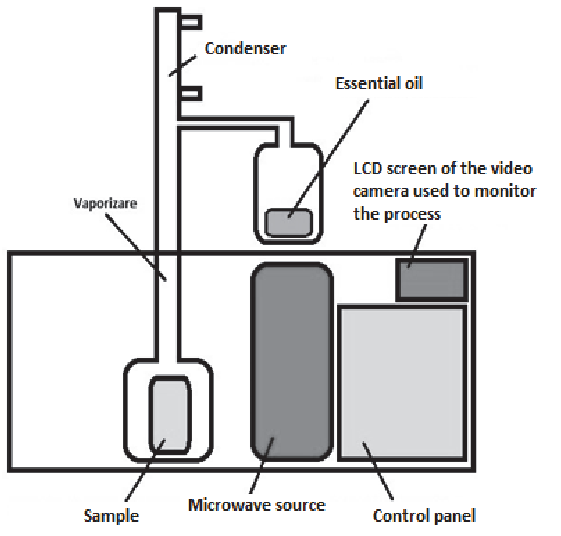


Fig. 1 – The schematic diagram of the microwave extraction proces of mint essential oil.

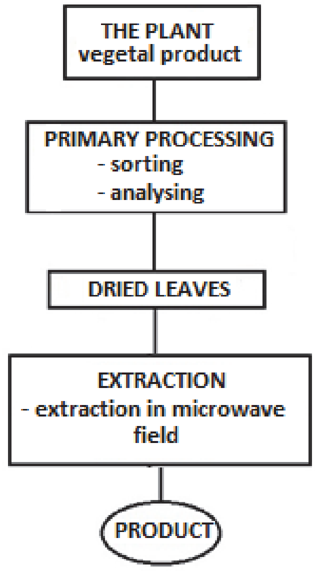


Fig. 2 – Valuation scheme.

The ethyl alcohol is a solvent that has a high dissolving capacity of organic substances and minerals, allowing greater dissolution of volatile oils. The same concentration of alcohol and demineralized water was used to prepare the solution used to perform the experiments.

The samples subjected to the experiments were cut with a kitchen blender, with no visible granulation differences, in order to allow the solvent to come into contact with a larger surface of the material inserted into a special paper filter, placed into the extraction space.

## 3. THE ELECTROMAGNETIC FIELD FORMULATION

In order to describe and compute the complex electromagnetic field problem for the considered application, a mathematical model must be associated to the microwave extraction method. In the following is presented the sinusoidal approach used in order to solve this type of complex problems. The guiding structures used in this application presents a rectangular shape with conductive boundaries. The accurate knowledge of the electromagnetic field distribution inside the microwave applicator makes possible the knowledge of propagation constant, its dependence on frequency and propagation speed and phase and attenuation constants.

The numerical computation of a parallelepiped resonant cavity will presume to solve the electromagnetic field problem as is described [29]:

$$\mathrm{rot}\,\nu\,\mathrm{rot}\,\underline{\boldsymbol{E}} - \omega^2 \varepsilon \underline{\boldsymbol{E}} = 0 \tag{1}$$

is solved by using the Galerkin technique together with the implementation of the finite element method.

It was considered $\underline{\boldsymbol{E}}$ as the electric field strength, ω the angular frequency ($\omega = 2\pi f$) and ε the dielectric constant

We suppose $\underline{\boldsymbol{E}}$, as being:

$$\underline{\boldsymbol{E}} = \underline{\boldsymbol{E}}_0 + \sum_{k=1}^{N_1} \underline{\alpha}_k N_k + \sum_{k=1}^{N_2} \underline{\beta}_k (-\,\mathrm{grad}\, V_k) \tag{2}$$

and the condition,

$$\mathrm{div}\,(\varepsilon\, \underline{\boldsymbol{E}}) = 0\,.$$

It was considered as well $N_k$ as the vector form functions with the following property: rot $N_k$ are linearly independent and $N_k = 0$ on $\partial\Omega$. The considered boundary conditions on $\partial\Omega$ are $V_n = 0$, and

$$\underline{\boldsymbol{E}}_0\big|_t = \underline{\boldsymbol{f}}\,.$$

Equation (1) is projected on the vector form functions and solving it is obtained the numerical equation (a valid equation for $\underline{\boldsymbol{H}}_t = 0$), the Neumann conditions:

$$\int_\Omega \nu \,\mathrm{rot}\, N_k \,\mathrm{rot}\, \underline{\boldsymbol{E}}\, \mathrm{d}\Omega = \omega^2 \int_\Omega N_k \varepsilon \underline{\boldsymbol{E}}\, \mathrm{d}\Omega \tag{3}$$

where $k = 1, 2, \ldots, N_1$.

Projecting equation (2) on the $V_k$ functions and integrating by parts is obtained:

$$\int_\Omega \varepsilon\, \underline{\boldsymbol{E}}\, \mathrm{grad} V_k \, \mathrm{d}\Omega = 0 \tag{4}$$

where $k = 1, 2, \ldots, N_2$ .

By using equations (3) and (4) results a system of ($N_1$+$N_2$) equations, that is capable to solve numerically the electromagnetic field problem inside the interior of the microwave applicator [29].

The only problem that occurs now is the choice of the $N_k$ and $V_k$ functions. Usually, the most convenient way to define the $N_k$ functions is given by the vectorial nodal elements or the edge elements, as well as for $V_k$ functions it is useful to use nodal elements.

The accurate knowledge of the dielectric properties is necessary for the development of new researches and passing to the large scale processing by using the microwaves in industrial processes.

The dielectric materials, as most of the vegetal products, converts the electric energy into heat at the microwave frequency. The temperature will increase for each specific sample due to dielectric heating and can be computed by using following equation [29]:

$$\rho \cdot C_p \cdot \frac{\Delta T}{\Delta t} = 5.563 \cdot 10^{-11} \cdot f \cdot E^2 \cdot \varepsilon'' \tag{5}$$

where $\rho$ is the material density (kg/m$^3$), $C_p$ is the specific heat (J·kg$^{-1}$·grad$^{-1}$), $\Delta t$ – time interval (s), $\Delta T$ – temperature variation (°C), $E$ – electric field strength (Vm$^{-1}$), $f$ – frequency (Hz) and $\varepsilon''$ is the dielectric loss factor.

## 4. RESULTS

The experiments, consisting in two cases for each sample mentha piperita and mentha crispa were performed under the same conditions. In the first case the applied microwave power was raised to 1 kW, and in the second case the sample under experiment was exposed to high frequency electromagnetic field applied in pulses for a 750 W power.

The temperature during the extraction process of volatile oil has been measured and recorded through an infrared thermometer. The Erlenmeyer flask in which the extracted liquid was stored has been let for five minutes in order to minimize vapour losses, so as will ensure that all vapours are condensed.

### 4.1. CASE I

First case of analysis presumes that the consisting mentha piperita sample has been exposed to the continue microwave radiation with it power of 1 kW for a period of 20 minutes. The obtained results presents information regarding the process (Table 1) pointing out the influence of the extraction time upon the extracted quantities for each time step.

*Table 1*

Experimental results obtained for the extraction of volatile oils from mentha piperita in high frequency electromagnetic field and 1 kW power

| Microwave power [W] | Weight of the sample [g] | Time step /Time [min] | Temperature [°C] | Extracted solution [ml] | The alcohol quantity in the extract [ml] |
|---|---|---|---|---|---|
| 1.000 | 150 | 2 / 5 | 45 | 12.6 | 8.3 |
| | | 3 /10 | 55 | 23.2 | 14.5 |
| | | 4 /15 | 65 | 38.4 | 23.7 |
| | | 5 /20 | 75 | 53.8 | 39.2 |

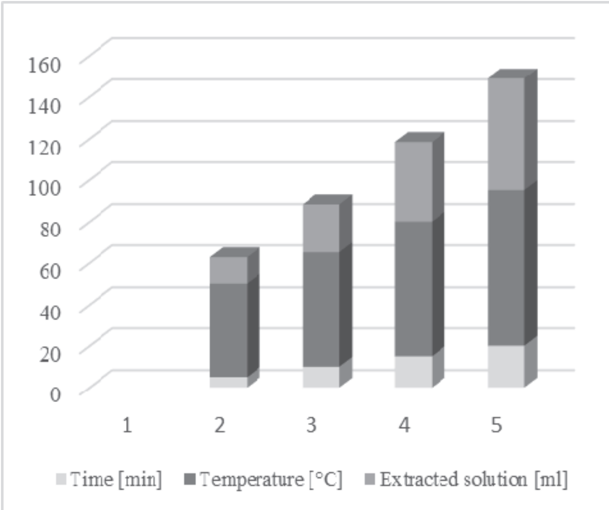


Fig. 3 – The extracted oil quantity depending on temperature for mentha piperita. at each time step.

The experimental results presented in Figs. 3 and 4 point out the extracted solution oil and solvent quantity for mentha piperita in the first studied case.

The test tube mass in which the sample undergoing the experiments was placed, was recorded to determine the weight change of the sample after extraction of the oil. The mint sample was collected from the test tube and subjected to a moisture analysis.

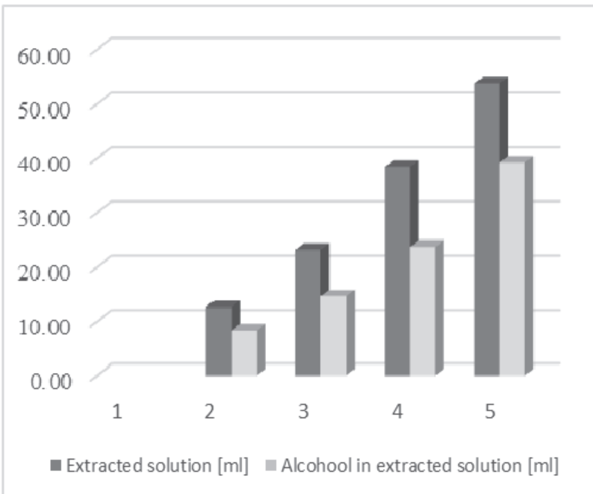


Fig. 4 – The amount of oil / ethyl alcohol in the extract for mentha piperita. at each time step.

### 4.2. CASE II

The second case of analysis presumes that the mentha piperita sample is exposed to a 750 W microwave power applied in pulses. During the experiments, the period of the applied pulse was variable in accordance with the values presented in Table 2. By using the microwave pulse model in the consider experiments are being created the conditions for reaching the corresponding frequency for breaking the chemical bond in the supposed tested sample. Table 2 points out the influence of extraction conditions upon the extracted solution for different time steps.

*Table 2*

Experimental results obtained for the extraction of volatile oils from mentha piperita in high frequency electromagnetic field and 0.75 kW power

| Microwave power [W] | Weight of the sample [g] | Time step /Time [min] | Pulses applied / min | Extracted solution [ml] |
|---|---|---|---|---|
| 750 | 150 | 2 / 2 | 10 | 2.1 |
| | | 3 / 4 | 15 | 4.3 |
| | | 4 / 6 | 20 | 10.5 |
| | | 5 / 8 | 25 | 15.6 |
| | | 6 / 10 | 30 | 20.4 |
| | | 7 / 15 | 35 | 35.2 |
| | | 8 / 20 | 40 | 51.7 |

Figure 5 presents the experimental results in terms of extracted solution, obtained for the second experimental case, when the sample of mentha piperita was exposed to high frequency electromagnetic field applied in pulses.

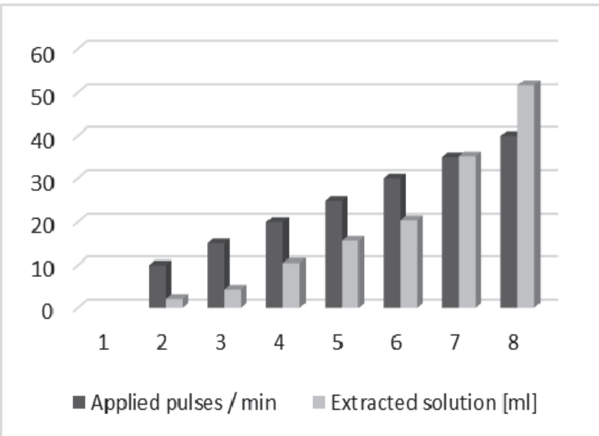


Fig. 5 – The amount of solution extracted according to the application time of the high frequency electromagnetic field for mentha piperita. at each time step.

During the experiments, the collected extract was kept warm by means of a heating nest in order to be held near the optimal separation temperature of about 30 °C. The extracted liquid was then separated in oil and alcohol by using a separating funnel and stored in a Berzelius beaker, so as to determine the mass of alcohol collected. The oil was left in the separating funnel and collected using a pipette and stored in an evaporation vessel where we could determine after the amount of extracted oil. Several tests were required to determine the optimum power, temperature, purity of the extract and the concentration of ethyl alcohol used to achieve maximum yield in the extraction process.

## 4.3. CASE III

In the following, we highlight the experimental results performed under the same condition as the ones presented in case I for a sample of mentha crispa. The obtained result regarding extraction parameters for each time step are presented in Table 3 and Figs. 6 and 7.

*Table 3*

Experimental results obtained for the extraction of volatile oils from crispa mint in high frequency electromagnetic field and 1 kW power

| Microwave power [W] | Weight of the sample [g] | Time step /Time [min] | Temperature [°C] | Extracted solution [ml] | The alcohol quantity in the extract [ml] |
|---|---|---|---|---|---|
| 1000 | 150 | 2 / 5 | 45 | 11.2 | 6.1 |
| | | 3 /10 | 55 | 20.4 | 11.4 |
| | | 4 /15 | 65 | 35.7 | 21.3 |
| | | 5 /20 | 75 | 49.5 | 37.6 |

By analysing the experimental data it can be observed that there is a small difference between the amount of oil extracted from the mentha piperita and mentha crispa subjected to the experiments.

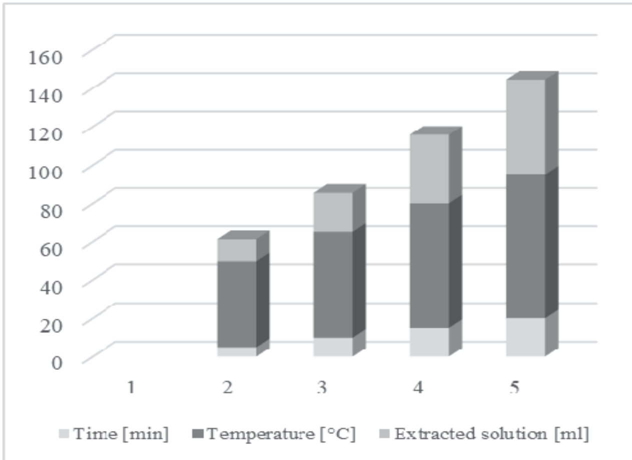


Fig. 6 – The extracted oil quantity depending on temperature for mentha crispa at each time step.

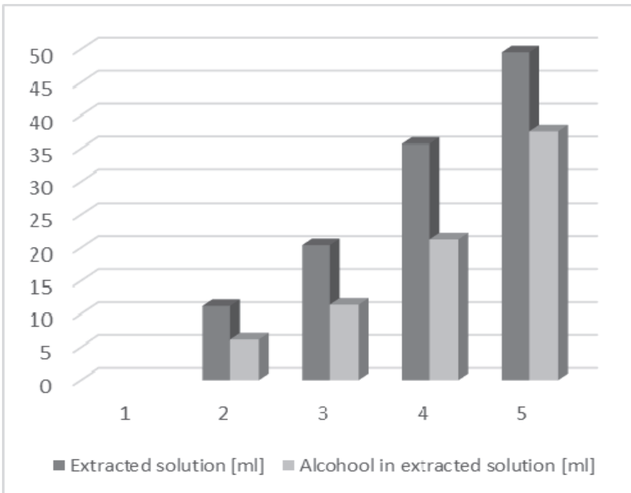


Fig. 7 – The amount of oil / ethyl alcohol in the extract for mentha crispa. at each time step.

## 4.4. CASE IV

The fourth case of analysis presumes that the considered mentha crispa sample is exposed to a 750 W microwave power applied in pulses under similar experimental conditions with the second case presented above. The obtain result with respect to extraction process are presented in Table 4 and Fig. 8 for each time step.

Figure 9 presents the comparing with respect to the extracted quantities between the results obtained for both species.

*Table 4*

Experimental results obtained for the extraction of volatile oils from crispa mint in high frequency electromagnetic field and 0.75 kW power

| Microwave power [W] | Weight of the sample [g] | Time step /Time [min] | Pulses applied / min | Extracted solution [ml] |
|---|---|---|---|---|
| 750 | 150 | 2 / 2 | 10 | 1.8 |
| | | 3 / 4 | 15 | 3.9 |
| | | 4 / 6 | 20 | 9.2 |
| | | 5 / 8 | 25 | 14.3 |
| | | 6 / 10 | 30 | 19.5 |
| | | 7 / 15 | 35 | 33.8 |
| | | 8 / 20 | 40 | 48.7 |

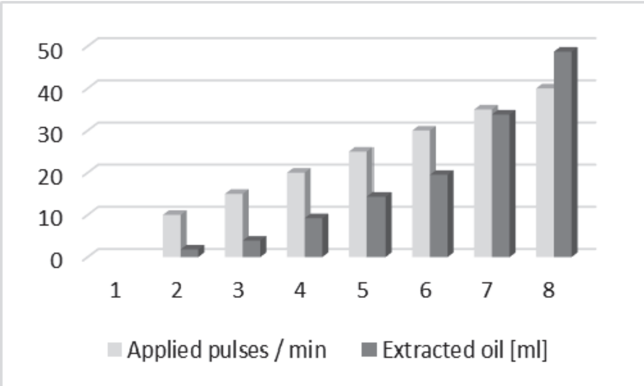


Fig. 8 – The amount of oil extracted according to the application time of the high frequency electromagnetic fields for mentha crispa. at each time step.

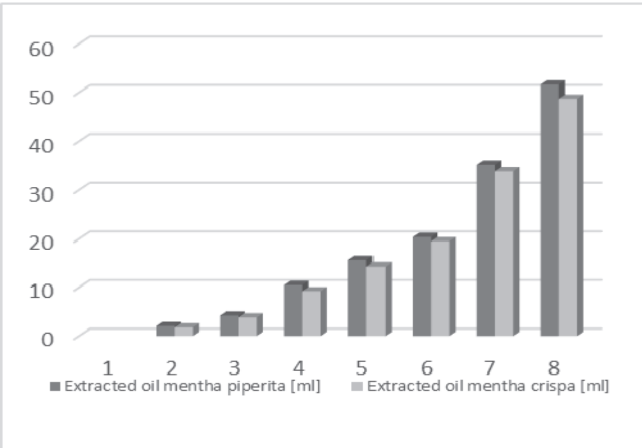


Fig. 9 – Amount of oil extracted from mentha piperita versus mentha crispa.

Analysing the obtained results for both samples, it was observed that between the two studied species, the extracted oil from mentha piperita presents the advantage that contains a higher menthol concentration, higher density, higher viscosity and optimum yield in oil extraction. In order to assure a better quality and a better extraction yield, the experiments were performed by using dried leaves and stalks harvested at the beginning of September. A microscopic view of the obtained oil is presented in Fig. 10 for both type of mentha. Figs. 10 a, b, c and d presents the microscopic view of the extracted solution from samples obtained at the beginning and the end of process for mentha piperita and menthe crispa.

The chemical composition of the oil and the extracted alcohol from the samples subjected to the experiments was analysed using several techniques in order to determine the chemical composition of the resulting solution (oil + alcohol). One of them is the gas chromatographic analysis of the resulting solution. The mixture being a heterogeneous (liquid + liquid), the separation was accomplished by decantation by means of a separating funnel

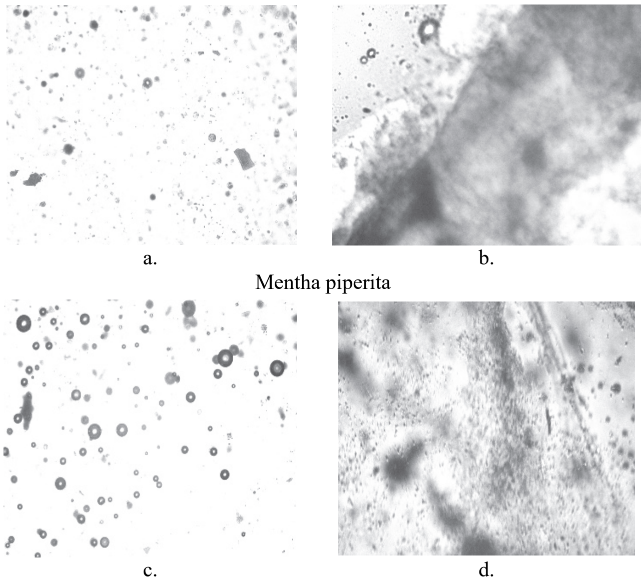


Fig. 10 – Microscopic view of the extracted volatile oil.

## 5. CONCLUSIONS

The performed experiments studied variables such as the applied power to the material undergoing the experiments, the extraction time, the duration of the applied pulses and their quantity / minute. The settings that gave the maximum yield in the ratio of power / quantity of the extracted oil: 1000 W for continuous magnetron operation and 750 W for impulse magnetron operation were chosen.

The minimum and maximum extraction times varied for different power settings. The extracted samples were collected and analysed in the chromatographic system in order to determine its composition. Some of these components like the total concentration were chosen to compare the quality of the oil between the two hybrids subjected to the experiments.

In all performed experiments it was observed a variation in the extracted oil rated from 5 % to 17 %. This variation is due, in part, to the small amount of material under test (the test sample contains 150 g of mint leaves) and the extracted solution (oil + alcohol) which is obtained, due to the fact that in the condensation zone can occur a considerable increase in experimental error. Also, leaf / leaf tail ratio in the used samples causes more variability than it would have been in the case for some larger samples. The variation in the amount of extracted alcohol was found to be less than that of the extracted oil. During experiments it was found that the volatile oil is available only in the leaves of the two studied hybrids in contrast to the tails of leaves, where the amount of oil is insignificant.

Since only a small amount of the extracted liquid is volatile oil, it can be assumed that the boiling point of the mixture is that of the ethyl alcohol. The final sample temperature after extraction shows that the temperature obtained during extraction process it reached at the maximum 75 °C. In the first part of the extraction process, a higher quantity of energy was needed until the alcohol started to evaporate. In the second part a lower quantity of energy was needed just to keep the temperature high enough to help the transport of the vapours. The required time to reach 75 °C for 1000 W was 3.2 minutes.

Experimental results have shown us that the most efficient power setting was for 1000 W. This setting allows extraction of 72.6 % of the total oil available in the plant with a variation coefficient of ± 3 % from the alcohol used as the solvent. The percentage of fluids remaining in the sample subjected to the experiments is 27.4 %. Figure 10 presents images of the extract from the beginning and the end stage of the experiments.

The composition of extracted oil varies in some experimental phases more than in others. In all stages, the concentration of menthol and other components is proportional to the extraction time. Their maximum concentration is reached for longer extraction time, usually in the last two extraction periods. In all cases, the concentration of menthol and other components decreases as the extraction time increases. Since the main component of the peppermint oil is menthol, it is desirable that the extraction time and the power setting that allow the concentration to have maximum yield.

During the experiments, oscillations of reflected power were found. This phenomenon proved to be more pronounced at high temperature values, indicating changes in the sample constants ie dielectric permittivity (ε') and the loss factor (ε").

The main advantage of the presented method consists in the fact that can be applied with success to various aromatic plants.

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