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# Microwave extraction of mint essential oil – Temperature calibration for the oven

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ABSTRACT

The efficiency of the microwave extraction process is linked to the control of many parameters, such as, temperature and extraction time. The objective of this work was to calibrate the microwave reactor unit for uses in different conditions of temperature and time of extraction. The research also includes a microwave extraction as an application of the procedure developed. Ethanol and hexane (3:7 v/v) were used as the solvent and the calibration was based on the calorimetric method. Microwave extraction was carried out using mint dried leaves and its essential oil was analyzed by GC/MS. As a result, time of extraction could be predicted for adjusted models – rational and linear equations, correlation coefficients of 0.998 and 0.999, respectively – and temperature maintained stable in the microwave oven unit, during extraction. Carvone was the major compound obtained by microwave extraction (0.011–0.091% d.b.).



## 1. Introduction

Mint is the common name for members of the Labiatae family, which includes many species, varieties, and hybrids. While it is a very popular plant around the world, it is native to the Mediterranean region (Doymaz, 2006; Park et al., 2002). The mint leaves are oval with serrated edges and are rich in aromatic oil; and its stalks are purple. There are many species with commercial and medicinal purposes, among them are mint (*Mentha spicata* Huds), peppermint (*Mentha arvenis* L. or *Mentha* x *piperita* L.), black mint (*Mentha vulgaris*), white mint (*Mentha officinalis*), spearmint (*Mentha spicata* L.), simple mint (*Mentha piperita* L.), pennyroyal (*Mentha pulegium* L.) and garden mint (*Mentha crispa* L.) (Park et al., 2002).

The essential oil of mint can have different compositions and chemotypes and this is the reason why single or hybrid species may have different smells, and the essential oils of the species could be altered just by changing in the chemical structure of the monoterpenes (Ringer et al., 2005). The essential oil is a mixture of many volatile compounds, which are influenced by environmental factors (interaction between the genotype and environment, irrigation method, time of harvest, season, and others) and processing parameters such as temperature, time, pressure, and method of extraction (Maia and Zoghbi, 1998; Verma et al., 2010).

Some researchers have reported biocidal activity for some varieties such as *Mentha piperita* (Ansari et al., 2000; Hori, 2003), *Mentha microphylla* (Papachristos and Stamopoulos, 2002), *Mentha viridis* (Papachristos and Stamopoulos, 2002), *Mentha pulegium* (Toloza et al., 2006); that is, the essential oil of mint could be effective against arthropod species (Kumar et al., 2011). Moreover, the oil is widely used in mouth fresheners, candies, treatment of diseases, etc. (Dwivedi et al., 2004; Nerio et al., 2010).

There are several extraction methods for essential oils such as pressing, *enfleurage* and distillation. However, the literature reports that these methods can affect the final product quality, due to losses of some volatile compounds during the procedure, low extraction efficiency, and degradation of unsaturated compounds due to thermal effects or solvent used in the extract (Shreve and Brink, 1997; Bayramoglu et al., 2008). Nowadays, new techniques are available that can reduce loss of bioactive compounds due to long extraction times. Among them, microwave-assisted extraction is becoming a good option in many fields, and especially in research on extractions from medicinal plants (Dai et al., 2010; Chan et al., 2011; Costa et al., 2013).

The process is based on heat generated by ionic conduction and/or dipole rotation, and its efficiency depends on the dielectric properties of the material. The extraction occurs when the water inside the plant absorbs energy coming from the microwaves and increases the pressure inside the material causing the cell structure

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to break allowing the solvent to penetrate into the matrix (Wang and Weller, 2006; Chan et al., 2011; Eskilsson and Björklund, 2000; Routray and Orsat, 2012).

Some parameters can influence the process performance, such as solvent composition, temperature and time of extraction. Among these factors, temperature is the most studied parameter due to the heating of the solvent, which can be above its atmospheric boiling point, thus increasing the efficiency and the speed of the extraction (Eskilsson and Björklund, 2000; Nemes, 2012). However, the temperature effects are not always intuitive and its adequate range for extraction depends on the stability and the target active compound.

Tsubaki et al. (2010) concluded that the optimum temperature for the extraction of phenolic compound from Oolong tea was 170 °C. Font et al. (1998) reported that the extraction of sulfonylurea herbicides at 100 °C provided low yields, due to decomposition of the analytes, and concluded that the extraction temperature had to be below 70 °C to increase the compound yields. On the other hand, the extraction yield of amino acids from cauliflower was not influenced by temperatures exceeding 40 °C (Kovács et al., 1998; Eskilsson and Björklund, 2000).

The conventional protocol for the temperature adjustment in a microwave cavity has two steps: setting the power level to attain the target temperature, and setting the period of time in which to maintain the target temperature, while avoiding overheating of the system. According to Collins and Leadbeater (2007), microwave power has to be directed to the sample during the initial heating step and after that, the power should be decreased such as to hold the reaction mixture at the desired temperature.

The calorimetric method is widely used by manufacturers and researchers to specify the nominal power of microwave ovens. Calibration of the equipment is very important since usually, the actual output power is different from the capacity declared by the manufacturers (Cheng et al., 2006; Swain et al., 2006; Nemes, 2012).

Usually, the microwave power is the parameter used as a controlled independent variable in experiment designs for microwave extractions. This parameter and temperature are interrelated, since the high microwave power increases the temperature of the system, causing variations in the solvent properties, such as viscosity and surface tension, beyond reductions in the extraction yield of thermo sensible compounds.

As the product quality is highly related to the temperature at which the material is exposed, the objective of this work was to calibrate the microwave reactor unit for uses under different temperatures and extraction times.

## 2. Materials and methods

### 2.1. Materials

A focused open-vessel microwave reactor (Star System 2™, CEM Corporation, Matthews, NC, USA) was used for the experiments (Fig. 1). The system mainly consists of a 2.45 GHz microwave generator, a 250 mL glass vessel placed inside the microwave cavity, and a control panel. The equipment has an infra-red (IR) temperature sensor to measure the temperature at the bottom of the glass vessel.

Ethanol (Anhydrous Ethyl Alcohol, Commercial Alcohols, Brampton, ON, Canada) and HPLC grade hexane (Fisher Scientific Co., Ottawa, ON, Canada) were the reagents used in the calibration procedures and for extraction experiments. A mixture using a proportion of 3:7 v/v was used in the calibration experiments and as solvent for microwave extraction of essential oil from mint leaves.

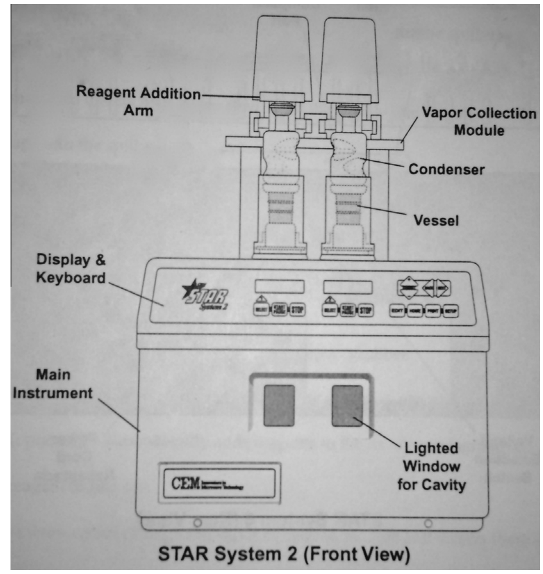


**Fig. 1.** Microwave reactor (manufacturer manual).

Mint was purchased at a local market. The plant material was washed with distilled water, cleaned and diseased leaves were removed. Excess superficial water was eliminated and the plant was dried in the freeze-dryer (Freezone® 2.5 L Freeze Dry Systems, model 7670520, Labconco Corporation, USA) until a constant mass was attained. Finally, the leaves were separated from the stalk, ground in a food processor (Magic Bullet®, model MB1001, Homeland Housewares, USA) and stored inside Mason jars at room temperature (24 °C) and at 30% RH.

Before calibration, the type of solvent and sample-to-solvent ratio had to be defined and they were based on a previous work of Dai et al. (2010). Even with the sample-to-solvent ratio fixed, only solvent was used inside the reactor tube for the oven calibration.

Ethanol was selected due to its ability to extract ingredients from plant matrix and its capacity to absorb microwave energy (dielectric constant$_{(20\,°C)}$ = 24.3; loss factor$_{(20\,°C)}$ = 0.94) (Mandal et al., 2007; Teixeira et al., 2010; Chan et al., 2011). The literature also indicates an option to add another solvent, hexane, to improve the extraction efficiency (Eskilsson and Björklund, 2000; Dai et al., 2010; Desai et al., 2010).

The yields of extracted compounds such as menthone, menthofuran and menthol were increased when a mixture of these two solvents was used. According to Dai et al. (2010) a ratio of 30% (v/v) of ethanol to 70% (v/v) of hexane provided the highest yield for menthone (about 4 mg), menthofuran (about 1.5 mg) and menthol (about 11 mg) for microwave extraction from *Mentha piperita* L. Thus, this was proportion of ethanol to hexane in this study for calibration of the reactor and for extraction process. In the extraction protocol, 2 g of powder sample was used in 60 mL of solvent mixture.

### 2.2. Methodology

#### 2.2.1. Calibration of the microwave reactor

The calibration of the reactor was carried out in two steps. First, heating and cooling rates of the solvent were measured in triplicate. Later, those data was used in the second step to define a time function to maintain the temperature of the solvent at the desired set value.

Heating rate was determined irradiating microwaves towards the solvent placed inside the cavity of the oven for 1 min. The radiation was cut off, the solvent was stirred and the temperature was