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# A comparison of essential oils obtained from lavandin via different extraction processes: Ultrasound, microwave, turbohydrodistillation, steam and hydrodistillation

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ABSTRACT

A total of eight extraction techniques ranging from conventional methods (hydrodistillation (HD), steam distillation (SD), turbohydrodistillation (THD)), through innovative techniques (ultrasound assisted extraction (US-SD) and finishing with microwave assisted extraction techniques such as In situ microwave-generated hydrodistillation (ISMH), microwave steam distillation (MSD), microwave hydrodiffusion and gravity (MHG), and microwave steam diffusion (MSDf)) were used to extract essential oil from lavandin flowers and their results were compared. Extraction time, yield, essential oil composition and sensorial analysis were considered as the principal terms of comparison. The essential oils extracted using the more innovative processes were quantitatively (yield) and qualitatively (aromatic profile) similar to those obtained from the conventional techniques. The method which gave the best results was the microwave hydrodiffusion and gravity (MHG) method which gave reduced extraction time (30 min against 220 min for SD) and gave no differences in essential oil yield and sensorial perception.


## 1. Introduction

Lavandin Grosso (*Lavandula intermedia* var. *Grosso*) is a sterile hybrid of *Lavandula angustifolia Miller* (formely *L. Officinalis*) and *Lavandula latifolia Vill* (formely *L. spica*) [1]. This plant is an aromatic-medicinal species that belongs to the Labiatae (Lamiaceae) family. Lavandin Grosso was selected because of its high essential oil yields [2]. Essential oil from lavandin can be used as a lavender fragrance for cosmetics, fine perfume, shampoos and in other applications such as household cleaners and detergents [3]. In addition, a large range of medical uses for this plant have also been reported. These include antispasmodic, sedative, antihypertensive, antiseptic, healing and anti-inflammatory properties, all of which render it highly appreciated in phytotherapy and aromatherapy [4]. In food manufacturing, lavandin essential oil has been employed in flavoring beverages, ice cream, baked goods and chewing gum [5,6]. Essential oil extraction from this plant can be achieved by hydrodistillation or steam distillation. These techniques take several hours of heating which can cause degradation of the thermo labile compounds present in the starting plant material and therefore odor deterioration [7].

Moreover, with increasing energy prices and the drive to reduce $CO_2$ emissions, universities and industries are challenged to find innovative technologies which can reduce energy consumption, meet legal requirements on emissions and achieve cost reduction and increased quality. For example, existing extraction technologies have considerable technological and scientific bottlenecks to overcome and often require more than 70% of total process energy used. Driven by these goals, advances in microwave extraction have resulted in a number of techniques such as microwave steam distillation (MSD) [8,9], microwave hydrodiffusion and gravity (MHG) [10,11], microwave steam diffusion (MSDf) [12] and solvent free microwave extraction (SFME) [13] being used. Microwave-mediated processes bring a number of advantages to essential oil extraction thanks to their reduced equipment size, ease-of-use, speed, ability to control a process via mild increments in heating and low solvent consumption, all of which contribute to reducing environmental impact and costs. Over the years procedures based on microwave extraction have replaced some of the conventional processes and other thermal extraction techniques that have been used in chemical laboratories for decades.

In this paper, we present a comparative study of the ability of a number of different methods to extract the essential oils from lavandin flowers in order to find the most advantageous in term of extraction kinetics, essential oil quality and quantity.

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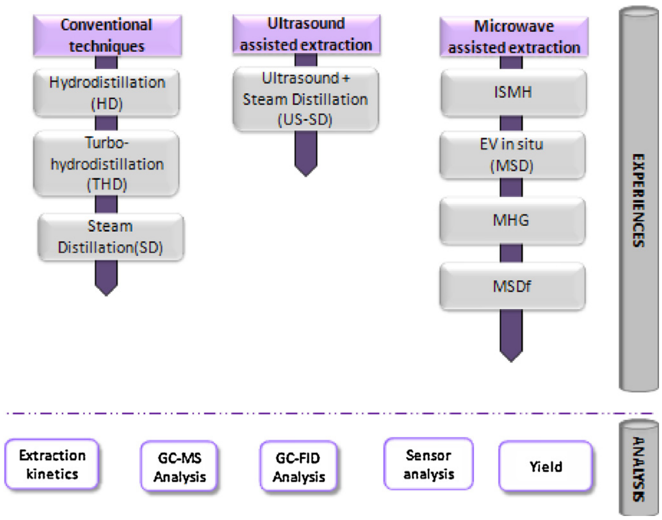


**Fig. 1.** The eight different extraction processes.

## 2. Materials and methods

### 2.1. Chemicals and plant material

Analytical grade anhydrous sodium sulfate was purchased from Fisher Scientific. (Leicestershire, UK). The lavandin (*Lavandula hybribia*) flowers used were collected from the south of France in July 2011.

### 2.2. Extraction procedures

The extraction of the essential oils from lavandin was performed using eight different methods; three classical methods and five innovative methods, as shown in Fig. 1.

#### 2.2.1. Hydrodistillation (HD) apparatus and procedure

250 g of lavandin was submitted to hydrodistillation (Fig. 2a) using a Clevenger-type apparatus, according to the European Pharmacopeia, and extracted with 2 L of water for 240 min (until no more essential oil was obtained). The essential oil was collected, dried under anhydrous sulphate and stored at 4 °C until used. Each extraction was performed at least three times.

#### 2.2.2. Steam distillation (SD) apparatus and procedure

To facilitate rigorous comparison, the same glassware and same operating conditions were used for conventional steam distillation (Fig. 2b). The vapor produced by the steam generator (with a steam flow rate at 25 g min$^{-1}$) [14] passed through the essential oil rich plant material before being condensed into a receiving Clevenger-type apparatus. The essential oil was collected, dried under anhydrous sulphate and stored at 4 °C until used. Each extraction was performed at least three times.

#### 2.2.3. Turbohydrodistilation (THD) apparatus and procedure

To facilitate rigorous comparison, the same glassware and same operating conditions were used for conventional turbohydrodistillation (Fig. 2c) [15]. In this method, the mixture was continuously agitated with a stainless steel stirrer at 100 rpm. The essential oil was collected, dried under anhydrous sodium sulphate and stored at 4 °C until used. Each extraction was performed at least three times.

#### 2.2.4. Ultrasound assisted extraction followed by steam distillation (US-SD) apparatus and procedure

The ultrasound-assisted extraction experiment (Fig. 2d) was performed using a sonotrode (BS2d34, Hielscher UIP 1000 hd, www.hielscher.com) and a glass system (Legallais, www.legallais-labo.fr). The double-layered mantle (with water circulation) allowed the extraction temperature to be controlled via a cooling system. Lavandin was extracted with 2 L of distilled water over 30 min. An amplitude of 60% was used continuously. After this pretreatment, lavandin was removed, and conventional steam distillation was performed to extract essential oil with the same operating conditions as previously described (Section 2.2.2). The essential oil was collected, dried under anhydrous sodium sulphate and stored at 4 °C until used. Each extraction was performed at least three times.

#### 2.2.5. In situ microwave-generated hydrodistillation (ISMH) apparatus and procedure

The in situ microwave-generated hydrodistillation (ISMH) (Fig. 2e) was performed in a Milestone NEOS microwave extraction laboratory oven (Milestone, Bergamo, Italy, www.milestonesrl.com).

It is a 2.45 GHz multimode reactor, with 10 W increments in delivered power up to a maximum of 900 W and is equipped with Pyrex extraction vessels with a capacity of 1500 mL. Time, temperature and power were controlled. Temperature was measured by an external infrared sensor. In a typical ISMH procedure performed at atmospheric pressure, 250 g of lavandin was heated using a fixed power of 500 W for 60 min after having been soaked in 1 L of distilled water during 10 min. A cooling system outside the microwave cavity condensed the distillate continuously. Condensed water was refluxed in the extraction vessel for the regulation of humidity. The essential oil was collected, dried under anhydrous sodium sulphate and stored at 4 °C until used. Each extraction was performed at least three times.

#### 2.2.6. Microwave steam distillation (MSD) apparatus and procedure

To facilitate rigorous comparison, the same microwave equipment (Fig. 2e), the same power setting and the same time were used in every experiment. An electrical steam generator (with a steam flow rate at 25 g min$^{-1}$) and a condenser placed outside a microwave zone were connected to specific glassware that contained 125 g of lavandin that had been soaked in 500 ml of distilled water for 10 min. The condenser was connected to a receiving Florentine flask, which should preferably be a separating funnel to enable the continuous collection of condensate essential oil and water. Microwaves distended the plant cells and lead to the rupture of the glands and cell receptacles. The steam passed through the sample, evaporating and carrying the essential oil, and directed it toward the condenser and the Florentine flask. The essential oil was collected, dried under anhydrous sodium sulphate and stored at 4 °C until used. Each extraction was performed at least three times.

#### 2.2.7. Microwave hydrodiffusion and gravity (MHG) apparatus and procedure

In a typical MHG procedure (Fig. 2f), 250 g of lavandin (after having been soaked in 1 L of distilled water for 10 min) was heated without the addition of solvent or water. The direct interaction of microwaves with biological water (i.e. steam produced from the water present in the plant material) facilitates the release of essential oil trapped inside the cells of plant tissues. Due to earth's gravity, a mixture of hot "crude juice" and steam (in situ water) naturally moves downwards into a condenser outside the microwave cavity where it is condensed. The oil condensate is collected