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Ultrasonics Sonochemistry 13 (2006) 54–60
Ultrasonics SONOCHEMISTRY
www.elsevier.com/locate/ultsonch

Comparison of distillation and ultrasound-assisted extraction methods for the isolation of sensitive aroma compounds from garlic (*Allium sativum*)

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Received 17 July 2004; accepted 11 December 2004
Available online 2 February 2005

Abstract

A comparative study of traditional simultaneous distillation extraction (SDE), microwave assisted hydrodistillation extraction (MWHD) and ultrasound-assisted extraction (USE) is presented, for the extraction of essential oils from fresh garlic (*Allium sativum*) cloves. Each method is evaluated in terms of qualitative and quantitative composition of the isolated essential oil. The highly reactive sulfur molecules of the garlic volatile fraction show variable response to the different isolation methods. The application of ultrasound for the extraction of the essential oil is considered to cause a lesser damage of thermal-sensitive molecules, thus, providing a better approach of the compounds primarily responsible for the characteristic odor and taste of freshly chopped garlic. All heat-involving isolation procedures have been shown to differentiate the volatile-fraction profile as analyzed by GC–MS. Especially when grouping the compounds into cyclic and acyclic, the percentage concentrations drop from 77.4% to 8.7% for the acyclic while that of the cyclic compounds increase from 4.7% to 70.8%. The observed fact may be attributed to the effect of the heat applied, which changes from harsh thermal treatment (SDE) to short time thermal (MWHD) and room-temperature isolation (USE). The use of USE proves to be crucial in order to provide reliable insight into garlic's chemistry.
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Keywords: Garlic; Sulfur compounds; Extraction; Distillation; Ultrasound; Microwave; SDE

1. Introduction

Garlic (*Allium sativum*) has played an important dietary as well as medicinal role for centuries [1]. Particularly today the medicinal use of garlic is widespread and growing. A wide array of therapeutic effects of garlic such as hypolipidaemic, antiatherosclerotic, hypoglaemic, anticoagulant, antihypertensive, antimicrobial, anticancer, antidote (for heavy metal poisoning), hepatoprotective and immunomodulation have been reported [2]. In addition insect and animal repulsive effects have been mentioned [3–6].

Various preparation techniques of garlic have been used to isolate and finally identify its components that mainly consist of organosulfur compounds. Raw garlic homogenate is the major preparation of garlic that has been subjected to intensive scientific study, as it better resembles the way that garlic is consumed. Raw garlic homogenate is similar to aqueous extract of garlic (filtrate of blended garlic and water). Allicin (allyl 2-propenethiosulphinate or diallyl thiosulphinate) is the major thiosulphinate compound found in garlic homogenate and it is thought to be the principal bioactive compound present in aqueous garlic extract [7,8].

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doi:10.1016/j.ultsonch.2004.12.003

When garlic is chopped or crushed, allinase enzyme, present in garlic, is activated and acts on alliin (present in intact garlic) to produce allicin [9–12]. The water extract of heat-treated garlic contains mainly alliin due to allinase inactivation by heat. The major compounds present in aged garlic extract are S-allylcysteine and S-allylmercaptocysteine [13,14].

Medicinally used garlic oil is mostly prepared by hydrodistillation of raw garlic homogenate. The garlic oil is insoluble in water, therefore can be separated or extracted afterwards. Garlic oil consists of the diallyl, allyl methyl and dimethyl mono to hexa sulfides [15–17]. Garlic oil from oil-macerated or ether extracted garlic homogenate contains the 2-vinyl-[4H]-1,3-dithiin and 3-vinyl-[4H]-1,2-dithiin, allyl sulfides and ajoenes (Z and E) [18–20].

The traditional methods for the isolation and purification of chemical constituents from plants tissues, present some disadvantages. Mainly they require long extraction time, big solvent amounts, and some times have low efficiency. Moreover, many natural products are thermally unstable and may degrade during thermal extraction and/or distillation [21,22].

The use an ultrasounic cleaning bath for the extraction of essential oil has shown to diminish the danger of thermal degradation and aid extraction by significantly reducing extraction times. The mechanical effect of ultrasound provides a greater penetration of solvent into cellular materials, via cavitation effects, and improves the release of cell contents into the bulk medium [23,24].

This work describes a comparative study of classical distillation solvent extractions and ultrasound-assisted extraction of garlic cloves, employing different extracting solvents.

2. Experimental

2.1. Plant material and reagents

Garlic cloves were purchased from local farmers, members of the Agriculture Union of N.Vissa. During the project, all the samples were maintained in our lab at appropriate conditions (dark, 25 °C). In each batch 100 g of garlic cloves were blended with 20 mL deionized water for 2 min in a commercial blender. Three different solvents were used for the distillations and the extractions of garlic samples, them being pure diethyl ether, hexane and ethyl acetate.

2.2. Isolation procedures

2.2.1. Simultaneous distillation solvent extraction (SDE)

Simultaneous distillation extraction was carried out using the Lickens–Nickerson apparatus (in low-density solvent configuration). The extraction solvent was 10 mL of diethyl ether. The sample flask was charged with the batch in 100 mL of deionized water. The micro-simultaneous steam distillation extraction procedure was carried out for 2 h. For the condensation of steams, the condenser was cooled with a solution of glacial water-glycol (−10 °C). For the determination of the procedure yield in garlic oil the solvent was evaporated under a gentle nitrogen blowdown stream. To prevent volatile garlic oil constituents evaporation the concentration procedure was held at low temperature. The absence of solvent was determined by GC analysis. The remaining oil was weighed on an analytical scale and all the procedures results are shown in Table 1. After weighing the whole sample was re-diluted in 10 mL of the extraction solvent. The experimental procedure was repeated once with hexane and once with ethyl acetate.

2.2.2. Microwave-assisted hydrodistillation extraction (MWHD)

The microwave-assisted distillation setup was performed in a modified LG 700 Watt max power household microwave oven, using a steam distillation solvent extraction apparatus equipped with a 1L sample flask, a pressure equalizing dropping funnel, and a steam condenser. This apparatus was used instead of the classical Lickens–Nickerson one because of the vigorous foaming phenomenon during the procedure. The extraction solvent was diethyl ether. The sample flask (1 L) was charged with the batch in 100 mL of deionized water and diethyl ether (50 mL). The distillation time was 30 min for the isolation of the essential oil at maximum power. The organic extract was concentrated by a gentle nitrogen blowdown stream to 10 mL volume and anhydrous magnesium sulfate was added. After filtration through a syringe filter the determination of the yield in garlic oil was held as in the SDE procedure. The experimental procedure was repeated once with hexane and once with ethyl acetate.

2.2.3. Ultrasound-assisted extraction (USE)

Ultrasound-assisted extraction was performed in an ultrasound cleaning bath (Sonorex, Super RK 255H type, $300 \times 150 \times 150$ mm internal dimensions) by the mode of the indirect sonication, at the fixed-frequency of 35 kHz using as the working liquid. The temperature of the sonicated bath was 25 °C. The 500 mL sample flask was charged with the batch and 50 mL of diethyl ether was added. Sonication was held for 30 min. After sonication the organic layer was transferred into a separation funnel and 20 mL of a saturated solution of NaCl were added. The separated organic layer was collected. The water layer was washed with another 20 mL portion of the solvent. The whole organic extract was introduced again in the separation funnel and washed with 20 mL of a saturated solution of NaCl. The batch was

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