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Valorization of citrus by-products using Microwave Steam Distillation (MSD)

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ABSTRACT

A microwave steam distillation (MSD) of essential oils from fresh citrus by-products (orange peels) was studied. The effectiveness of this innovative method in extraction of citrus essential oils have been evaluated and compared to conventional steam distillation. MSD offers important advantages like shorter extraction time (6 min), cleaner features and provides an essential oil with better sensory properties (better reproduction of natural fresh fruit aroma of the citrus essential oil) at optimized power (500 W). Results from chemical and cytological approaches confirm the effectiveness of this new technique, that allows substantial savings in terms of time and energy.

Industrial relevance: The treatment of by-products represents a strong demand for industrial fruits processing, which produces tonnages of waste material such as peels, seeds and fibers. The disposal of these materials usually represents an industrial legal restriction problem. Moreover, the waste treatment represents significant costs and is often misjudged by companies. Transformation of waste products with high value-added allows companies to reduce the global treatment costs, sometimes even to take some profits and thus improve their competitiveness. Moreover, the recovery process of by-products is part of the current existing sustainable development and environmental protection.

1. Introduction

Citrus is the most abundant crop in the world, with about 64 million tons of orange and 13 millon tons of lemon products produced during 2004 (Laufenberg, Kunz, & Nystroem, 2003). The amount of residue obtained from citrus fruits account for 50% of the original amount of whole fruit (Chon & Chon, 1997). Produced in tones per day, citrus by-products represent a problem for management, pollution, and environmental issues, due to microbial spoilage. Drying of those products is necessary before processing. Since the cost of drying, storage, and transport engender additional economical limitations to by-products processing instead of its integration in animal feed or fertilizer. Thus new aspects concerning the use of these by-products for further exploitation on the production of food additives or supplements with high nutritional value and economically attractive have gained increasingly interest.

Citrus juice by-products are mainly constituted by peels (albedo and flavedo), seeds, and fruit pulp remaining after juice extraction (Braddock, 1995). Citrus peel of fruit processing which provides a great potential for further commercial use. During the process of juice extraction oil sacs break and release volatile oils which are in pockets localized in the external part of the mesocarpe of fruit (flavedo). These oils are used in food and pharmaceutical industries, but can also provide flavoring ingredients to drinks, ice creams and other food products. In addition, substantial quantity of these oils is also used in the preparation of toilet soaps, perfumes, cosmetics and other home care products (Raeissi, Diaz, Espinosa, Peters, & Brignole, 2008). Virot, Tomao, Ginies, Visinoni, and Chemat (2008) reported that the *d*-limonene, major component of the oil extracted from citrus peels, could be used as green solvent instead of hazardous petroleum solvents for fats and oils determination. d-limonene is considered as a very versatile chemical which can be used in a wide variety of applications. It is safer and more effective than typical cleaning solutions.

It is also well known that terpenes which constitute the major part of citrus oils have a strong antifungal activity (Caccioni, Guizzardi, Biondi, Renda, & Ruberto, 1998). Few studies have been conducted on the extraction of citrus essential oils in general and oil from orange peel in particular (Berna, Tárrega Blasco, & Subirats, 2000; Bousbsia, Vian, Ferhat, Meklati, & Chemat, 2008; Ferhat, Meklati, Smadja, & Chemat, 2006; Raeissi et al., 2008), although studies on the deterpenation or separation of the different hydrocarbons composing citrus oils are more

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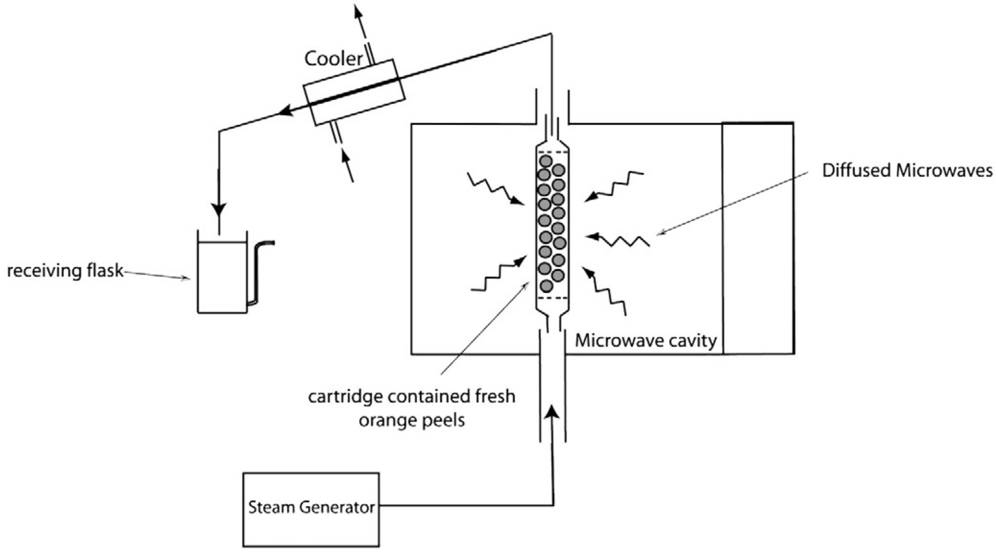

Fig. 1. Microwave Steam Distillation (MSD) apparatus.

abundant (Arce, Pobudkowska, Rordíguez, & Soto, 2007; Danielski et al., 2008; Diaz, Espinosa, & Brignole, 2005; Dugo et al., 1995; Raeissi & Peters, 2005).

The traditional method to extract essential oils from the citrus peels is by cold pressing. The oil is present in oil sacs or oil glands located at different depths in the peel and the cuticles of the fruit. Peel and cuticle oils are removed mechanically by cold pressing and since this procedure yields a watery emulsion, this emulsion is then centrifuged to recover the essential oil, that is relatively low (0.05%) (Ferhat, Meklati, & Chemat, 2007). Distillation is also used in some countries as an economical way to recover the oils, with a better yield of 0.21% vs 0.05% for cold pressing (Ferhat et al., 2007). During distillation, the Citrus peels exposed to boiling water or steam release their essential oils through evaporation. As steam and essential oil vapors are condensed, both are collected and separated in a vessel traditionally called "Florentine flask" (Guenther, 1974).

The development of new extraction techniques for chemical, food and pharmaceutical industries has lately received a lot of attention due to the increasing energy prices and the drive to reduce CO_2 emissions. These shortcomings have led to the use of new techniques in the extraction of natural substances which typically use less solvent and energy such as supercritical fluid extraction, ultrasound extraction, the controlled pressure drop process, and sub-critical water extraction. Researchers in many universities are working on novel techniques that could lead to more compact, safer, more efficient, energy saving, and sustainable extraction processes (Ganzler, Salgo, & Valko, 1986; Lucchesi, Chemat, & Smadja, 2004; Luque de Castro, Jiménez-Carmona, & Fernández-Pérez, 1999; Reverchon, 1997; Rezzoug, Maache-Rezzoug, Mazoyer, Jeannin, & Allaf, 2000; Vinatoru, 2001).

In this study, microwave steam distillation (MSD) apparatus has been used for extraction of essential oil from orange peels. Comparisons have been made to conventional steam distillation (SD) in terms of extraction time, yield and composition of essential oil. To highlight the physical action of both extraction systems, scanning electron micrographs supplemented the chemical analysis studies.

2. Materials and methods

2.1. Plant material

In this study, about 100 kg of orange (*Citrus sinensis* L. Osbeck from Valencia late cultivar) peel were collected locally after juice extraction (which separates the external part of the orange (peel), giving an orange peel yield of approximately 20% (w/w) of the whole fruit). The initial moisture content of orange peel was 75%. Fresh plant material was employed in all extractions.

2.2. MSD apparatus and procedure

MSD has been performed in a Milestone "Dry Dist" batch reactor (Bergamo, Italy). It is a multimode microwave reactor operating at 2.45 GHz with a maximum delivered power of 1000 W variable in 10 W increments. During experiments time, temperature and power were recorded and controlled.

An electrical steam generator and a condenser placed outside the microwave oven are connected to a cartridge containing citrus by-products via Pyrex connecting tubes. The condenser is connected to a receiving Florentine flask which is preferably a separating funnel to enable the continuously collection of condensate essential oil and water. This system presents the advantage that the cartridge containing plant materials can be easily and quickly replaced and cleaned after each cycle of extraction (Fig. 1).

The cartridge containing 100 g of citrus peels is subjected to microwave heating and vapor which cross the plant material. Microwaves distend the plant cells and lead the rupture of the glands and cell receptacles. The steam passes through the sample, evaporating and carrying the essential oil directed towards the condenser and the Florentine flask. The extraction was continued until no more essential oil was obtained. The essential oil is collected, dried with anhydrous sodium sulfate and stored at 4 °C until used. Extractions were performed at least three times, and the mean values were reported.

Table 1
Results of preliminary study.

Q ($g \cdot min^{-1}$)	Yield (%)	P (W)	Yield (%)
2	5.40 ± 0.07	200	5.40 ± 0.07
4	5.20 ± 0.02	300	5.42 ± 0.07
6	5.40 ± 0.07	400	5.44 ± 0.07
8	5.41 ± 0.07	500	5.45 ± 0.07
10	5.41 ± 0.07	700	5.45 ± 0.07
14	5.46 ± 0.07		
18	5.46 ± 0.07		