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COMPARATIVE STUDY OF TWO EXTRACTION METHODS OF ESSENTIAL OILS FROM ORANGE PEEL: MICROWAVE-ASSISTED HYDRODISTILLATION AND CONVENTIONAL HYDRODISTILLATION

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Abstract

Description of the subject. There is a growing body of literature that recognizes the importance of the extraction technology of bioactive compounds in development of the sector of agro-food industries.

Objective. The aim of this study is to extract essential oil from orange peel (*Citrus sinensis* L.), a byproduct, by two methods that are Conventional hydrodistillation and microwave-assisted hydrodistillation.

Methods. Conventional hydrodistillation (CHD) and microwave-assisted hydrodistillation (MAHD) methods were used to extract essential oil from Algerian sweet orange peel and their results were compared.

Results. MAHD was more efficient by reducing the time required for the extraction from 110 min, in CHD method, to 30 min. There was no significant difference in physicochemical characteristics and chemical composition between the essential oils obtained by the 2 methods.

Conclusion. Returning to the question posed at the beginning of this study, it is now possible to state that We can say that MAHD method was undoubtedly the more effectiveness method.

Keywords: *Citrus sinensis*; Conventional hydrodistillation; Microwave-assisted hydrodistillation; Essential oil

ÉTUDE COMPARATIVE DE DEUX METHODES D'EXTRACTION DES HUILES ESSENTIELLES DE L'ÉCORCE D'ORANGE : HYDRODISTILLATION ASSISTÉE PAR LES MICRO-ONDES ET HYDRODISTILLATION CONVENTIONNELLE

Résumé

Description du sujet. Il existe beaucoup de travaux qui affirment que la maîtrise et l'optimisation de l'extraction des molécules bioactives est très importante pour le développement du secteur des industries agro-alimentaires.

Objectifs. Le but de ce travail est d'extraire les huiles essentielles à partir du zeste d'orange (*Citrus sinensis* L.), un sous-produit industriel, par deux techniques à savoir l'hydrodistillation classique (HDC) et l'hydrodistillation assistée par micro-onde (HDMO).

Méthodes. Les huiles essentielles ont été extraites via deux méthodes d'extraction (HDC et HDMO) et les résultats ont été comparés.

Résultats. L'hydrodistillation assistée par microondes est beaucoup plus préformante puisqu'elle a permis de réduire le temps nécessaire pour l'extraction de 110 min, dans le cas de l'HDC, à seulement 30 min. Les caractéristiques physicochimiques et chimiques des huiles essentielles ont été similaires.

Conclusion. L'hydrodistillation assistée par microondes est incontestablement une méthode plus performante que l'hydrodistllation conventionnelle et peut constituer une potentielle technologie verte.

Mots clés: *Citrus sinensis*; Hydrodistillation conventionnelle; Hydrodistillation assistée par microondes; Huiles essentielles.

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INTRODUCTION

Orange peel is generated with large quantities as an agro-indusial byproduct which should be valorized [1]. This byproduct can even cause pollution problems for the environment due to high associated chemical and biological oxygen demand, because of its amount of essential oil, pectin and sugar if it has not been processed [2, 3]. The incorporation of those compounds in the formulation of some food products could offers potential ways to their valorization. One of the most important compounds that are contained in orange peel is essential oil. Indeed, the essential oil of orange peel is actually used in many products such as soft drinks, ice creams, perfumes, etc. [4]. This increasing interest is due to the different properties of essential oils in particular anticancer, antiviral, antibacterial, insecticidal and antioxidant properties [5, 6, 7].

However, essential oils are obtained with a very low yields making it fragile substances, rare, but still valuable. Thus the different extractions methods of essential oils must in first, consider these characteristics and secondly, provide quantitative performance. The extraction technology is a key for sustainable development of the sector of agro-food industries. Indeed, essential oil can be isolated using several methods that differ from one another by the time required for extraction, performance, energy consumption, etc. [8, 9]. The conventional hydrodistillation remains the most common method used for the extraction of essential oils. However, this extraction method has several disadvantages such as loss of the ability of some compounds of the extract due to thermal degradation, a long extraction time and the considerable consumption of energy [10, 11, 12, 13]. Nowadays, the use of microwaves for the extraction of essential oils has become a source of interest to many researchers [8, 14, 15, 16, 17, 18].

According to Özek et *al.* [19], the advantages of this extraction technology are many, namely: the heating efficiency, the fast heat transfer and the reduced size of equipment.

Usually emerging technologies have a high degree of uncertainty and complexity, and these make their industrialization difficult. Industries have to decide which emerging technology is more suitable to be used as a substitution of traditional methods. Comparing novel technologies which can have the same application and new advantages will facilitate industrial decision [20]. However, there has been little discussion about how the combination of hydro-distillation and microwave heating affects extraction. In this paper, we present a comparative study of the ability of 02 methods (conventional hydrodistillation and microwave-assisted hydrodistillation) to extract essential oils from sweet orange peel *Citrus sinensis* L. in order to find the most advantageous in term of extraction kinetics, essential oil quality and quantity.

MATERIEL AND METHODES

1. Plant material

Sweet orange fruits (*Citrus sinensis* L.) were purchased from a wholesale market in Bejaia, Algeria, in March. Oranges were washed with tap water. The orange peel was grated (within 3 days) into small pieces in order to increase the contact surface between the secreting cells and the extraction solvent (distilled water) to rise the extraction efficiency.

2. MAHD apparatus and procedure

100 g of grated peel was introduced in 1 L flask (Pyrex) containing distilled water (600 mL). The flask was placed at atmospheric pressure in a domestic microwave oven (Samsung, Malaysia, 2.45 GHz, 900 W) which was modified for the extraction. The microwave oven was operated at 700 W power level for a period of 33 min. The flask was setup within the microwave oven cavity and a condenser was used on the top to collect the extracted essential oils (Fig. 1).

3. CHD apparatus and procedure

The pilot plan hydrodistillation equipment used in the present study was the same with the MAHD, but we replaced the microwave by a heating mantle (Nahita, model 665). 100 g of grated peel was added in 1 L

flask (Pyrex), dissolved in 600 mL distilled water. Hydrodistillation was carried out for 120 min and the condenser was cooled in water.
The condensed liquid was placed in a separating funnel where the oil is separated from the water by density difference. After decantation, the essential oils extracted by these two methods were dried with anhydrous sodium sulfate (Na_2SO_4) and after filtration, stored in amber vials at 4 °C until tested and analyzed.

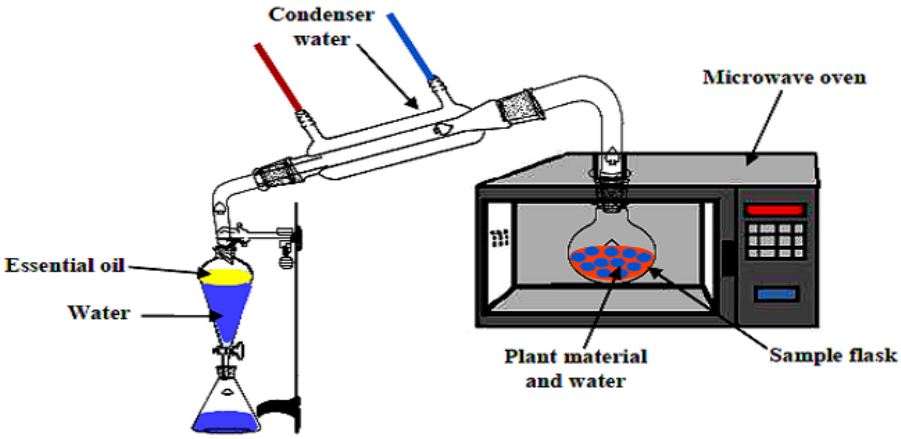

Figure 1 : Schematic representation of MAHD apparuts.

4. Calculation of the extraction yield and study of the kinetics of extraction

The yields of *Citrus sinensis* L. essential oil, was expressed as the weight of extract relative to the weight of the starting plant material. The yield is expressed in percentage (%) and calculated using eq. (1):

$$Y = (WE / WP) \times 100 \quad (1)$$

Y: Yield of essential oil (%); WE: Weight of the essential oil in (g); WP: Weight of plant in (g).

The kinetics was explored by sampling at regular intervals of time on the aqueous and organic phases. In order to monitor the kinetics of the extraction, we recovered samples of the essential oil at a time intervals of 10 min for CHD method and 3 min for MAHD method.

5. Analytical study of essential oils

5.1. Organoleptic characteristics

The organoleptic characteristics (appearance, color and odor) of the essence of orange peel were noted.

5.2. Physical constants

The methods used to determine the refractive index and specific gravity of the essential oil were measured according to the method suggested by AFNOR [21] and were carried out at 20 °C.

5.3. Gas chromatography–mass spectrometry (GC–MS)

The essential oil was analyzed by gas chromatography coupled to mass spectrometry GC-MS using a GC-MS Thermofisher Trace GC Ultra DSQII with autosampler Triplus AS equipped with a capillary column TR-5MS (30 m × 0.25 mm, film thickness 0.25 µm). Helium was the carrier gas at a flow rate of 1 ml / min. The temperature of the injector and the MS transfer line was set at 200°C and 270°C, respectively; Injection Split 1: 100; injection volume 1 µl. The oven temperature programme was 60°C for 1 min increased at 3°C/min and held at 240°C for 1 min. For MS, the source temperature was 200°C. The ionization was electronic impact at 70 eV. Electron ionization mass spectra were acquired over the mass range 35–450 µm. The identification of the components of the essential oil was based on the comparison of peaks and their Kovats retention indices with those of a database available from the US National Institute of Standard Technology (NIST) library [22].

6. Statistical Analysis

All tests were done in triplicate except GC-MS. Student test for comparisons were used to determent significant differences (α = 0.05) between results except Chemical composition, using the software STATISTICA (Version 7.1., Statsoft Inc, France, 2005).

RESULTS AND DISCUSSION

1. Comparison of extraction kinetics of and extraction yield

As shown in Fig. 2, essential oil yield (w/w) extracted from Algerian sweet orange peel *(Citrus sinensis* L.) by the two extraction methods was 2.24±0.06 and 2.22±0.09% for the CHD and MAHD, respectively.

From the graphs above we can see that there was no significant difference between the yields of the two extraction methods (p>0.05). Our results of yield were higher than those obtained by Romdhane & Chemat [9], this can be explained in the case of these authors by the loss which may be observed at the time of extraction of juice and separation of peel.

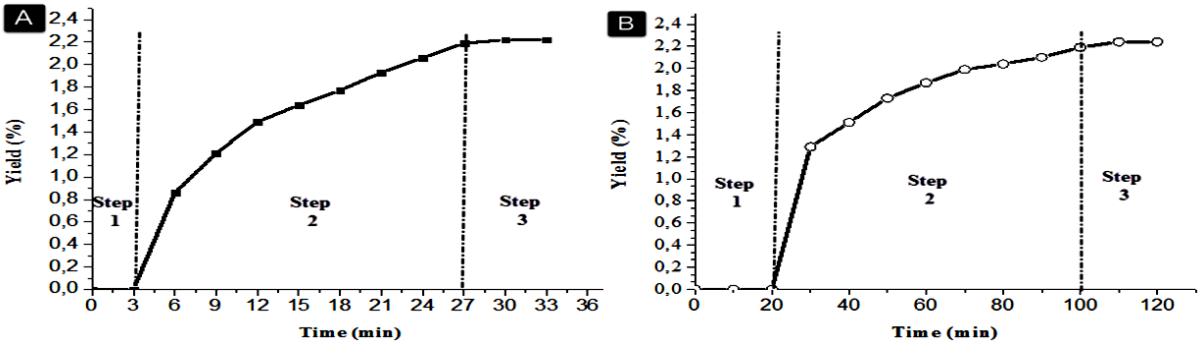

Figure 2. Yield profiles as a function of time for the MAHD (A) and CHD (B) isolations of the essential oil from orange peel.

The kinetic of CHD (Fig. 2B) was divided into three steps: In the first, there was a waiting phase corresponding to the heating phase of the matrix to the boiling temperature; The second corresponded to a fast rise of the amount of essential oil recovered (20-100 min); Finally, the last step corresponds to a horizontal line which marks the end of the extraction process (no more essential oil was obtained). This result showed that it would be economically profitable to fix the time of the CHD method to 90 min because beyond it there will be absolutely a waste of energy without getting too much of the essential oil. The kinetic of the extraction by the MAHD method (Fig. 2A) showed the same evolution as the kinetic of the CHD method where the three phases were observed. However, the duration of the heating phase of the matrix is very short, and the boiling point (100°C at atmospheric pressure) was reached in 3 min, the heat is generated by ionic conduction and/or dipole rotation. In other words, MAHD gathers the first droplet of essential oil after about 03 min while CHD does this only after about 20 min. The MAHD was able to extract in 20 min the same amount of essential oil as the CHD method in 60 min; This is probably due to a more efficient heat flow in the biological matrix when heated by microwaves [14], and it could be explained by the fact that in the case of CHD, mass transfer occurred from the inside to the outside whilst heat transfer occurred from the outside to the inside. However, in the MAHD the two transfer phenomenons are in the same direction from the inside to the outside of the peel [9]. According to Lucchesi et *al*. [23] this method of heating is instantaneous inside the volume and not on the surface of the matrix. Thermal conduction phenomena and convection play only a secondary role of tempering. These observations were also in accordance with the observations of Li et *al*. [15]. The fast increasing in the yield of essential oil during the second phase was explained by Damjanović et *al.* [10]; Özek et *al*. [19], by the volatilization of the essential oils that were on the surface of the plant.

Likewise, the internal heating of the *in situ* water within the plant material distends the plant cells [24]. The advantage of the MAHD extraction method was the short time need for the extraction (extraction of about 50 % of essentials oils in only 10 min). It is obvious that the reduction of the time necessary for the extraction (heating for only 30 min) will reduce the total cost energetic of essential oil isolation, which will decrease the environmental invoice in term of the carbon dioxide emission. So, the reduced cost of extraction is incontestably advantageous for the MAHD method in term of energy consumption. For example, if we take the minute 21, we can clearly deduce the effectiveness of MAHD method, which could give a yield of 1.9% at this moment (about 85% of the final yield). In the mine-time, the CHD method just starts to give the first essential oil droplet.

2. Analytical study of essential oils

2.1. Organoleptic characteristics

Essential oils obtained with the two extraction methods, was very clear with a strong and pleasant citrus odor. It had a light yellow color. Organoleptic parameters of our essential oil were consistent with those listed in the AFNOR standards cited by Garnero [25].

2.2. Physical constants

The physical properties of essential oils extracted by both methods and the AFNOR standard reference values were shown in Table 1. It is apparent from this table that the two extraction methods produced essential oils with similar ($P > 0.05$) refractive indexes and densities. Results show clearly that the physical constants tested for the extracted essential oil were in accordance with the AFNOR standards [21].

Table 1: Physical constants of orange peel essential oils obtained by MAHD and CHD.

Physical constants at 20°C	CHD	MAHD	AFNOR standards [21]
Specific gravity	0.794±0.004[a]	0.789±0.004[a]	≤ 0.844
Refractive index	1.473±0.002[a]	1.472±0.001[a]	1.470-1.480

Values are Mean ± SD. Values followed by different letters in the same line differ significantly ($P < 0.05$).

So, considering the physical properties of essential oil extracted by the MAHD, which is a new extraction method, it does not affect the quality of the essential oil extracted from sweet orange peel; these results were in accordance with the observations noted by Bousbia et *al*. [8]; Golmakani and Rezaei [14] during the extraction of essential oils from *Rosmarinus officinalis* leaves and *Thymus vulgaris* L., respectively.

2.3. Chemical composition of essential oils

The identified compounds with the area percentage of each peak were reported in Table 2 following their elution order. There were no considerable differences among the chemical profiles generated by the traditional and the alternative techniques. The GC-MS profile of the essential oil, obtained by CHD and MAHD from the Algerian sweet orange peel, showed the presence of 23 identified volatile secondary metabolites in both essential oils, representing 99.96% and 99.91% of the total chemical composition of the essential oil extracted by MAHD and CHD, respectively.

CHD oil contained higher percentages of β-Phellandrene (0.50%), β-Myrcene (3.36%) and Eremophilene (1.53%) than MAHD oil, which contained 0.06, 1.42 and 0.5 %, respectively. In contrast, the essential oil obtained by MAHD method contained a higher percentage of β-Citral, α-Citral and limonene.
Biochemically, the essential oil of *Citrus sinensis* L. consisted mainly of monoterpene hydrocarbons compounds with limonene (compound No.04) as undoubtedly the most abundant component in both essential oils (85.77% for the essential oil of the CHD method and 88.50% for the essential oil of the MAHD method) and followed by minor compound like β-Myrcene (which is the second important compound) and β-Linalool (an oxygenated monoterpene). The GC-MS composition obtained were in accordance with the study of Romdhane & Chemat [9] who reported the same observation, noting that limonene is the major component of the essential oil of *Citrus sinensis* L. peel. This was also observed by Lin et *al*. [26]; Singh et *al*. [27] ; Velazquez-Nuñez et *al*. [28].

What is comes out from this study is that *Citrus sinensis* peel essential oil present a high monoterpene content (about 90%). From the aromatic profiles of the essential oils, MAHD enhanced the extraction performance without any major modification in the volatile secondary metabolite compounds of *Citrus sinensis* L. peel.

Romdhane & Chemat [9] reported that the microwaves energy induced only a thermal effect during the extraction process. Therefore, the extraction of essential oils by microwave-assisted hydrodistillation was an efficient alternative method for extracting essential oils without altering their chemical compositions.

Table 2: Chemical compositions of orange peel essential oils obtained by MAHD and CHD

No.	RT[a] (min)	Compounds	CHD (%)	MAHD (%)
		Mononterpenes	90.55	90.52
01	5.65	Δ3-Carene	0.14	0.01
02	6.48	β-Phellandrene	0.50	0.06
03	7.39	β-Myrcene	3.36	1.42
04	8.95	Limonene	85.77	88.50
05	9.89	α-Terpinene	0.06	0.07
06	10.91	Terpinolene	0.13	0.16
		Oxygenated monoterpenes	5.42	5.93
07	7.99	Geranial	0.59	0.30
08	11.59	β-Linalool	2.99	2.79
09	11.86	Nonaldehyde	0.18	0.13
10	13.04	Cis-limonene oxyde	0.05	0.03
11	13.21	(E) limonene oxyde	0.12	0.15
12	13.83	β-Citronellal	0.28	0.46
13	15.12	Terpin-4-ol	0.24	0.09
14	15.83	α-Terpineol	0.51	0.54
15	16.67	β-Citral	0.41	0.70
16	19.00	α-Citral	0.64	1.04
		Sesquiterpenes	2.30	1.23
17	23.16	α-Cubebene	0.23	0.22
18	26.38	β-Farnesene	0.14	0.16
19	28.10	Eremophilene	1.53	0.50
20	28.53	α-Farnesene	0.10	0.07
21	29.12	α-Cadinene	0.30	0.28
		Oxygenated sesquiterpenes	0.16	0.25
22	36.06	β-Sinensal	0.16	0.25
		Other oxygenated compounds	1.53	1.98
23	16.18	Decanal	1.53	1.98
		Extraction time (min)	30	110
		Yield (%)	2.24±0.06	2.22±0.09

CONCLUSION

The use of microwave-assisted hydrodistillation method for the extraction of essential oils from sweet orange peel had more advantages than the conventional hydrodistillation method. The CHD method give a same yield in essential oil as MAHD method, but it taken much time and consuming a lot of energy which were increasing the costs of the extraction. The MAHD method was more performant than the CHD method by dividing the extraction time by three (3 times lower than in the CHD) and at the end we the same yield with reduction in the energy consumption.

The results of this study shown that the MAHD extraction method, of the essential oils, was a method of choice especially for its speed, ease to use, lower energy consumption and the essential oil obtained by this method had the same quality and the same chemical composition us the essential oil obtained by the CHD extraction method.

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