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Extraction of Essential Oils from Citrus By-Products Using Microwave Steam Distillation

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Abstract

The main objectives of this research is to extract essential oil from: orange *(citrus sinensis*), lemon*(citrus limon*) and mandarin*(citrus reticulata*) peels by two methods: steam distillation (SD) and microwave assisted steam distillation (MASD), study the effect of extraction conditions (weight of the sample, extraction time, and microwave power, citrus peel type) on oil yield and compare the results of the two methods, the resulting essential oil was analyzed by Gas Chromatography (GC).

Essential oils are highly concentrated substances used for their flavor and therapeutic or odoriferous properties, in a wide selection of products such as foods, medicines and cosmetics. Extraction of essential oil is one of the most time and effort consuming process. Microwave-assisted extraction is a green technique for the extraction of natural products. (MASD) was better than (SD) in terms of rapidity, energy saving and yield. (MASD) gave higher yield than (SD) with shorter extraction time, yield of orange oil extracted by (MASD) was (1.150%) in (35min.) compared to (1.095%) in (45min.) by (SD) process, same results obtained for lemon and mandarin.

The optimal microwave power was (135W) gave oil yield: (1.150%, 1.115%, 0.940%) for orange, lemon and mandarin respectively, (MASD) increased extraction temperature in short time and to a higher level compared to (SD). The optimal weight was (398.56gm) gave yield in (SD): (1.095%) and MASD (1.091%) for orange oil, same results obtained for lemon and mandarin. The best citrus peel type which gave the highest yield was orange followed by lemon then mandarin in both processes.
Limonene is the most abundant component in citrus essential oil, (GC) analysis showed that (SD) was more convenient to give high amount of limonene because of the graduate temperature rise, while in microwave extraction exposure to low microwave

Key Words: Essential oil, extraction, steam distillation, microwave assisted steam distillation, orange, lemon, mandarin, citrus peels, yield

Introduction

Essential oils are volatile natural, complex mixtures comprising many unique compounds [1]. Essential oils extracted from several plant organs including flowers, leaves, twigs, seeds, fruits, roots, wood or bark are valuable natural products [2]. Nowadays, people worldwide are looking towards natural base products since there are no side effects when taken accordingly. Furthermore, there is also an interest in the production of functional, high value, natural products without chemical modification and residues of solvents or additives [3] , for these reasons essential oils are used as raw materials for many products including food flavorings, additives, flavoring agents used in the cigarette industry and in the compounding of cosmetics and perfumes [2].They are used also in air fresheners and deodorizers as well as in all branches of medicine such as: pharmacy, balneology, massage and homeopathy [3].

Citrus is the largest fruit crop in the world (100 million cubic tons per year) and the orange account for 60% [4]. The remaining orange peel account for approximately 45% of the total bulk [5], represent a problem for management, pollution, and environmental issues, due to microbial spoilage. 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 [6].

The traditional method to extract essential oils from the citrus peels is by cold pressing. 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 [7].

Nowadays, many novel techniques for the extraction of essential oils that could lead to more compact, safer, more efficient, energy saving, and sustainable extraction processes, including ultrasound-assisted extraction (UAE), microwave-assisted extraction (MAE), supercritical fluid extraction (SFE) and accelerated solvent extraction (ASE) have become relatively mature and widely accepted by industries [8].

Microwave is intrinsically one type of electromagnetic waves with frequency ranging from $300MH_Z$ to $300GH_Z$, which can penetrate biomaterials and interact with polar molecules such as water in the biomaterials to create heat [9]. Due to it's high selectivity, microwave heating is capable of expanding and rupturing of cell walls followed by the liberation of chemicals into surrounding solvent in short time [10].

Sahraoui et al. [11] extracted essential oil from citrus by-products (orange peels) with a new process, microwave steam distillation (MSD). Compared to the conventional steam distillation (SD), the results obtained confirm the effectiveness of this new technique, which allows substantial savings in term of extraction time and energy. MSD highly accelerated the extraction process, without causing changes in the volatile oil composition. MSD offered important advantages like shorter extraction time, with MSD 6 min provides yields comparable to those obtained after 2 h by SD, which is the reference method in essential oil isolation. The values of yield obtained are respectively 5.43 ± 0.03% for MSD and 5.45 ± 0.04% for SD. MSD offered cleaner features and provided an essential oil with better sensory properties (better reproduction of natural fresh fruit aroma of the citrus essential oil) at optimized power (500 W).

Cassel et al. [12] used rectification extraction (RE) to isolate essential oils from Chinese herbs, RE and steam distillation (SD) techniques were applied to isolate essential oils from three typical Chinese herbs *Bupleurum*, *Pogostemon cablin (Blanco) Benth* (PCB), *Pericarpium Citri Reticulatae* (PCR). The experiment shows that SD technique cannot get volatile oils and only aromatic water from *Bupleurum* but RE can prepare both. RE technique increases 10% oil yield of PCB and 20% of PCR compared with SD, respectively. Therefore, RE would be more effective and suitable for enriching volatile oils from plants or herbs in which volatile oils are water-soluble and with low-content. Therefore, RE can significantly shorten the operation time of extracting volatile constituents of herbs such as Chuanxiong and *Bupleurum*, and reduce energy consumption relatively during isolating process. The common components in the oils prepared by SD and RE, occupy more than 91% of the volatile oil samples. Compared with SD, RE technology not only increases volatile oil yield, but also barely changes the constituents and compositions of volatile oils.

Farhat et al. [13] Microwave steam diffusion (MSD) was developed as a cleaner and new process design and operation for isolation of essentials oils from dry lavender flowers and was compared to conventional steam diffusion (SD). The essential oils extracted by MSD for 3 min were quantitatively (yield) and qualitatively (aromatic profile) similar to those obtained by conventional steam diffusion for 20 min. In addition, an optimal operating steam flow rate of 25 g min^{-1} and microwave power 200 W were found to ensure complete extraction yield with reduced extraction time. MSD was better than SD in terms of energy saving, cleanliness and reduced waste water.

Lucchesi etal. [14] used two different extraction methods for a comparative study of Algerian Myrtle leaf essential oils: solvent-free-microwave-extraction (SFME) and conventional hydrodistillation (HD). Essential oils analyzed by GC and GC-MS presented 51 components constituting 97.71 and 97.39% of the total oils, respectively. Solvent-Free-Microwave-Extract Essential oils SFME-EO were richer in oxygenated compounds. Several advantages with SFME were observed: faster kinetics and higher efficiency with similar yields: 0.32% dry basis, in 30 min as against 180 min for HD.

Ginies et al. [15] used 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)) to extract essential oil from lavandin flowers and their results were compared. The method which gave the best results was the microwave hydrodiffusion and gravity (MHG), it gave the maximum yield (5.4%) in only 15 min (120 min for SD) and consumed 1.3 kWh (against 8.06 kWh for SD). The essential oil obtained was of excellent quality (low degradation) and natural odor (similar smell to the original lavender).

Vieira et al. [16] studied Supercritical carbon dioxide extraction of essential oil from *Thymus vulgaris* leaves in the Florys S.p.A. laboratory at 40 °C and 20 MPa for three different particle sizes and three different flow rates. A mathematical model, proposed in the literature, based on the local adsorption equilibrium of essential oil on lipid in leaves was used to calculate internal and external mass transfer resistances. The adsorption equilibrium constant was fitted to these experimental data, and preliminary results suggested that the extraction process is controlled by mass transfer resistance due to intraparticle diffusion.

In this study, microwave steam distillation (MSD) apparatus has been used for extraction of essential oil from three types of fresh citrus peels: orange (*citrus sinensis*), lemon(*citrus limon*), and mandarin (*citrus reticulate*). Comparisons have been made to conventional steam distillation (SD) in terms of extraction time, yield, weight of the sample, and composition of essential oil. The essential oils were analyzed by gas chromatography to determine the concentration of limonene in each produced sample for qualitative study.

Experimental Work

Collection of Plant Material

Citrus fresh fruits: orange *(citrus sinensis)*, lemon *(citrus limon)*, mandarin *(citrus reticulata)* brought from local markets. Each type of citrus fruit was washed and peeled separately. The fresh peels were grated using an electrical grater that results small particles of the peels. They were divided into three groups having three different weights: (398.56gm, 281.8gm, and 116.76gm)

Experimental Procedure

1- Extraction of Essential oil by microwave assisted steam distillation (MASD)

In Microwave Assisted steam distillation (MASD) unit an Pyrex flask which contained boiled distilled water generating steam and a condenser placed outside the microwave oven are connected to a cartridge containing grated citrus peels via Pyrex connecting tubes. The condenser is connected to a receiving 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 as shown in Fig.1.

The cartridge containing the required weight for each run (398.56gm for full flask, 281.8gm for half full, and 116.76gm the quarter of flask) of citrus peels is subjected to microwave heating and vapor which cross the plant material. The probe of the sensor was placed on the extraction vessel in order to record temperature, as the steam rises up to the grated peels charged with the essential oil on it's way to the condenser with the first drop of condensate flows in the separation funnel the microwave irradiation power is switched on.

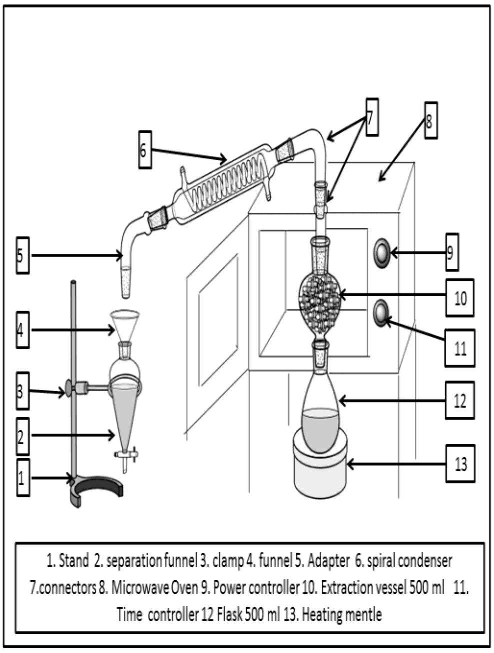

Fig.1, Schematic Diagram of the Experimental Extraction Unit of Microwave Assisted Steam Distillation

Microwave energy can interact selectively with the free water molecules contained in plant cells and causes localized heating, the final effect is a sudden non-uniform rise in temperature and dramatic expansion in volume with in plant cells. The cell walls cannot stand with such a high internal pressure leading to cell wall distraction and allowing the substance inside of plant cells to flow freely toward surrounding steam the resulting distillate is collected in a separation funnel. The extraction was continued until no more essential oil was obtained.

2- Extraction of essential oil by steam distillation (SD)

For a rigorous comparison, the same glassware and same operating conditions have been used for conventional steam distillation. The vapor produced by the boiled distilled water crosses the plant, charged with essential oil and then passes through the condenser to a receiving flask [17].

3- Separation of Essential oil from Water

In separation funnel the essential oil will float on top of the hydrosol (distilled water component) also called floral water and may be separated off. Due to immiscibility of the oil and water at room temperature for essential oil is lighter than water, citrus essential oil has a density in the limits of $0.8085 gm/cm^3$ while density of water is $1 gm/cm^3$.

Water layer was carefully run out from the bottom of the funnel by opening the tap leaving the oil, which was dried over anhydrous sodium sulphate and kept in a dark glass vial at temperature of 4 °C for further analysis.

The yield percent was calculated by using the following relationship:

$$\text{Yield}=\frac{\text{amount of citrus oil extracted}}{\text{total amount of grated citrus peels}}\ 100\%$$

Parameters studied for both extraction methods (MASD and SD)for three types of citrus peels; orange *(citrus sinensis)*, lemon *(citrus limon)*, mandarin *(citrus reticulata)*:

- Extraction time
- Extraction yield
- Microwave irradiation power:(135W, 265W, 445W)
- Weight of grated peels(398.56gm, 281.8gm, and 116.76gm)

Result and Discussion

1- Effect of Extraction Time

Essential oil was extracted from two types of citrus peels orange *(citrus sinensis)*, and mandarin *(citrus reticulata)* each having three weights : (398.56 gm, 281.8 gm, 116.76 gm), at (SD) process and (MASD) process exposing to (265W). Extraction time was different for these three weights and extraction yield was studied until equilibrium was reached.

Fig. (2) & (3) show oil yield in (SD) process: the weights(398.56gm, 281.8gm) had longer time than (116.76gm); at these two weights the yield of orange oil was: (1.095%, 0.860%) with extraction times: (45min., 50min.) respectively, while the weight(116.76gm) gave yield for orange oil: (0.969%) with extraction time(35min). Mandarin had longer extraction time than orange with lower yield since mandarin peel is so tender not much oil sacks or glands contained in peels, oil yield for weight(398.56gm) after (75min.) extraction time was (0.707%), for weight(281.8gm) after (55min.) extraction time the yield was (0.702%) and for weight(116.76gm) after(50min.) extraction time the yield was(0.848%).

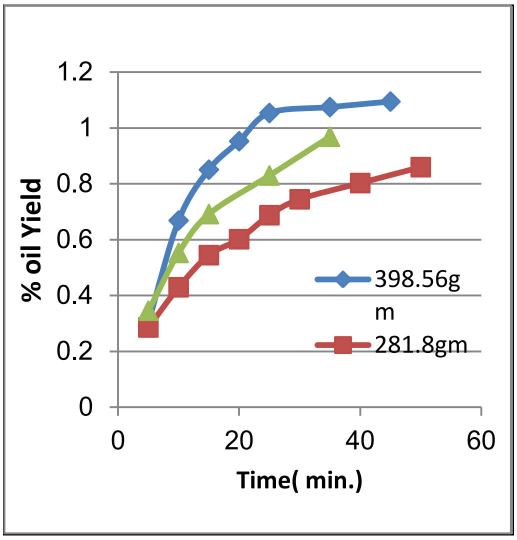

Fig. 2, Effect of Extraction Time on Yield of Orange Oil Extracted By (SD) for the Three Weights

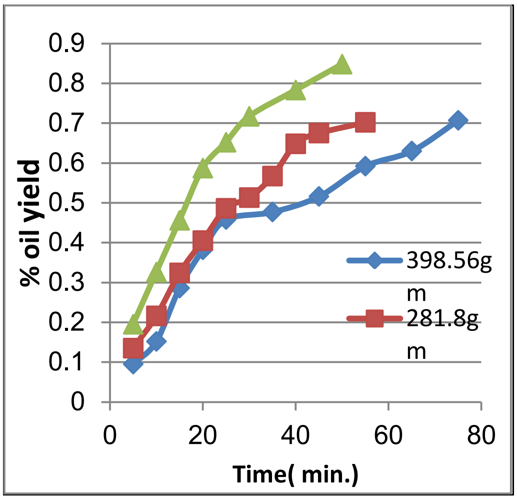

Fig. 3, Effect of Extraction Time on Yield of Mandarin Oil Extracted By (SD) for the Three Weights

Fig. (4) & (5) show oil yield in (MASD) process: the weights(398.56gm, 281.8gm) both had extraction time (35min.), for orange the yield for these two weights was: (1.058%, 0.588%), while for (116.76gm) after (20min.) extraction time the yield was: (0.516%). For mandarin having weight (398.56gm) after (35min.) extraction time the yield was: (0.846%), for weight (281.8gm) after (30min.) extraction time the yield was: (0.558%), and for weight (116.76gm) after (15min.) extraction time the yield was: (0.385%).

By analyzing data in figures above the essential oil extracted by (SD) process had longer time and higher yield than oil extracted by (MASD) process after exposing to microwave power (265W) which delivered heat higher than (SD) process and that affected oil yield, since citrus essential oil is highly sensitive to heat applied by microwave irradiation which is higher than heat applied by (SD) [11].

It can be notes from these figures that extraction of oil increased with time for the two methods: (SD), (MASD) but, the yield of oil extracted by (MASD) reached equilibrium in shorter time than that of (SD) with differences in yields. The ability of (MASD) method to accelerate extraction of essential oil is by rapid increase in temperature causing a dramatic expansion in volume within plant cells, the cell walls cannot with stand such high internal pressure resulting with rupture of the cells and glands of plant material more rapidly than conventional (SD) [10], these results are in agreement with results obtained by Sahraoui et al. (2011) [11].

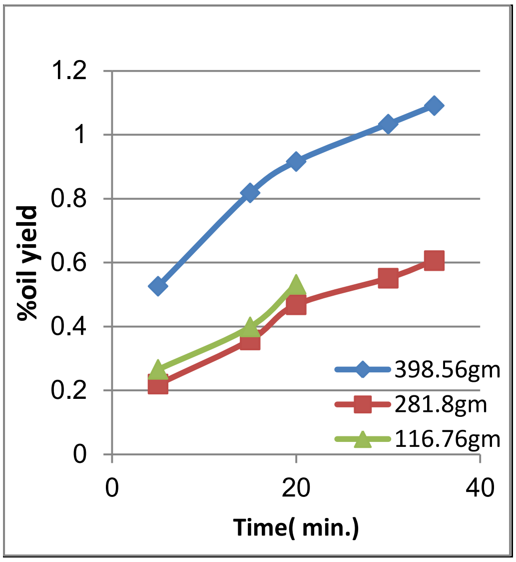

Fig. 4, Effect of Extraction Time on Yield of Orange Oil Extracted By (MASD) For the Three Weights.

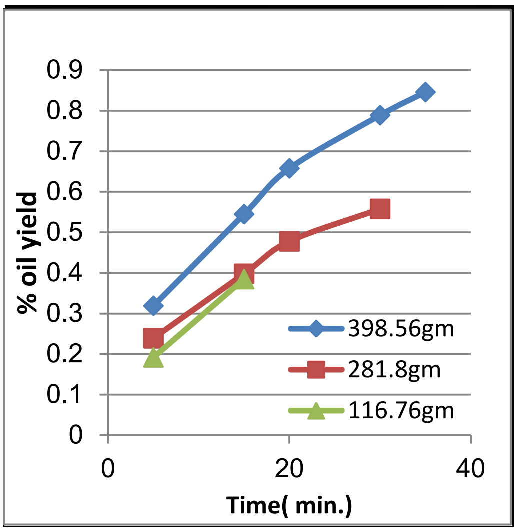

Fig.5 Effect of Extraction Time on Yield of Mandarin Oil Extracted By (MASD) For The Three Weights.

2- Effect of Grated Peels Weight

Fig.(2) & (3) show that for (SD) process since the extraction vessel is spherical shaped the action of steam was affected by the content of grated peels in the vessel. For the weight (398.56gm) the vessel was fully loaded and for weight (281.8gm) the vessel was half loaded when the steam passes from the bottom of vessel through the grated peels toward the top few amounts of peels at the side were far from the action of steam and extraction was at the upper and lower layers of peels where the steam moved freely.

It can be notes that (116.76gm) weight had the least space in extraction vessel and it was totally exposed to steam where it moved freely charged with essential oil, for this reason the weight (116.76gm) subjected to (SD) process gave yield (0.969%), while for mandarin with weight(116.76gm) after (50min.) the yield was (0.848%).

In (MASD) process oil yield increases with increasing the weight exposed to microwave power as shown in Fig. (4) & (5). For orange peels having weight(116.76gm) exposed to microwave power (265W) for (20min.) the yield was (0.532%), while for weight(281.8gm) exposed to same power for (35min.) the yield was (0.606%) and for weight (398.56gm) under same conditions the yield was (1.091%).

For the weight(398.56gm) the yield obtained by (SD) and (MASD) processes were close no striking differences found but the weights (281.8gm, 116.76gm) their yields obtained by (SD) were higher than (MASD) this means that microwave irradiation is inefficient with low weights, for orange having weights(281.8gm, 116.76gm) the oil yield extracted by (SD) was: (0.860%, 0.969%) while for (MASD) for these weights the yield was: (0.606%, 0.532%).

(MASD) method need the presence of water as "in situ" water in the plant material tissue, it is already known that only water in a liquid state absorbs microwaves but steam or even ice do not absorb microwaves because in the gas state the molecules are too far each other to have frictions, and in solid state the molecules are not free to move and rotate to heat [13] , for this reason (MASD) process is more efficient with high weights of peels for their high water content in tissues and the yield decreases with low weights for their low water content in their tissues. These results are in agreement with results obtained by Margosan et al. (2001) [18].

3- Effect of Microwave Power

According to previous research reports, essential oil yield differs in different microwave powers therefore; the effect of microwave power was studied. In principle, higher electromagnetic energy absorption could result in more power dissipated inside the plant material and generating more effective molecular movement and heating, leading to an improvement in the extraction efficiency [10].

As shown in Fig. (6) & (7) essential oil was extracted from three types of citrus peels each type having a weight(398.56gm), exposed to microwave powers:(135W, 265W, 445W) with exposure time to irradiation power:(5min., 10min.). In this method the yield of essential oil was affected by the amount of heat applied by microwave power.

At the beginning of extraction process oil yield increased by increasing microwave power from (135W) to (265W) for orange the yield was after (5min.) of exposure to these powers: (0.194%, 0.526%) respectively and after (10min.) of exposure to these powers the yield increased: (0.701%, 0.818%) respectively, moreover increasing in extraction time the yield increases slightly by power (265W) in comparison with power (135W), the yield was increasing until the extraction ended after (35min.) with yield: (1.150%, 1.091%) for powers: (135W, 265W) respectively; same results was obtained for mandarin.

When the power was raised to (445W) the yield was lower than yield obtained by the powers (135W, 265W) from the beginning to the end of extraction process.

Citrus essential oils are highly sensitive to the variation of processing temperature result from high microwave power (265W, 445W) , they can cause thermal degradation to essential oil, this fact explain why oil yield was lower for high microwave powers (265W, 445W). So in order to avoid that it would be better to use low microwave power (135W) for complete recovery of essential oil, these results are in agreement with results obtained by Chun-Hui et al.(2012) [19].

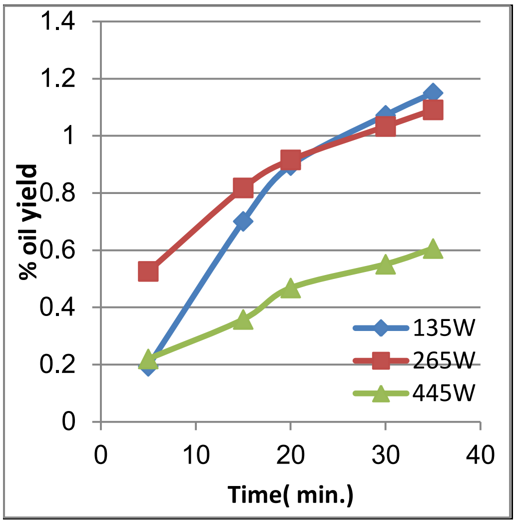

Fig. 6, Effect of Microwave Power on Yield of Orange Oil Extracted By (MASD)

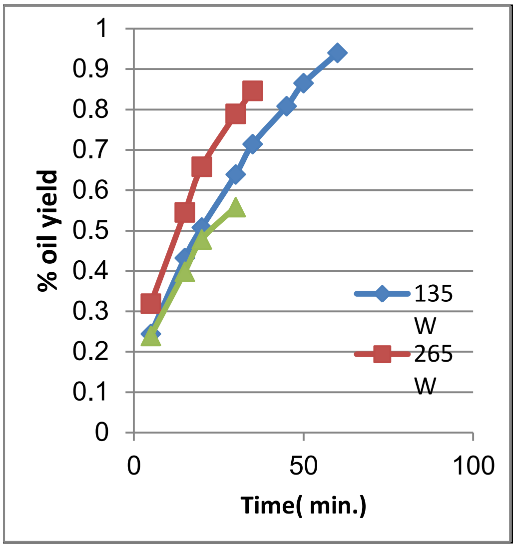

Fig. 7, Effect of Microwave Power on Mandarin Oil Extracted By (MASD)

4- Effect of Citrus Peel Type

Essential oil is present in citrus peels in oil sacks and glands which are located at different depths in citrus peel, the amount of this oil differ from citrus type to another, also conditions of extraction process play role in the complete recovery of essential oil [20].

It can be notes from Fig. (8) & (9) that for orange and lemon with weight (398.56gm) the yields were close for (SD) with extraction time (45min.): (1.095%, 1.061%) respectively, and they were higher than mandarin:

(0.707%) with extraction time (75min.) while for (MASD) the yield of orange and lemon after (35min.) extraction time was: (1.150%, 1.115%) while for mandarin after (60min.) the yield was (0.940%).

For both methods the yield of orange and lemon were close same and were higher than that for mandarin, where oil yield for mandarin had longer extraction time and gave lower yield than orange and lemon in both methods, the reason for that is the nature of mandarin peel tissue, it do not have much oil glands contained in peels to be ruptured by steam or microwaves to release the oil [18], these results are in agreement with results obtained by Meklati et al. (2007) [21].

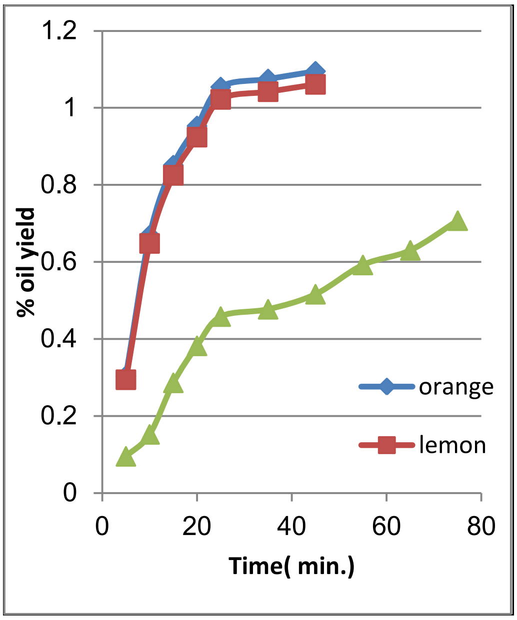

Fig. 8, Effect of Citrus Type on Yield of Essential Oil Extracted By (SD) From Citrus Peel Weight (398.56gm)

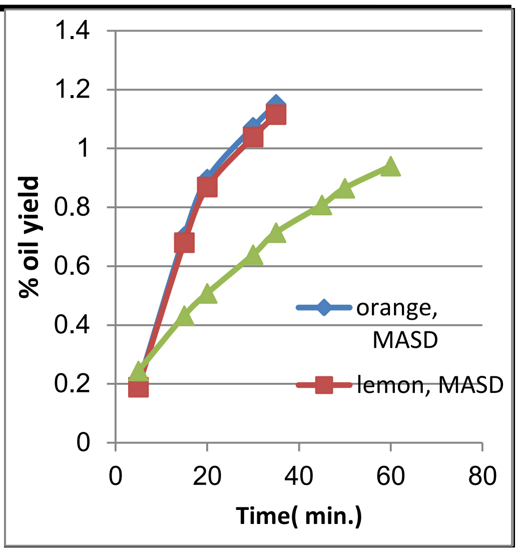

Fig. 9, Effect of Citrus Type on Yield of Essential Oil Extracted By (MASD) From Citrus Peel Weight (398.56gm)

5- Extraction Kinetics

As shown in Fig. (10) & (11) the (MASD) process can give essential oil with higher yield and better quality in shorter extraction time than (SD) process when the power is low, the use of longer exposure time would be helpful for the complete recovery of essential oil when microwave power is not too high to avoid thermal degradation of essential oil.

By analyzing these figures two phases are observed in (MASD) yield the first step is represented by a rapid increase in the yield followed by a second step corresponding to a slight increase until reach the end of extraction, the rapid increase in the yield during the first step suggests that the essential oil is easily accessible by the steam due to action of microwaves in rupturing oil glands while the steam passes charged with essential oil. For (SD) method three phases are observed the first step is represented by an increasing line which characterizes the first quantities of extracted essential oil this phase is followed by second increasing line representing the diffusion of essential oil from the middle layers of citrus peels, the third phase corresponds to a horizontal line which marks end of extraction, these results are in agreement with results obtained by Farhat et al. (2009) [13].

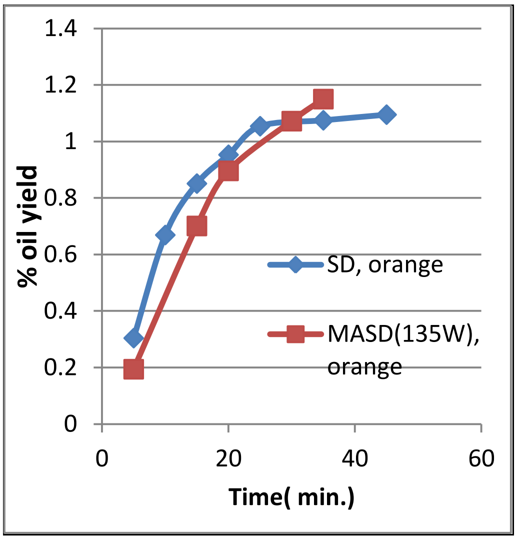

Fig. 10, Yield of Orange Oil Extracted By (SD) and (MASD) As Function Of Extraction Time

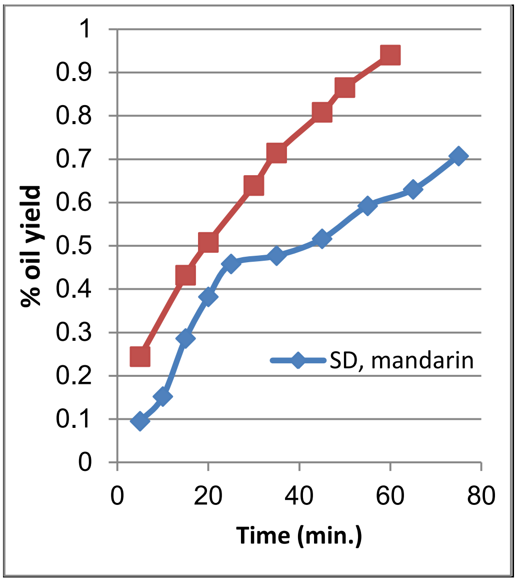

Fig. 11, Yield of Mandarin Oil Extracted By (SD) and (MASD) As Function Of Extraction Time

6- Gas Chromatography (GC)

Chemical identification of essential oil was investigated by Gas Chromatography equipped with (FID) detector (BACKARD, 438A, U.S.A). Essential oil extracted from (orange, lemon, mandarin) peels is mainly composed of Limonene, Linalool, Citronellal, Nerol, Geranial etc. among many other components. The relative amounts of these components varied according to type of citrus peel and conditions of extraction process.

From Table (1) for mandarin oil the amount of limonene extracted by (MASD) in low microwave power with long extraction time was (84.3891% at 135W in 60min.), while for (SD) it was (83.0271% in 75min.) and decreased with increasing microwave power, for orange oil it was (80.9661% at 265W in 35min.) while for (SD) it was (83.2189% in 45min.). For orange and lemon when microwave power was increased the amount of limonene decreased and was lower than that extracted by (SD).

So when using microwave power for extraction the best condition is exposure to low microwave power for long time. The influence of microwave energy on extraction is strictly thermal and it highly accelerated extraction process, phenomenon which was already described by Chemat et al. (2006) [22].

Table 1, Amount of Limonene extracted from citrus peels by (SD), (MASD) and conditions of extraction.

Citrus peel	Limonene content% [23]	Amount of limonene % for (SD)	Amount of limonene % for (MASD)
Mandarin	94.62%	83.0271	84.3891
Orange	91.40%	83.2189	80.9661
Lemon	65.44%	65.2867	59.156

Conclusions

According to the results obtained from this study, the following conclusions are obtained:

1- The extraction yield of oil increased with time for the two methods: (SD), (MASD) but, the yield of oil extracted by (MASD) reached equilibrium in shorter time than that of (SD).
2- The weight (116.76gm) subjected to (SD) process: in orange and lemon gave yield (0.969%, 0.939%) higher than weight (281.8gm) which gave yield (0.860%, 0.834%) respectively, while for mandarin this weight gave yield (0.848%) higher than the other weights (398.56gm, 281.8gm) which gave yields (0.707%, 0.648%) respectively.
3- It would be better to use low microwave power (135W) for complete recovery of essential oil and higher microwave power (265W) can be used but for short exposure time to avoid thermal degradation of essential oil.
4- Oil yield for mandarin had longer extraction time and gave lower yield than orange and lemon in both methods.
5- The amount of limonene extracted by (MASD) was higher than that extracted by (SD) for mandarin.

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