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Extraction of Essential Oils from Some Types of Umbelifera Family using Microwave-Assisted Water Distillation

Asaad Rehman Saeed Al-Hilphy Dhia Falih Al-fekaiki Rafal Abdul hussein
Food Science Dept., Agric. College, Basrah University, Basrah, Iraq

Abstract
Microwave oven has been modified to essential oil distiller. Essential oils were extracted from seeds of caraway, cumin, aniseed and fennel by using microwave-assisted water distillation (MAWD) and traditional water distillation (TWD). Extraction temperature, oil yield (%), consumed power (kWh) and physical characteristics have been investigated. Mathematical modeling was used to predict of oil yield values. The results showed that the extraction temperature of essential oils by microwave-assisted water distillation was higher than traditional water distillation. Oil yield extracted from caraway, cumin, aniseed and fennel by using MAWD was significantly higher than TWD. The consumed power by MAWD was significantly less than TWD. A theoretical data from mathematical modeling was closed to practical data, moreover the determination coefficient ranged between 0.93-0.99. Essential oils extracted by MAWD and TWD have a high effect on the inactivation of Bactria. The results showed that the activity of linoleic acid antioxidants of extracted essential oils by MAHD and THD were convergent.
Keywords: essential oil, microwave, water distillation, Umbelifera

1. Introduction

Aromatic plants such as caraway, cumin, aniseed and fennel are belong to Umbelifera family. Essential oil of cumin (cumin cyminum) and fennel (Foeniculum Vulgare) are used to food preservation and food flavor (Riaz et al.2012; Hel and Huang, 2011). As well known, the caraway (Curium Carvi) is used to inhibit of microbes and fungus (Seidler et al., 2013). Essential oil of aniseed (Pimpinella Anisum) is added to beverages, candy and pastries for providing it with the flavor (Zonglialng, 2012). Microwave energy is used in many applications with frequency of 2.45 GHz. such as active ingredient extraction from plants (Chan et al., 2011), essential oil extraction (Wei et al., 2013; Fadel et al., 2011).

Essential oils are extracted by using several methods are: water distillation, steam distillation, water and steam distillation, organic solvent extraction, microwave hydr-odiffusion and gravity (Bousbia et. al., 2009), microwave-assisted hydro-distillation, extraction by using supercritical CO2 (Glisic, 2010), ultrasonic extraction and solvent free microwave extraction (Lucchesi et al., 2004) and Ohmic heating extraction (Al-Hilphy, 2014). On the other hand, many researchers are used microwave energy in some new applications (Wakte et al., 2011; Sahraoui et al., 2004; Liu et al., 2012; Sarker and Nahar, 2012). In fact, there is a few published researches have studied the microwave-assisted water distillation. Microwave-assisted water distillation has been reported for essential oil extraction from Rosemary (karakaya et al., 2012), Rosmarinus Dwarfed Cinnamomum Camphora var. Linaolifera Fujito (Fadel et al., 2011) and Lippia alba (Stashenko et al., 2004). Wei et al. (2013) have extracted the essential oils (Owarfed Cinnamomum Comphora var Linaolifera Fujita) using microwave-assisted hydro-distillation and compared it with the conventional hydro-distillation, they found that the required time to extract of essential oil using microwave-assisted hydro-distillation and traditional hydro-distillation were 37.5 and 120 min. respectively and oil yield was 1.73 and 1.71 % respectively.

Fadel et al.(2011) stated that the total yield of essential oils obtained by using hydro-distillation and microwave-assisted hydro-distillation were 1.21 and 1.47% respectively. There are no published articles about extraction of essential oil from caraway, cumin, aniseed and fennel by using microwave-assisted water distillation. Therefore, the present study aimed to extraction of essential oils from caraway, cumin, aniseed and fennel and study of oil yield and extraction time, in addition to the use of mathematical modeling of oil yield.

2.0 Materials and methods

2.1 Microwave-assisted water distillation

Microwave oven (power of 1000W and frequency of 2.45 GHz) has been modified to essential oil distiller by water distillation method as shown in figures 1and 2. 500 W power levels were used to carry out experiments. 40 g of caraway, cumin, aniseed and fennel were placed in a 500 ml. volumetric flask containing 400 ml. of distilled water. The flask was put within the microwave oven. The condenser put on the top of microwave and it connected with the flask. This setup is like the Clevenger apparatus but the source of energy in microwave-assisted water distillation is the electromagnetic, and resistance heater was used in Clevenger apparatus (traditional water distillation).

Traditional water distillation (TWD)

2.2 Clevenger apparatus (traditional water distillation) was used to extract the essential oil from caraway, cumin, aniseed and fennel seeds. A 40 g weighted of each seed type and placed into extraction flask of the apparatus then 500 ml. of water has been added into the flask.

2.3 Extraction temperature
Extraction temperature was measured by the thermocouple type cu-constantan.

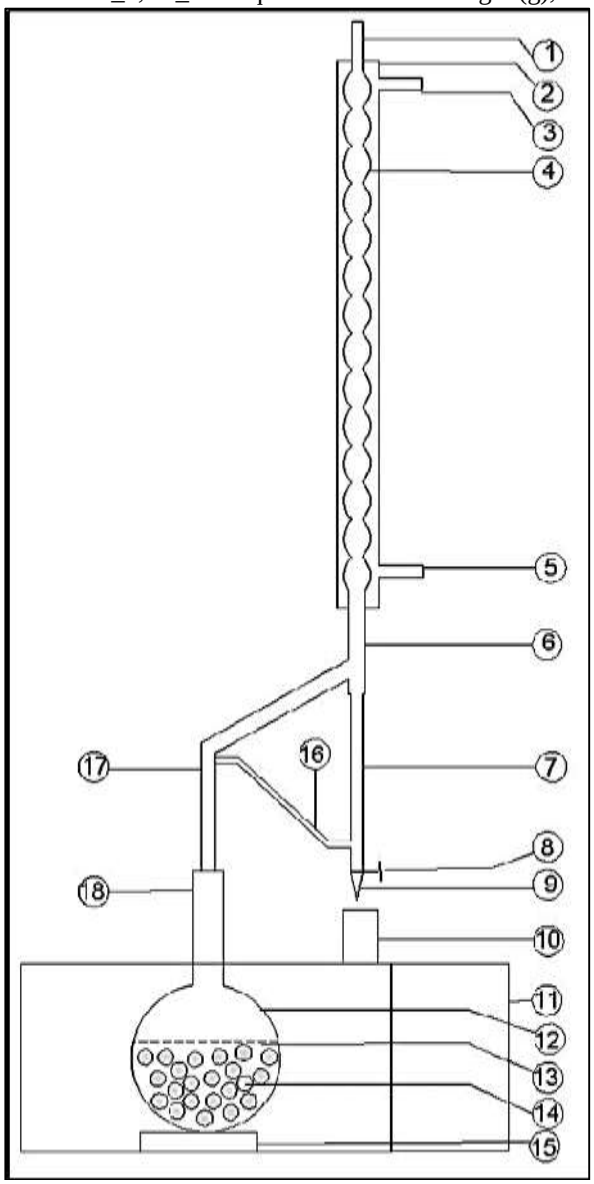
2.4 Consumption power
Consumption power has been calculated according to the following equation:

$$P = IVt \quad (1)$$

Where: P, V, t are consumption power (W), voltage and extraction time respectively.
Oil yield was calculated from equation 2:

$$q = \frac{W_o}{W_s} \times 100 \quad (2)$$

Where: W_o, W_s and q are essential oil weight (g), seed weight and oil yield (%) respectively.



1.ventilation port, 2.condenser, 3.outlet hot water, 4.requlated tube, 5.inlet cold water, 6.glass pipe, 7.measuring tube, 8.valve, 9.discharge port, 10.container, 11.microwave, 12.round flask, 13.water level, 14.aromatic plant, 15.base, 16.recycling tube or return tube, 17.vertical glass tube,18. Rubber tube connector
Figure 1. Microwave-assisted water distillation apparatus.

Figure 2. Photograph of microwave-assisted water distillation apparatus (A) and traditional water distillation (Clevenger) (B).

2.5 Mathematical modeling

The solver in excel program has been used to predict of the constants (A and k) which used in the following equation:

$$\frac{q_o - q}{q_o} = Ae^{-kt} \quad (3)$$

Where: q, qo, k, A are mean of essential oil yield at time t, essential oil yield into seeds, effective diffusion coefficient and constant.

Root mean square error (RMSE), Chi square (x2) and determination coefficient were calculated from the following equations:

$$RMSE = \frac{\sqrt{\sum_{i=1}^{N}\left(q_{theo.} - q_{prac.}\right)^2}}{N} \quad (4)$$

$$X^2 = \frac{\sum_{i=1}^{N}\left(q_{prac.} - q_{theo.}\right)^2}{N - n} \quad (5)$$

$$R^2 = \frac{\sum_{i=1}^{N}\left(q_{theo.} - \overline{q_{prac.}}\right)^2}{\sum_{i=1}^{N}\left(q_{prac.} - \overline{q_{prac.}}\right)^2} \quad (6)$$

Where: q_(prac..), q_(theo..), N, n are practical oil yield (%), theoretical oil yield (%), values number and

constants number respectively.

2.6 Specific gravity of essential oils
Specific gravity of essential oils was determined by pycnometer of 10 ml. at 25 oC temperature according to Pearson (1976).

2.7 Viscosity of essential oils
Viscosity of essential oils has been measured by Ostwald apparatus type D according to Sathe and Salunkne (1981).

2.8 Refractive index of essential oils
Refractive index has been measured of essential oils at 25 oC temperature by refractometer according to A.O.C.S (1971).
Antioxidant activity has been calculated according to Bersuder et al., 1998. Inactivation Diameter of Bactria has been measured according to Gupla et al.,(2008). Completely randomize design has been used for analyzing data.

3.0 Results and discussion
Figures from 3 to 6 illustrate changing temperature of essential oils extraction from caraway, cumin, aniseed and fennel by using microwave-assisted water distillation (MAWD) and traditional water distillation (TWD). The results showed that the extraction of essential oils from caraway, cumin, aniseed and fennel was increased significantly ($p<0.05$) with increasing extraction time. Heating by using MAWD was higher than TWD. Maximum temperature of essential oils extraction from cumin by using MAWD and TWD reached to 98.65 and 96.21 oC respectively while in the case of fennel it reached to 88.88 and 93.76 oC respectively. These differences in heating temperatures among aromatic plants are due to variation of dielectric constant of the plants.

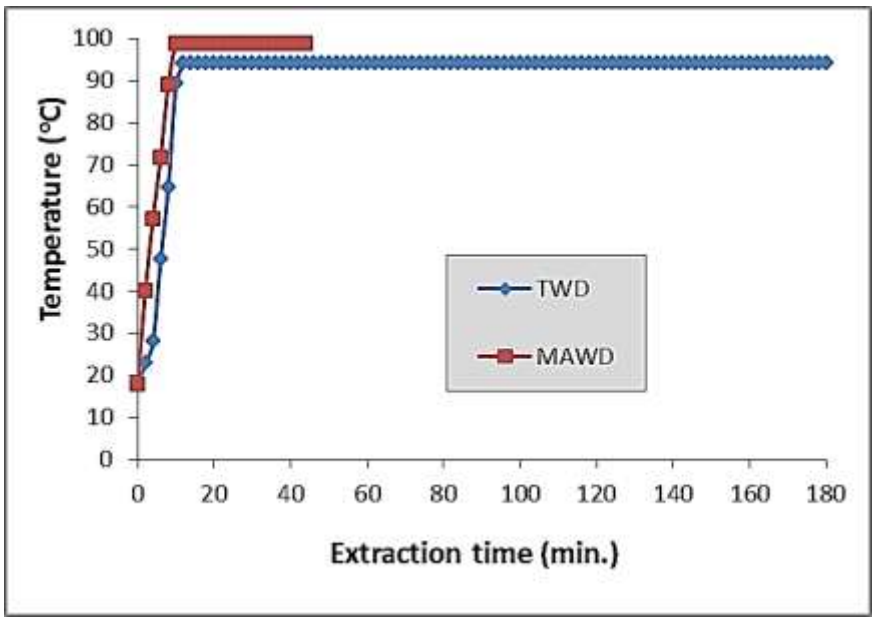


Figure 3.Temperature of oil extraction from caraway by microwave-assisted water distillation (MAWD) and traditional water distillation (TWD).

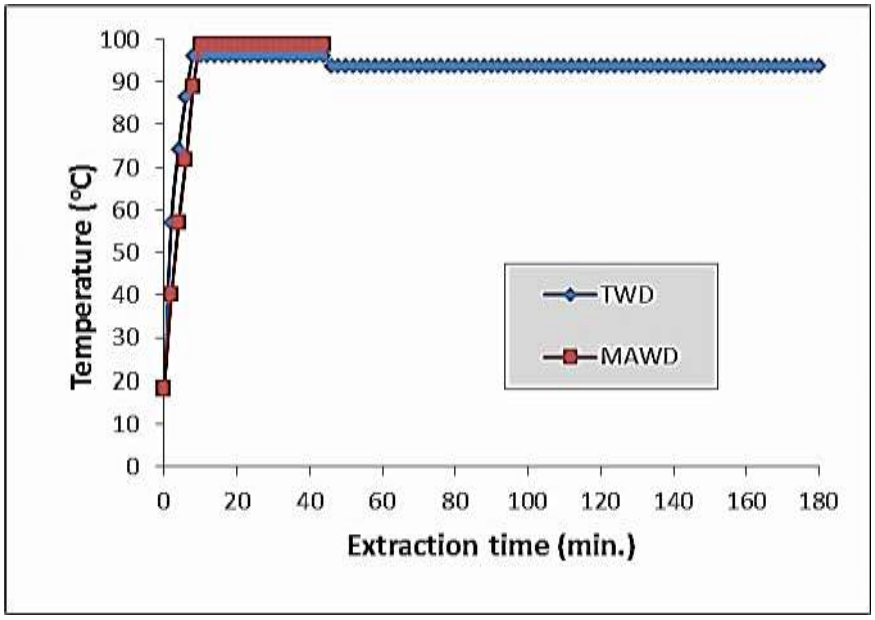


Figure 4.Temperature of oil extraction from cumin by microwave-assisted water distillation (MAWD) and traditional water distillation (TWD).

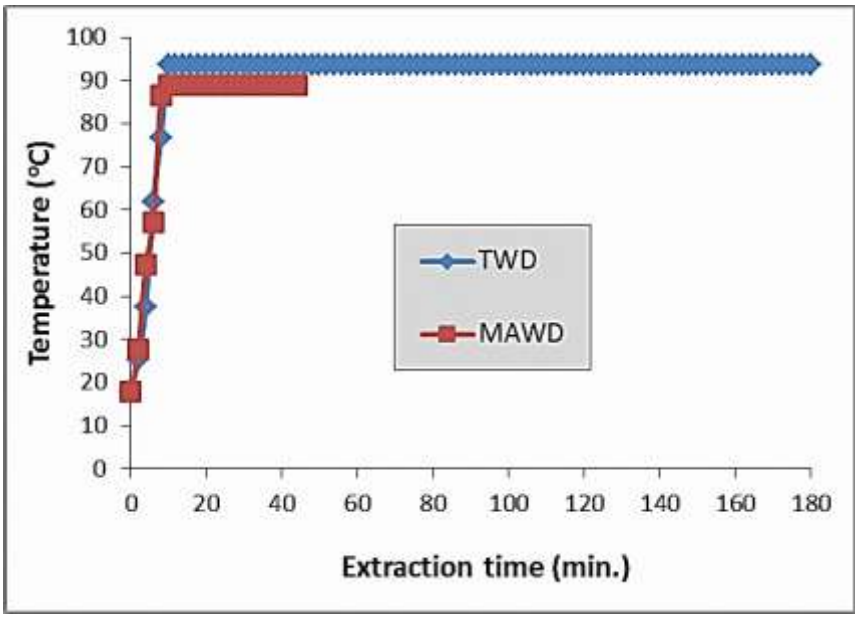


Figure 5.Temperature of oil extraction from fennel by microwave-assisted water distillation (MAWD) and traditional water distillation (TWD).

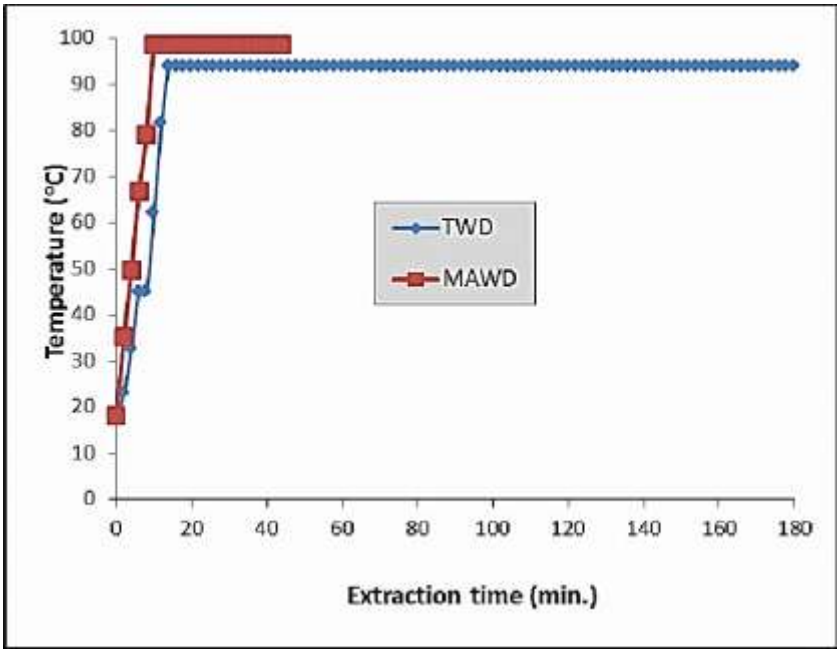


Figure 6.Temperature of oil extraction from aniseed by microwave-assisted water distillation (MAWD) and traditional water distillation (TWD).

Figure 7 illustrates the essential oil yield (%) extracted from aromatic plants (caraway, cumin, aniseed and fennel) by using microwave-assisted water distillation (MAWD) and traditional water distillation (TWD) by Clevenger apparatus. The results showed that the oil yield extracted from aniseed and fennel by MAWD was significantly ($p<0.05$) higher than TWD. For example, the oil yield extracted from aniseed by MAWD and TWD were 3.7 and 2.8 % respectively. These because of increasing heat effective due to increase randomize ions movement velocity that led to increase the impacts among them. All these factors led to destroy of cell walls which containing oil and release of a bigger quantity of oil into water. Chemat et al., (2005) showed that heat by microwave produces variation in the pressure between inside and outside of plant cell and most compounds are readily release with increase mass transfer coefficient and outside cell wall is destroyed completely. Kapas et al. (2011) stated that essential oil extracted by microwave was higher by 7.5 % compared with hydro-distillation. Also, Azar (2011) demonstrated that oil yield by using microwave was higher than water distillation. The results also showed that the difference between MAWD and TWD in oil yield % extracted from caraway and cumin was insignificant.

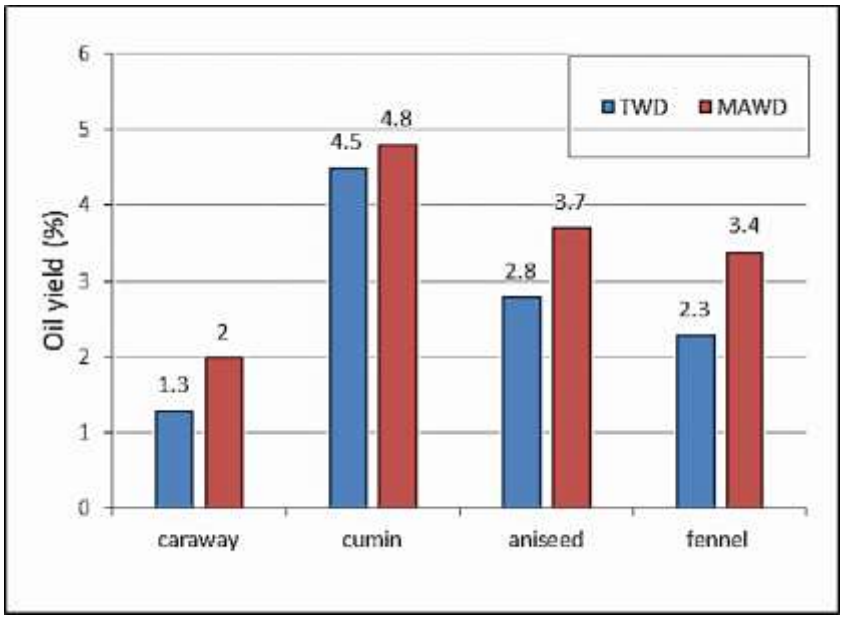


Figure 7. Oil yield extracted from aromatic plants by MAWD andTWD.

It can be seen from figures 8, 9, 10 and 11 that the practical and theoretical oil yield extracted from caraway, cumin, aniseed and fennel by using MAWD and TWD has been increased significantly ($P<0.05$) with

increasing extraction time. Also, the results showed that required time to extract essential oil from cumin, fennel, caraway and aniseed by MAWD was 44 min., while the extraction time was increased to 180 min. by using TWD for every type of seeds in the present study. This because of the heat transfers from inside to outside and heat loss is very little. In addition, most of electric energy has converted to heat energy absorbed by water and made the heating by MAWD faster than TWD. In the case of TWD, the heat flows from external to internal which causes great heat loss. So, the MAWD requires extraction time lesser than TWD. Frahat (2011) announced that the extracted essential oil from orange hells by MAWD and TWD required of 12 and 40 min. respectively. The results showed that there is a great convergence between practical and theoretical data with determination coefficient ranged between 0.939-0.999 and RMSE and Chi square values were very small, this refers to a good fitting between practical and theoretical. On the other hand, the constants A and k for all essential oils has been found via mathematical modeling by using solver program within excel program according to statistical parameters as shown in table 1.

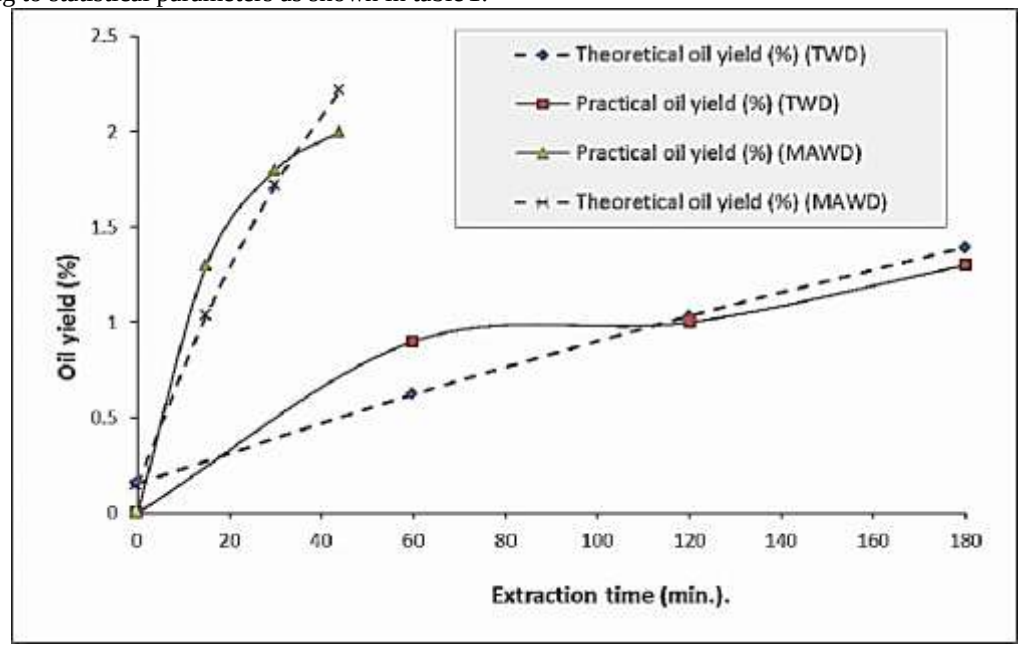


Figure 8. Theoretical and practical oil yield extracted from caraway by MAWD and TWD.

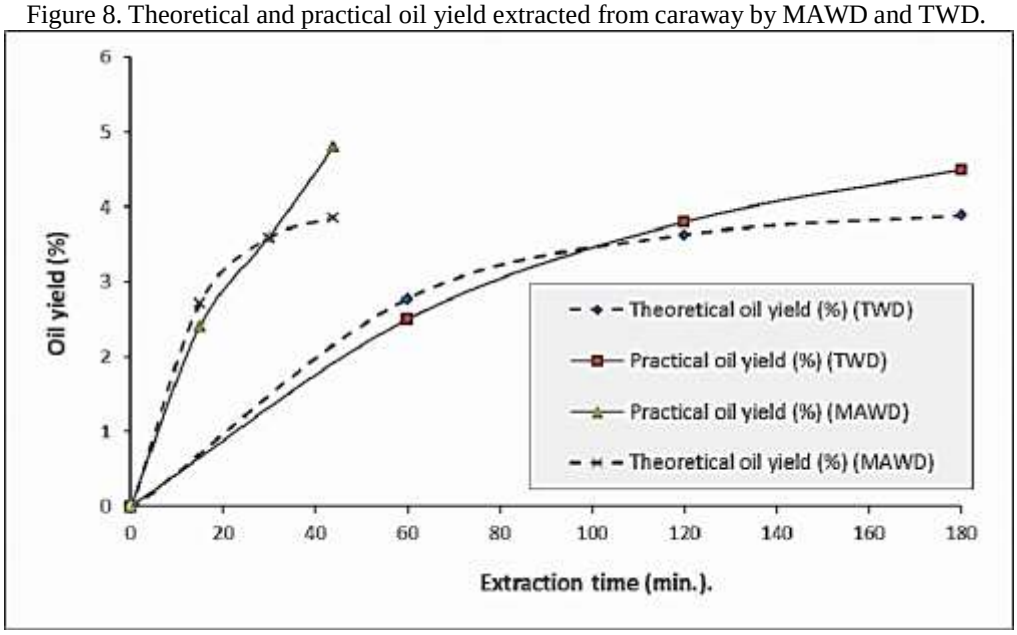


Figure 9. Theoretical and practical oil yield extracted from cumin by MAWD and TWD.

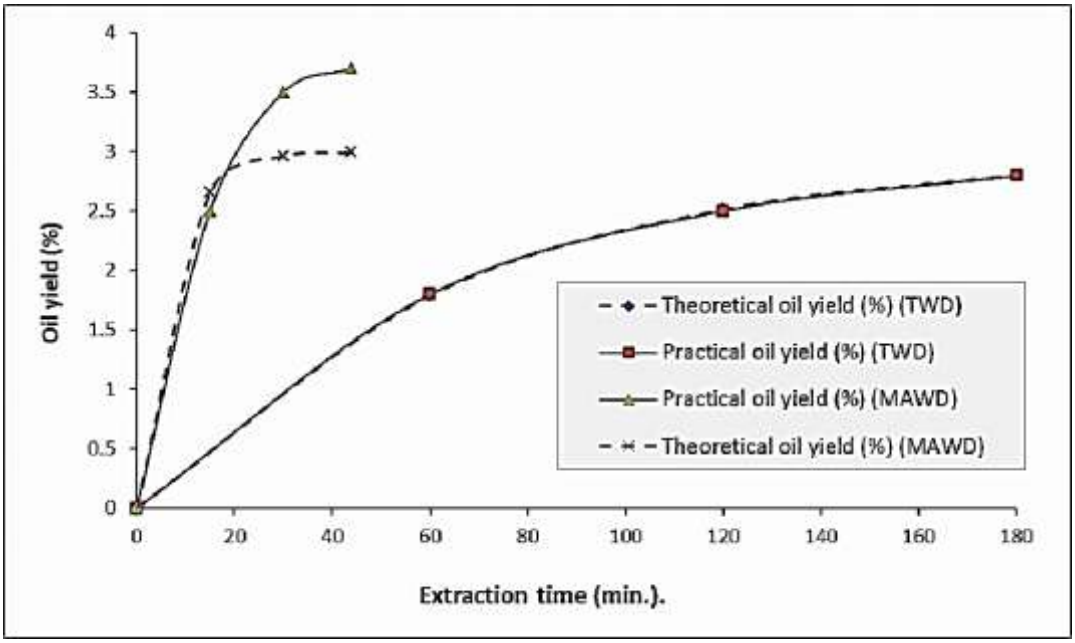


Figure 10. Theoretical and practical oil yield extracted from aniseed by MAWD and TWD.

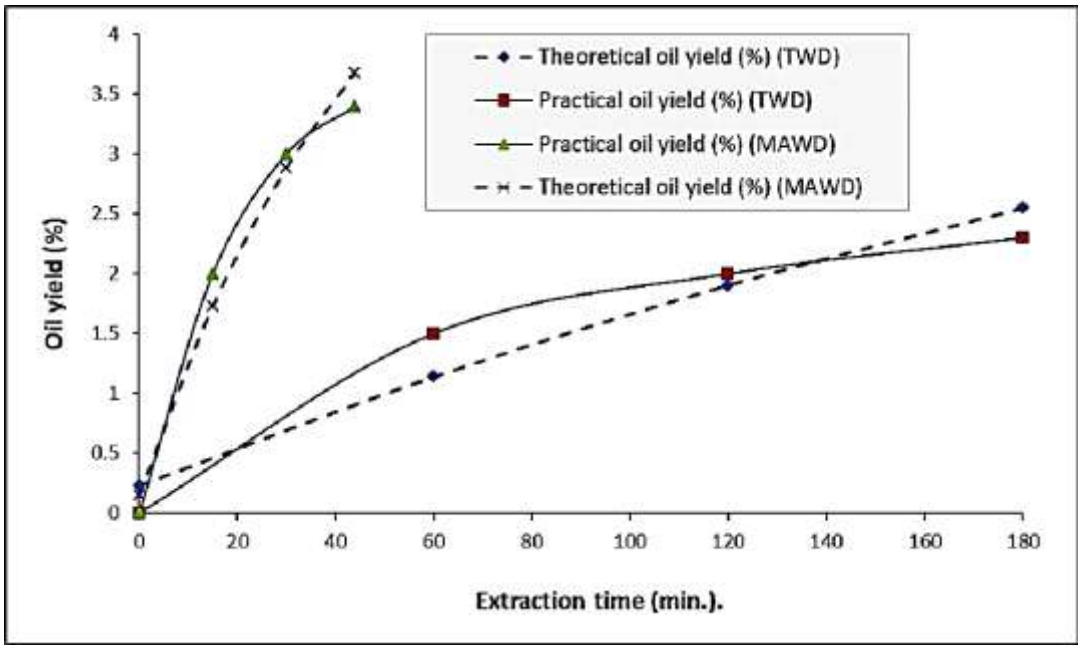


Figure 11. Theoretical and practical oil yield extracted from fennel by MAWD and TWD.

Table 1. Constants and statistical parameters calculated by mathematical modeling.

Plants types	Microwave-assisted water distillation					Traditional water distillation (Clevenger)				
	k	A	R^2	X^2	RMSE	k	A	R^2	X^2	RMSE
caraway	0.01750	0.96304	0.97029	0.07197	0.21905	0.00216	0.96149	0.93926	0.05547	0.19230
cumin	0.07639	1.01604	0.96995	0.49222	0.57284	0.01990	1.01246	0.98563	0.24091	0.40076
aniseed	0.14441	1.00315	0.97831	0.40629	0.52044	0.01511	0.99927	0.99997	0.00012	0.00908
fennel	0.02098	0.97501	0.98676	0.09282	0.24875	0.00285	0.96212	0.95867	0.12736	0.29139

It can be seen from figure 12 that the consumed power by MAWD and TWD were 0.73 and 6 kWh respectively. Therefore, using MAWD is economical in electric energy consumption. Fraht (2011) concluded that the required energy for extracting by microwave-assisted hydro-distillation is too lesser than other

traditional extraction methods.

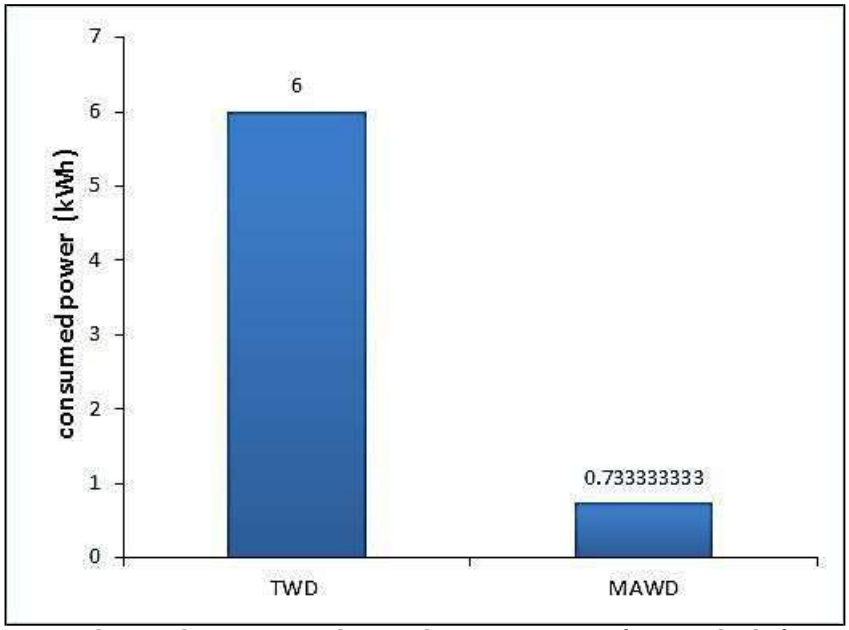


Figure 12.The consumed power by MAWD and TWD during extraction of essential oils from aromatic plants.

It can be seen from table 2 that refractive index of essential oil extracted by MAWD was less than TWD for all plants except Fennel. For example, refractive index of essential oil extracted from Cumin and Fennel by MAWD and TWD were 1.5056 and 1.5059, also 1.5561 and 1.5449 respectively. These results were higher than results of Sainin et al. (2014) who found that the refractive index of essential oil of cumin is 1.4675. Also, abo zaid (1992) stated that the refractive index of essential oil of cumin is1.4720. On the other hand, Abo zaid (1992) stated that the refractive index of essential oil of Caraway ranged between 1.4860 - 1.4878. Al mayah (2001) stated that presence of slow evaporation ingredients in essential oil such as high ratio of oxygenated compound leads to increase refractive index of essential oils. As well as, the variation in the refractive index is attributed to presence of high ratio of unsaturated bonds (Al mayah, 2001). The results also showed that the refractive index of essential oil extracted from Fennel by MAWD was higher compared with other essential oils because of increasing number of unsaturated bonds that led to rise of refractive index of essential oil.

Table 2. Refractive index of essential oil had extracted by MAWD and TWD.

essential oils	MAWD	TWD
Caraway	$1.4941^{a} \pm 0.050$	$1.4843^{a} \pm 0.155$
Cumin	$1.5056^{a} \pm 0.169$	$1.5059^{a} \pm 0.115$
Aniseed	$1.5451^{a} \pm 0.173$	$1.5559^{a} \pm 0.112$
Fennel	$1.5561^{a} \pm 0.115$	$1.5449^{a} \pm 0.173$

The numbers shared with the same symbol are not significant.

Table 3 illustrates viscosity of essential oil extracted by MAWD and TWD. The results showed that the viscosity of essential oil extracted from aniseed, fennel and caraway by MAWD was insignificantly less than TWD. While, the viscosity of essential oil extracted from Cumin by using MAWD and TWD have the same value of viscosity.

Table 3. Viscosity (pa.s.) of essential oil had extracted by MAWD and TWD.

essential oils	MAWD	TWD
Caraway	$1.762\times10^{-3\,a} \pm 0.00023$	$1.763\times10^{-3\,a} \pm 0.00028$
Cumin	$2.091\times10^{-3\,a} \pm 0.00057$	$2.091\times10^{-3\,a} \pm 0.00057$
Aniseed	$3.495\times10^{-3\,a} \pm 0.\ 0.00011$	$3.595\times10^{-3\,a} \pm 0.00011$
Fennel	$2.882\times10^{-3\,a} \pm 0.000115$	$2.883\times10^{-3\,a} \pm 0.00028$

The numbers shared with the same symbol are not significant.

Table 4 showed that the specific density of essential oils extracted from caraway, cumin, aniseed and fennel by MAWD and TWD was less than the specific density of essential oils that presence in the other literatures. Abo zaid (1992) stated that the specific density of essential oil extracted from cumin is 0.887. On the other hand, specific density of essential oil extracted from cumin by TWD and MAWD were0.724 and 0.744 respectively. These results agreed with Sainin et al. (2014) who found that the specific density of essential oil of cumin was 0.7455. Naher et al. (2012) found that the specific density of essential oil had extracted from aniseed

is 0.824; this result is higher than the specific density of essential oil extracted from aniseed by MAWD and TWD as illustrated in table 4. Damayanti and Setyawn (2012) pointed that the specific density of essential oil extracted from Fennel was ranged between 0.978-0.988.

Table 4. Specific density of essential oils extracted by MAWD and TWD.

essential oils	MAWD	TWD
Caraway	0.646 [a]±0.115	0.641 [a]±0.115
Cumin	0.744[a] ±0.173	0.724 [a] ±0.115
Aniseed	0.733 [a]±0.173	0.737 [a]±0.176
Fennel	0.825 [a]±0.057	0.927 [a]±0.057

The numbers shared with the same symbol are not significant.

Effect of essential oils concentration on inactivation diameter of Bactria:

Tables from 5 to 8 illustrate effect of essential oils concentration extracted from seeds of caraway, cumin, aniseed and fennel by using microwave-assisted water distillation (MAWD) and traditional water distillation (TWD) on the inactivation diameter of Bactria. The results showed that efficacy of essential oils in inactivation of Bactria was increased with increasing essential oils concentration and the concentration of 100% gave a highest inactivation. On the other hand, the efficacy of essential oils in the Bactria inactivation was varied. Caraway oil had a high inactivation effect compared with other essential oils in this study, where the inactivation diameters of Staphylococcus aureus , Escherichia coli, Pseudomonas aeruginosa Bactria by caraway oil extracted by TWD were reached 32, 28, 19 mm respectively at concentration of 100% and reached 22, 14, 18 mm respectively by using MAWD. From these results, inactivation of Bactria by essential oils extracted by TWD was higher than MAWD, it may be attributed to privation or damage of some compounds from essential oils during extraction by MAWD. In general, all essential oils in this study have a huge effect on the inactivation of Bactria. These results were agreed with many researchers who stated that essential oils extracted from different aromatic plants have a high effect on the positive and negative microorganism's inactivation. (Moreira et al., 2005; Hammer et al., 1999; Veldhuizen et al., 2007; Dimic et al., 2012). Iacobellis et al. (2005) announced that caraway oil contains many compounds which have prevent Bactria growth and fungi such as linalool (0.3 %) , carvone (23.3%), limonene (18.2%) and carvacrol (6.7%). Coisin et al. (2012) stated that cyclic phenolic compounds in the essential oils have inactivation effect on the microorganisms via changing composition and function of cytoplasm membrane which lead to cells death. Mohammad et al. (2013) found that inactivation diameter of Bactria by caraway oil reached 21.7, 22, 13 and 21 mm for L. monocytogenes ,S. aureusB. cereus S. typhimurium respectively.

Table 5. Effect of different concentrations from essential oil extracted from caraway by THD and MAHD on the mean diameter (mm) of Bactria growth inactivation

Essential oil concentration (%)	*Staphylococcus aureus*		*Escherichia coli*		*Pseudomonas aeruginosa*	
	THD	MAHD	THD	MAHD	THD	MAHD
0	0	0	0	0	0	0
10	5	5	0	0	0	0
20	7	5	5	5	0	0
40	10	6	8	7	8	6
60	20	21	22	18	12	11
80	26	25	25	21	15	14
100	32	30	28	25	19	16

Table 6. Effect of different concentrations from essential oil extracted from cumin by THD and MAHD on the mean diameter (mm) of Bactria growth inactivation.

Essential oil concentration (%)	*Staphylococcus aureus*		*Escherichia coli*		*Pseudomonas aeruginosa*	
	THD	MAHD	THD	MAHD	THD	MAHD
0	0	0	0	0	0	0
10	0	0	0	0	0	0
20	5	5	5	5	0	0
40	11	9	6	6	6	5
60	15	16	11	10	10	8
80	22	19	16	13	18	19
100	24	22	21	20	20	20

Table 7. Effect of different concentrations from essential oil extracted from fennel by THD and MAHD on the mean diameter (mm) of Bactria growth inactivation.

Essential oil concentration (%)	*Staphylococcus aureus*		*Escherichia coli*		*Pseudomonas aeruginosa*	
	THD	MAHD	THD	MAHD	THD	MAHD
0	0	0	0	0	0	0
10	0	0	0	0	0	0
20	0	0	0	0	0	0
40	7	6	9	7	6	5
60	10	11	11	8	9	7
80	13	12	16	12	13	12
100	16	14	20	17	14	13

Table 8. Effect of different concentrations from essential oil extracted from aniseed by THD and MAHD on the mean diameter (mm) of Bactria growth inactivation.

Essential oil concentration (%)	*Staphylococcus aureus*		*Escherichia coli*		*Pseudomonas aeruginosa*	
	THD	MAHD	THD	MAHD	THD	MAHD
0	0	0	0	0	0	0
10	0	0	0	0	0	0
20	0	0	0	0	0	0
40	8	7	5	5	5	5
60	10	10	8	9	6	7
80	17	15	10	10	8	9
100	20	18	13	12	10	9

Antioxidant Activity of Extracted Essential Oils:

Table 9 illustrates antioxidant activity of extracted essential oils from caraway, Cumin, aniseed, and fennel by using MAHD and THD. The results showed that the activity of linoleic acid antioxidants to extracted essential oils by MAHD and THD were convergent. This activity was increased with increasing essential oils concentration and the concentration of 100% gave a high value of antioxidant activity reached 69, 78, 60, and 73% for Cumin, caraway, aniseed, and fennel respectively extracted by THD, also reached 65, 80, 59, and 68% respectively by MAHD. The results also showed that activity of caraway oil as an antioxidant more superiority compare with other oils, when the concentrations of essential oils extracted by THD and MAHD were 10, 20, 40, 60, 80 and 100, their activity reached 15, 18, 32, 55, 68, and 78%, 16. 16, 37, 52, 65, and 80% respectively. These results were agreed with many researchers about essential oil have a high activity of antioxidant where Singh et al. (1998) concluded that the activity of antioxidant of cumin oil added to sunflower oil was higher compare with artificial BHT. In addition, the results showed that the phenolic compounds in extracted caraway have higher effect in hunt of free radical compare with artificial BHT (Bamdad et al., 2006). From these results, the essential oils can be used as a food additives instead of artificial antioxidants also, these essential oils have not any harm effect on the health.

Table 9. Antioxidant activity of essential oil extracted by MAHD and THD.

Essential oil concentration (%)	Antioxidant activity (%)							
	Caraway		Cumin		Aniseed		Fennel	
	THD	MAHD	THD	MAHD	THD	MAHD	THD	MAHD
10	15	16	11	10	9	11	10	12
20	18	16	13	12	10	13	11	14
40	32	37	20	18	23	20	15	16
60	55	52	30	31	37	32	21	17
80	68	65	45	48	45	45	59	54
100	78	80	69	65	60	59	73	68

4.0 Conclusions

In conclusion, extraction of essential oils by microwave-assisted water distillation was faster than traditional water distillation. Also, consumed power by MAWD was less than TWD. In addition, oil yield by MAWD was higher than TWD. Possible utilizing the mathematical model of ((qo-q)/qo)=Ae-kt to predict of oil yield. The effect of essential oils extracted from Cumin, caraway, aniseed, and fennel by using MAHD and THD on the Bactria inactivation diameter and antioxidant activity were significant.

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