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Development of steam essential oils extractor

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Abstract: *Steam distillation method of essential oils has been developed via design of apparatus consists of a long cylinder, it makes steam pass into it and reduces of steam velocity and temperature to less than 85 ^oC.The cylinder has a reciprocating net made of stainless steel which fixed on the shaft put into the cylinder. The aim of the shaft is to control on the quantity of plants and the distance from steam entrance port. The developed steam extractor compared with traditional steam extractor. An extraction temperature, oil yield (%), oil density (gm/cm^3), oil viscosity (pa.sec.), reflectance index, mean energy consumption per ml. of oil (kW. hr/ml.) were studied as well as, studying effect of oil extracted by developed steam extractor and the traditional on the diameter inactivation of microorganisms. GC-technique had been used to detection of chemical compounds of clove oil that extracted by developed team extractor and the traditional. The results showed that oil yield, viscosity and reflectance index of extracted oil by developed steam extracted was significantly higher than the traditional, but no significant effect of two extractors on the oil density and mean energy consumption per ml. of oil. In addition, the result showed that the inactivation diameter of Bactria using clove oil extracted by using developed steam extractor was significantly higher than oil extracted by using traditional steam extractor. On the other hand, there is no significant effect between two extractors on the inactivation diameter of E.Coli bacteria.*

Key words: *steam, extraction, clove oil.*

I. Introduction

Since the beginning of the middle Ages, water distillation and steam distillation devices are used to separate the volatile oils then developed during the Greeks, Romans and Arabs civilization (Abo zaid, 1992). Essential oils known as volatile substances evaporate easily when heated and contain a many different chemical compounds which give them their characteristics and their independent effects (Al-Tememi, 2009).

Essential oils are extracted from different parts such as roots, stems, leaves, buds , fruits, flowers, seeds, bark (Alhakeem and Hassan, 1985), essential oils are used in the medical and pharmaceutical purposes, food industries, cosmetics and perfumes, physical therapy, the fight against insects , diseases, dye fabrics, incentives jellies, plant growth regulators and paper industry (Munir and Hensel, 2009a).

There are a different techniques used to extract the essential oils such as hydro-distillation, soaking, solvents and steam distillation. Advantages of distillation methods are extraction of pure aromatic oils via evaporation of volatile aromatic compounds and staying away from harmful substances (Munir and Hensel2007b). There is a new method for extracting is extraction of essential oils by Ohmic heating through the conversion of plants to electric resistance, the electric current passes into plants (Al-Hilphy, 2014 and Hazwan*et. al*., 2012). Supercritical carbon dioxide is used to extraction of oils via raising the temperature of CO_2 to higher than 31.1 ^oC and pressure to higher than 73 bar(Handa etal., 2008). As well as, the microwave was used to extractessential oils. Also, the extraction by cold press, chemical extraction and extraction by solar steam generation via solar collector and transferred to the extraction unit (Al-Behadli, 2014).Most of oils are extracted at the beginning of the extraction process then decrease with time after that. The specific gravity and refractive index give an important information about the purity of the essential oil. Abu Zaid (1992) and Essien*et. al.* (2008) stated that the essential oils have a refractive index ranges between 1.4 - 1.69 , the low value of the refractive index means cheating essential oils by adding terpenes compounds.

As well as, essential oils color is an important property which determines the consumer acceptance of the oil product (Hamzah,*et al*., 2014). Pronpunyapat*et al.*, (2011) announced that the high refractive index and high relative density give a dark color of essential oils. Essential oil which has a high-density contains a resinous aromatic compounds that distinguish it from the rest of the other oils.

Chemat, (2010) noticed a pores resulting from the explosion of vesicles containing oil particles, this led to movement the oil out of the cells. Hamzah*et al*.(2014) stated that It's not getting a complete extraction of the oil when the steam is at atmospheric pressure and makes the cell incubator for oil more solid. In order to get the a lot of amount of extracted oil, it must be the extraction at highest pressure.Vinatoru et al.(1999) found that the high-pressure steam causes breaking and separation of the cell walls and produced larger pores, this led to passing a largest amount of steam through the pores, as well as a large amount of oil diffused from the cells.

The drawbacks of steam extraction method is the oil yield less and gets lose in some volatile compounds and breaking the heat- sensitive compounds , especially if the ranging temperature between 150-200

Celsius and steam pressure of 5-10 bar (Ghasemi*et. al*., 2011 ;Lawrence, 1995).93% of the various essential oils are extracted by steam distillation method and 7% of them is extracted by other extraction methods (Masango, 2005).Al-Hakim and Hassan (1985) stated that should note does not rising aromatic plant temperature too much, this causes drying steam. So, the steam distillation at low temperatures (such as 84 ^oC) prevents loss of essential oil and avoid drying aromatic plants by dry steam, also they pointed out that a high-speed steam entering into the distillation device leads to the exit of some metals and micro plant particles with extracted oil, while the slow speed of the steam helps to moisturize the raw materials by condensation of steam on them.

The extract clove oil from dried buds has significance in terms of flavor and its use in the food industry, such as meat, sausage, baked food, sweets, pickles and chewing gum, as it is fought for the bacteria and is used medically in the gurgling and enters in the manufacture of perfumes and soaps (Al-Hakim and Hassan 1985). The aim of this study is to extract essential oil at temperature less than 85 ^oC by new design of steam essential oil extraction apparatus.

II. Materials and methods

Developed steam extractor

Developed steam extractor consists of the following parts (Figure1-A and figure 2)

1. Steam generator.
2. Extraction unite: it consists of a tube made of stainless steel type 304, length of 120 cm and the outside diameter of 5 cm and a thickness of 2 mm. cylinder has a side slot in the bottom is used to entering steam, also it has another slot in the bottom with a valve to drain the condensate water in the cylinder. There is a slot in the upper of the cylinder is used to exit both the steam and oil together. There is a shaft inside the extraction cylinder, length of 125 cm provided with slide net base made of stainless steel. A thermocouple put in the extraction cylinder to measurement the plant temperature there.
3. Condensation unit: It is a tube heat exchanger made of glass has an internal corrugated tube in order to increase the heat exchange area also, transfer the steam and oil to the separator.
4. Separator: It is a flask of 1 liter capacity, provided with valve. Separator is used to separate the oil from water.

Traditional steam extractor

Traditional steam extractor consists of the following parts (Figure1-B and figure 3)

1. Steam generator.
2. Extraction unit: it consists of a tube made of stainless steel type 304 length of 32 cm and the outside diameter of 11.3 cm and a thickness of 4 mm has a side slot from the bottom to input steam. The tube provided with a cone containing a slot is used to exit oil and steam. Extraction unit was insulated by asbestos of 5 cm thickness.
3. Condensation unit: It is a tube heat exchanger made of glass has an internal corrugated tube in order to increase the heat exchange area also, transfer the steam and oil to the separator.
4. Separator: It is a flask of 1 liter capacity, provided with valve. Separator is used to separate the oil from water.

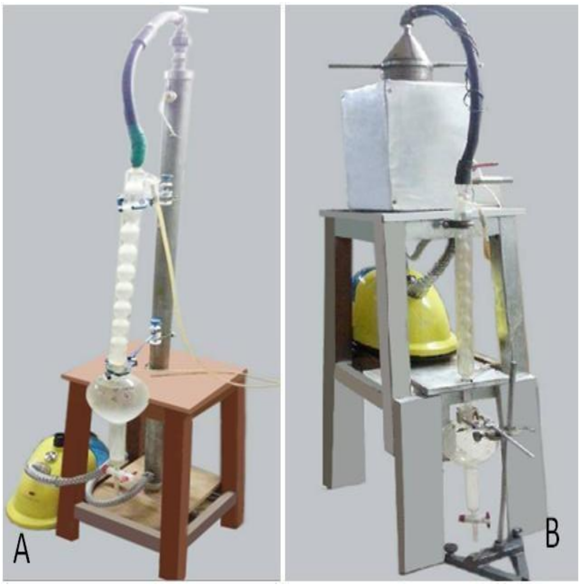
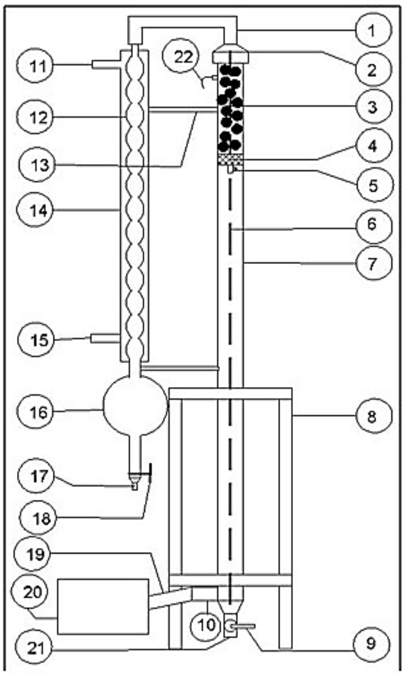
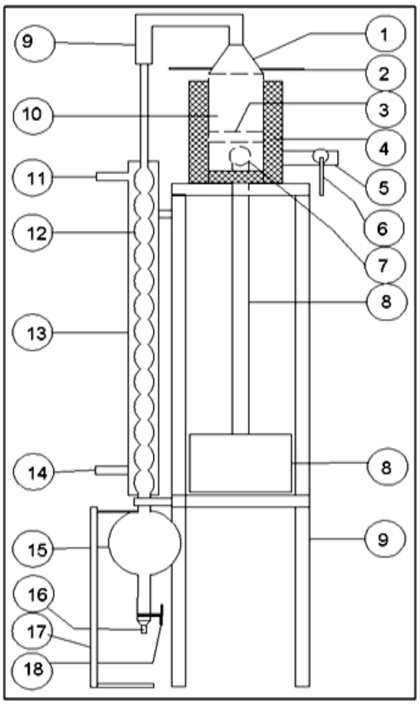


Figure 1**.** A-Developed steam extractor and B-Traditional steam extractor.



1. Heat plastic pipe, 2.cover, 3. Clove flowers, 4. Net metal, 5. Slide cylinder, 6.bar, 7. Cylinder 8. Wood matrix, 9. Valve, 10. Steam inlet, 11. Water outlet, 12. Corrugated pipe, 13. Bar, 14.outer cylinder, 15. Water inlet, 16. Separation flask, 17. Water out let, 18. Valve, 19. Heat pipe, 20. Steam generator, 21. Water out let, 22. Thermocouple.

Figure 2. Developed steam extractor.



1.Cover, 2.arm, 3. Metal net, 4. Insulator, 5. Drain pipe, 6.valve, 7. Steam inlet 8. Stem inlet, 9. Steam generator, 10. Wood matrix, 11. Heat plastic pipe, 12. Extraction cylinder, 13. Water outlet port, 14.condensation pipe, 15. Outer cylinder of condenser, 16. Cold water inlet, 17. Separation flask, 18. Water outlet, 19. Iron bearing, 20. Valve.

Figure 3. Traditional steam extractor.

Clove flowers

Clove flowers *(Eugeuia caruophyllus*) were bought from Basrah local market then washed by water. 150 g of clove flowers were put into the cylinder of extractor.

Temperature measurement

Clove flowers temperature during extraction had been measured by thermos-couple type cu-constantan. The temperature was calculated from the following calibration equation:

$$T = T_{amb} + 24.4416 * mV \quad (1)$$

Where: T is the clove flowers temperature during extraction (^oC), T_{amb} is the ambient temperature (^oC) and mV is the measured voltage (mv) by thermo-couple.

Oil yield

Yield of Clove oil extracted by developed and traditional method was calculated from the following equation:

$$Y = \frac{W_O}{W_S} \times 100 \quad (2)$$

Where: Y is the oil yield (%), W_O is the weight of extracted oil (g) and W_S is the sample weight (g).

Calculation of ratio of extracted oil

Ratio of extracted oil is calculated from equation 1, which explains that extraction of essential oil occurs via washing and diffusion mechanisms as follow (Milojevic et. al., 2013):

$$\frac{q}{q_\infty} = 1 - fe^{-k_1 t} - (1 - f)e^{-k_2 t} \quad (3)$$

Where: q is the quantity of clove oil extracted at time (t), $q\infty$ is the quantity of clove oil found in clove flowers at 100% extraction, f is the washing factor, k_1 is the washing constant, $k^2$is the diffusion constant.
If washing is very fast and occurs instantaneously $k \to \infty$ so equation 2 becomes:

$$\frac{q}{q_\infty} = 1 - (1 - f)e^{-k_2 t} \quad (4)$$

If no washing of the essential oil occurs (f=0), then equation 3 becomes:

$$\frac{q}{q_\infty} = 1 - e^{-k_2 t} \quad (5)$$

Clove oil density

Clove oil density was determined by using bottle density, it's volume of 10 ml. at 25 ^oC temperature, according to the method that cited in (20).

Refractive Index

Refractive Index of clove oil measured at 20 ^oC temperature using Abbe refractometer made of England according to the method cited in American Oil Chemists Society.

Clove oil viscosity

Clove oil viscosity was measured by using Ostwald instrument size D according to the method of Sathe and Salunkne (23).

Inactivation Diameter of Bactria

Clove oil extracted by developed extractor and traditional method by adding 0.2 ml. of clove oil. The following used isolates:

Staphylococcus aurens , Pseudomonas sp. , Escherichia coli , Bacillus sp. , Salmonella sp.

Bactria inactivation was tested by using clove oil extracted against Bactria according to agar well diffusion technique. Also, Bactria isolates has been inactivated for 18 – 24 hours at 37 ^oC temperature then the nutrient agar was prepared and cast into sterilized dishes with 20 ml. volume for every dish, when nutrient agar become solid, 0.1 ml. of suspension of activated Bactria then loop was used to spread on the consolidated dish then hole done of 7 mm diameter by using cork drill then eliminated for hole formation, after that 0.2 ml. of clove oil was added into the holes and the dishes incubated in the incubator at 37 ^oC temperature for 24 hours. Then the inactivation diameters had been measured.

Determination of effective compounds in clove oil

Effective compounds in clove oil extracted by developed and traditional extractor have been determined by using GC-Mass apparatus (GCM-QP2010 Ultr) type Shimadzu.

Statistical analysis

Completely randomize design was used for analyzing data, least significant differences (LSD) at significant level of 0.05 was used for comparing among data. Analyzing data was carried out by using SPSS program (24).

III. Results and discussion

Clove temperature

It can be seen from figure 1 that clove temperature by using developed steam extractor was lower than traditional steam extractor. This because the steam lost a part of its energy through the cylinder using developed steam extractor. Mean temperature reached to 84.77 and 97.76 ^{o}C by using developed steam extractor and traditional steam extractor respectively. Also, the required time to reaching these temperatures are 30 and 15 min. respectively. In addition to the required total time to complete extraction were 180 and 150 min. respectively.

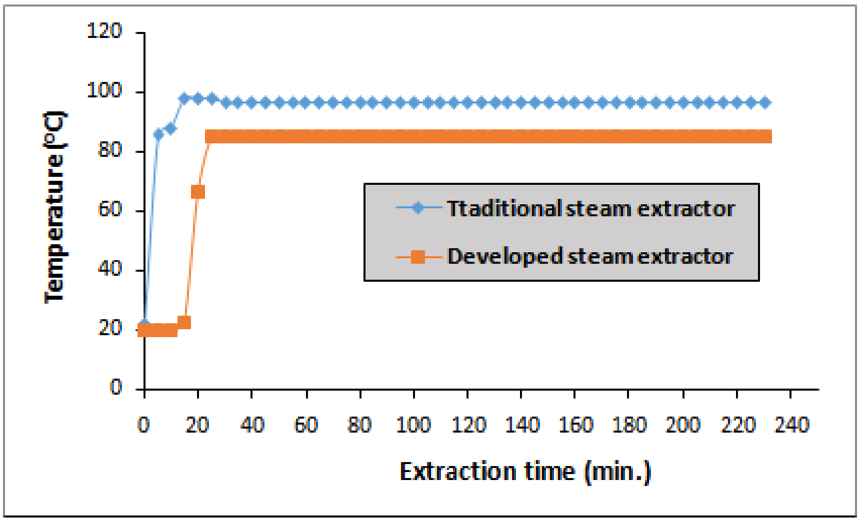


Figure 5. Clove oil temperature during extraction by developed and traditional extractor.

Physical properties of clove oil extracted by developed and traditional extractor:

Table 1. Shows that oil yield of clove oil extracted by developed extractor was significantly higher than oil yield of clove oil extracted by traditional extractor. Oil yield of clove oil extracted by developed and traditional extractor reached 13.5 and 11 % respectively. This because oil did not decomposition and did not loss its volatile compounds at using developed extractor compare with traditional extractor. There is a significant differences in the density of oil extracted by developed and traditional extractor. Oil density extracted by developed extractor was significantly higher than oil density extracted by traditional extractor.

Also, table 1. Illustrates that oil viscosity extracted by developed extractor was significantly higher than oil viscosity extracted by traditional extractor. This is due to increase oil density at using developed extractor. Oil viscosity extracted by developed and traditional extractor was 0.011029 and 0.00947 pa.sec. respectively. Refractive index of oil extracted by developed and traditional extractor were 1.53 and 1.45 respectively as showed in table 1. There is no significant differences between developed and traditional extractor in energy consumption per ml.

Table.1. physical properties of clove oil extracted by developed and traditional method.

Extractor type	Energy consumption per ml. kW.h/ml.	Reflective index	Oil viscosity Pa sec.	Oil density g/cm^3	Oil yield)% (
developed	a0.1875	^{a}1.5324	^{a}0.1029	^{a}1.0896	^{a}13.5
traditional	^{a}0.1904	^{b}1.4509	^{b}0.0947	^{b}1.0012	^{b}11

Each number had represented mean of three replicates. The different symbols refer to a significant

Inactivation diameter for microorganisms

It can be seen from table 2 that the inactivation diameter for microorganisms by clove oil extracted using developed extractor was significantly higher than traditional extractor for all microorganisms except *E.Coli.,* where the differences between them not significantly. These results refer to the quality of clove oil extracted by developed extractor is better than traditional extractor.

Table 2. Inactivation diameter (mm) of bacteria by using clove oil

Bacteria type	Extraction method	
	Traditional	Developed
E.Coli	[a]40	[a]38
Salmonella	[b]30	[a]34
Staph. Aureus	[b]30	[a]40
Bacillus	[b]40	[a]48
Pesudomonas	[b]25	[a]35

Each number hadrepresented mean of three replicates.
The different symbols refer to a significant effect.

Ratio of extracted oil

Table 3 shows that there is a large convergence between the practical and theoretical results of the extracted oil proportion q / q∞ by the developed extractor and the traditional extractor. MRPD values of the developed extractor less than the traditional extractor, in addition the fitting theoretical results with the practical using the developed extractor is better than the traditional extractor. The constants f, k_1, k_2 are showed in Table 3 are very important to predict q / q∞. f, k_1, $k_2$which calculated by using solver in excel 2013.

Table 1. Theoretical and practical essential oil ratio extracted by developed and traditional extractors, and constants of equations (3, 4 and 5) with statistical parameters.

Extractor type	Equation type	q/q∅			constants		
		Practical	Theoretical	MRPD	f	k_1	k_2
Developed	1	0.84375	0.8437499	6.75E-08	0.268468	0.951563	0.549283
	2	0.84375	0.8437499	7.74E-07	0.278131	0.81281	0.510129
	3	0.84375	0.8437497	9.02E-06	0.177246	0.81281	0.618766
Traditional	1	0.6875	0.6874999	1.21E-06	0.262343	0.949356	0.376125
	2	0.6875	0.6874999	7.12E-07	0.25552	0.81281	0.347233
	3	0.6875	0.6874999	1.28E-06	0.177246	0.81281	0.46526

Separation of chemical compounds using GC-MS technique

Table 4 and figure 6 illustrate the chemical compounds in the clove oil extracted by using developed extractor. The important chemical compounds in this oil are Eugenol (,72.69%), 3-Allyl-6-methoxyphenyl acetate (18.83%), Caryophyllene (3.10%). The oxygenate compounds (95.62%) are more than other compounds in clove oil extracted by using developed extractor, while ratio of terpene, nitrogen and phosphor compounds reached 4.06, 0.07 and 0.25% respectively.

It can be seen from table 4 and figure 7 that the important chemical compounds in oil extracted by traditional extractor are Eugenol,(78.1%), 3-Allyl-6-methoxyphenyl acetate(14.85%), Caryophyllene (1.89%), while ratio of oxygenate, terpene, nitrogen compounds reached 97.52, 2.37 and 0.25% respectively. some compounds did not appear in the table 4 because they decomposed due to rising temperature of cloveoil. On the other hand, the method of extraction has a significant effect on the quality of oil.

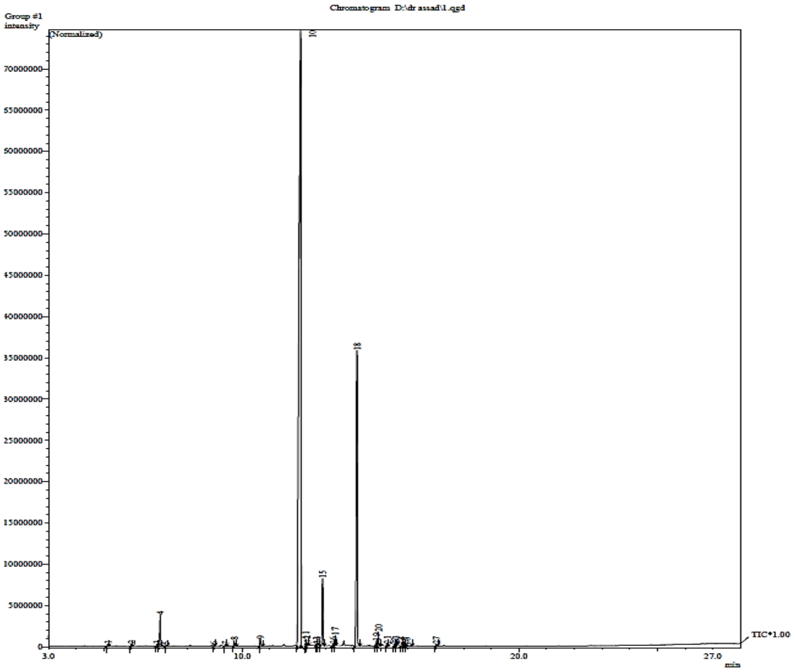


Figure 6. Separation of chemical compounds using GC-MS of clove oil extracted by developed extractor.

Table 4 of chemical compounds using GC-MS of clove oil extracted by developed extractor

Peak	Name	R. Time	Area %	Formula	M.W
1	1R-.alpha.-Pinene	5.138	0.12	C10H16	136
2	Bicyclo[3.1.1]heptane, 6,6-dimethyl-2-meth	5.999	0.09	C10H16	136
3	Benzene, 1-methyl-3-(1-methylethyl)-	6.915	0.09	C10H14	134
4	Eucalyptol	7.037	1.64	C10H18O	154
5	Acetic acid, sec-octyl ester	7.243	0.06	C10H20O2	172
6	Bicyclo[2.2.1]heptan-2-one, 1,7,7-trimethyl-	8.985	0.07	:C10H16O	152
7	Cyclohexanone, 4-(benzoyloxy)-, oxime	9.388	0.07	C13H15NO3	233
8	Methyl salicylate	9.713	0.27	C8H8O3	152
9	Phenol, 4-(2-propenyl)-, acetate	10.652	0.42	C11H12O2	176
10	Eugenol	12.114	72.69	C10H12O2	164
11	Palladium(0), bis(.eta.-2-butadiene) 1,1,4,5,8	12.293	0.25	C36H74P4Pd2	842
12	Copaene	12.342	0.17	C15H24	204
13	Benzene, 1,2-dimethoxy-4-(2-propenyl)-	12.675	0.07	C11H14O2	178
14	Phenol, 2-methoxy-4-(1-propenyl)-, (E)-	12.723	0.14	C10H12O2	164
15	Caryophyllene	12.906	3.10	C15H24	204
16	Phenol, 2-methoxy-4-(1-propenyl)-, (E)-	13.283	0.11	C10H12O2	164
17	1,4,7,-Cycloundecatriene, 1,5,9,9-tetramethy	13.359	0.49	C15H24	204
18	3-Allyl-6-methoxyphenyl acetate	14.149	18.43	C12H14O3	206
19	1H-Cycloprop[e]azulen-7-ol, decahydro-1,1	14.853	0.25	C15H24O	220
20	Caryophyllene oxide	14.917	0.70	C15H24O	220
21	12-Oxabicyclo[9.1.0]dodeca-3,7-diene, 1,5,	15.241	0.11	C15H24O	220
22	Cubenol	15.508	0.08	C15H26O	222
23	Tetracyclo[6.3.2.0(2,5).0(1,8)]tridecan-9-ol,	15.558	0.17	C15H24O	220
24	Cubenol	15.765	0.09	C15H26O	222
25	Ar-tumerone	15.862	0.08	C15H20O	216
26	Kauran-18-al, 17-(acetyloxy)-, (4.beta.)-	15.940	0.14	C22H34O3	346
27	Benzyl Benzoate	17.017	0.09	C14H12O2	212
			100.00		

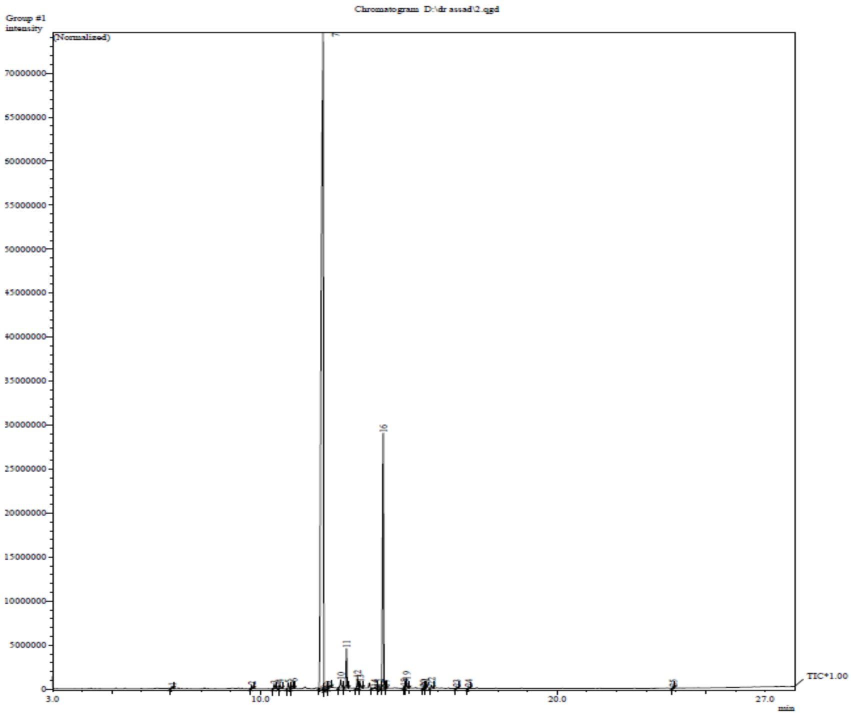


Figure 7. Separation of chemical compounds using GC-MS of clove oil extracted by traditional extractor.) Al-Hilphy,2015)

Table 5 of chemical compounds using GC-MS of clove oil extracted by traditional extractor

Peak	Name	R. Time	Area %	Formula	M.W
1	Eucalyptol	7.034	0.15	C10H18O	154
2	------------------------------------	9.711	0.16	------	------
3	Benzaldehyde, 4-(1-methylethyl)-	10.472	0.30	C10H12O	148
4	Phenol, 4-(2-propenyl)-, acetate	10.648	0.33	C11H12O2	176
5	2-Propenal, 3-phenyl-	10.941	0.30	C9H8O	132
6	Benzene, 1-methoxy-4-(1-propenyl)-	11.110	0.27	C10H12O	148
7	Eugenol	12.114	78.71	C10H12O2	164
8	Dimethyl 2,7,12,18-tetramethyl-3,8-di(2,2-d	12.158	0.11	C46H54N4O8	790
9	------------------------------------	12.295	0.25	------	------
10	Phenol, 2-methoxy-4-(1-propenyl)-, (E)-	12.710	0.55	C10H12O2	164
11	Caryophyllene	12.903	1.89	C15H24	204
12	Phenol, 2-methoxy-4-(1-propenyl)-, (E)-	13.273	0.59	C10H12O2	164
13	1,4,7,-Cycloundecatriene, 1,5,9,9-tetramethy	13.357	0.31	C15H24	204
14	1,3-Cyclohexadiene, 5-(1,5-dimethyl-4-hexe	13.850	0.05	C15H24	204
15	1,3,6,10-Dodecatetraene, 3,7,11-trimethyl-,	13.967	0.03	C15H24	204
16	3-Allyl-6-methoxyphenyl acetate	14.138	14.85	C12H14O3	206
17	Cyclohexene, 3-(1,5-dimethyl-4-hexenyl)-6-	14.217	0.04	C15H24	204
18	1H-Cycloprop[e]azulen-7-ol, decahydro-1,1	14.851	0.07	C15H24O	220
19	Caryophyllene oxide	14.915	0.49	C15H24O	220
20	2-Naphthalenemethanol, 1,2,3,4,4a,5,6,7-oc	15.500	0.07	C15H26O	222
21	Tetracyclo[6.3.2.0(2,5).0(1,8)]tridecan-9-ol,	15.550	0.12	C15H24O	220
22	2-Naphthalenemethanol, decahydro-.alpha.	15.757	0.17	C15H26O	222
23	Spiro[5.5]undec-2-ene, 3,7,7-trimethyl-11-m	16.617	0.05	C15H24	204
24	Benzyl Benzoate	17.018	0.07	C14H12O2	212
25	1,2-Benzenedicarboxylic acid, diisooctylest	23.900	0.05	C24H38O4	390
		100.00			

IV. Conclusions

The developed steam extractor can be used to essential oil extraction, moreover oil yield by using developed extractor was higher compare with traditional method. Extraction temperature was lesser than 85 ^{o}C at using developed extractor. No heat decomposition in the oil extracted by developed extractor contrary to traditional extractor that produced oil decomposition. On the other hand, the physical properties of clove oil extracted by developed extractor was best compare with traditional extractor.

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