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The Distillation of Thymus Vulgaris Essence to Demonstrate TBP Curve

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Abstract
Based on a research project on Shirazi Thymus of the Eksir Gol Sorkh company, the essence of this Thymus included five main components, which based on the results gained from GC device, there are 5 main components in this Thymus namely α-Pinene, α-Terpinene, P-Cymen, γ-Terpinen, and Thymol, each of which has a percentage as follows 1.118, 3.831, 25.815, 14.771, and 54.465. With increasing the amount of Distillation, more Thymol can be achieved, and in this experiment, the amount of Thymol increases 18 percent to 92 percent. With passing of 81min from the 170 degrees Celsius, the temperature of essence solution, which is a stable 250 degrees Celsius in Heating (Oil/sand) bath, reaches 240 degrees Celsius. With an increase in the volume of Distillation material, the output water of condenser reaches up to 22.7 degrees Celsius from 21.9 degrees Celsius in such a way that incoming cold-water temperature has increased up to 21.9 degrees Celsius from 21.4 degrees Celsius by passing of time. In TBP diagram, with an increase in Distillation volume from 2 percent to 72 percent, the boiling temperature of Thymus essence solution has reached 240 degrees Celsius from 160 degrees Celsius.

KEYWORDS: Distillation; Thymus; TBP; GC

1. Introduction

Thymus is an aromatic plant from Lamiaceae family, and considered one of the popular plants in traditional and western medicine [1]. There are 215 types of Thymus in the world and 14 types of have been reported to exist in Iran [2]. Traditionally, this plant is used to treat anti-tympanites, improve digestion, remove spasm, coughing, and extra nasal mucus, and because of having Thymol, it is used in food, pharmaceutical, sanitary, cosmetic industry [3]. The water, water-alcoholic, and propylene glycol essences of this plant are used in making shampoos, moisturizers, and ointments [4]. Anti-molding and anti-bacterial effects of this plant along with therapeutic effects of this plant are used to treat asthma, multiple coughing, and bronchitis [5]. Different types of medical pills, syrups, tablets, etc. of this plant have been created under the approval of the organization of sanitation, treatment, and medical education of the country. Thymol is the main component in Thymus and its amount differs from 40 percent of mole in mountain thymus to more than 70 percent of mole in Shirazi type [6]. According to the results, as Figure1 revealed, lab analyses produced by Science and Technology Park of Khorasan Razavi, the essence of Shirazi Thymus of the Eksir Gol Sorkh Company included 54.46 percent Thymol, contained Para Cymene with 25.81 percent and Gama Terpinene with 14.77 percent mostly in Table1.

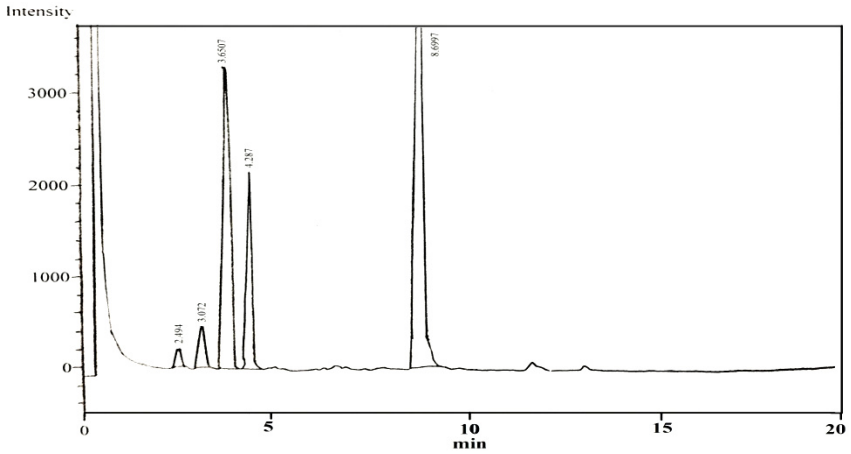

Figure1. Type and components of Shirazi Thymus essence using GC device

Table1. Essence components

Number	Component	RI	Percent	Number	Component	RI	Percent
1	Tricyclene	925	0.01	13	Cis-Sabinene Hydrate	1074	0.05
2	α-Pinene	928	1.118	14	Terpinolene	1088	0.05
3	α-Tujene	931	0.02	15	Trans-Sabinene Hydrate	1097	0.07
4	Camphene	945	0.03	16	Borneol	1164	0.09
5	Myrcene	992	0.03	17	Terpinene-4-Ol	1188	0.1
6	α-phlladrene	1005	0.04	18	Carvacrol/Methyl Ether	1246	0.1
7	α-Terpinene	1016	3.831	19	Thymol	1297	54.265
8	P-Cymene	1027	25.715	20	β-Caryophylene	1416	0.2
9	Limonene	1032	0.07	21	Germacrene B	1487	0.2
10	Carvacrol	1035	0. 09	22	α-Murolene	1499	0.1
11	1,8-Cineole	1043	0.03	23	β-Bisabolene	1509	0.3
12	γ-Terpinene	1062	14.571	24	δ-Cadinene	1525	0.01
				Total			100

Table2. Main Thymus essence components

Number	Time Min	Area	Height	Mole Fraction	Component

1	2.494	1475	212	1.118	α-Pinene
2	3. 072	5057	494	3.831	α-Terpinene
3	3.650	34074	3349	25.815	P-Cymene
4	4.287	19497	2205	14.771	γ-Terpinene
5	8.699	71891	6520	54.465	Thymol
Total		131994	12780		

According to the analyses done by GC/Mass, as Table2 revealed, Shirazi thymus essence is comprised of 5 main components namely, α-Pinene, α-Terpinene, P-Cymene, γ-Terpinene, and Thymol, each of which has the percentage of 1.118, 3.831, 25.815, 14.771, and 54.465.

2. Distillation

When a liquid is trapped in a closed container, liquid molecules will steam of the surface of the liquid and then return into the liquid, and balance is brought about by this. In other words, the rate of molecules travelling away from the liquid is the same as the rate of molecules travelling back to the liquid. The gaseous molecules of the liquid apply some pressure, which is called liquid steam pressure (liquid balance steam pressure). Liquid steam pressure depends on its temperature and its amount is stable in any given temperature. If the liquid is heated up so that its steam pressure surpasses that of the atmosphere, the liquid begins to boil. The boiling point of a liquid is reached when its steam pressure reaches the standard amount (one atmosphere). If the atmospheric pressure reduces, the temperature needed to increase liquid steam pressure up to the atmospheric pressure, is also reduced. The boiling point of many liquids reduced by 0.5 degrees when 10ml reduction (around 760mm of mercury). Therefore, the boiling point works as an agent in determining the Distillation pressure. Distillation of pure liquid: Distillation includes heating a liquid up to its boiling point and then passing it through a device called Condenser, and then collecting the liquid. If a pure liquid is condensed and the temperature curve is drawn based on the amount of Distillation, which Figure2 showed, the result will be a straight line.

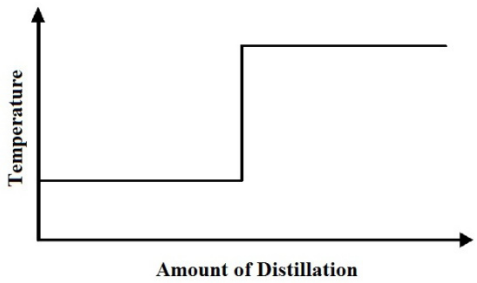

Figure2. TBP diagram of pure materials

3. Distillation of Solutions

In a mixture of two or more items, the total steam pressure is based on the steam pressure of each of the components and their mole fraction. However, the only exception in this case is azeotrope of this mixture. Based on Raoult's law**,** the minor steam pressure of a highly evaporating mixture in an ideal solution equals the product of steam pressure and mole fraction. The connector of these items with the total steam pressure P and minor steam pressure Pn and Mole fraction Nm is defined as follows for each item:

$$P = Pa.Na + Pb.Nb + Pc.Nc + \ldots \quad (1)$$

Boiling takes place when P=Patm. The Distillation curve for separating chemical components with a close boiling point in a Distillation device is as follows:

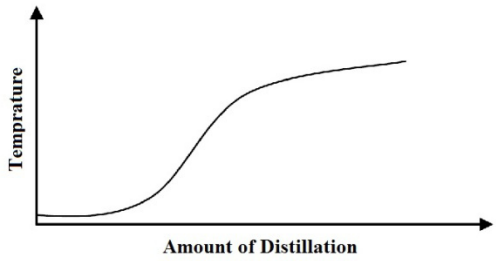

Figure3. TBP diagram of mixtures

If the temperature of the liquid is highly increase so that it is close to the atmospheric liquid pressure (boiling point) and remains in this temperature, the whole liquid will evaporate. This is called Distillation. Now, if all of the evaporated liquid is condensed on the other side after cooling, all of it becomes liquid again. In chemists' opinion, when there is Distillation, there are two main phenomenon including boiling and then cooling. Every pure natural item can be condensed in a fixed temperature which is called the boiling point. In contrast, if that item has a high boiling point, it will indicate some impurities in that item. Not to mention that all the liquids which have a low boiling point, are a hundred percent pure [7].

4. Simple Distillation

As Figure4 showed, a simple condensing device is comprised of a flask which is connected to condenser with 3 tubes. The thermometer is installed in such a way that its mercury reservoir is 10mm lowers than the output and is in touch with the evaporated liquid. First of all, the joints should be greased using silicon oil. In order to collected the pure condensed liquid an Erlenmeyer flask and a scaled cylinder are necessary. In order to use the condenser, the device must be in touch with the free air to stop the risk of increased pressure while heating. A flame is used as a heater since the required temperature is more than 200 degrees Celsius. Heating (Oil/sand) bath is placed on top of a tripod and a metal net.

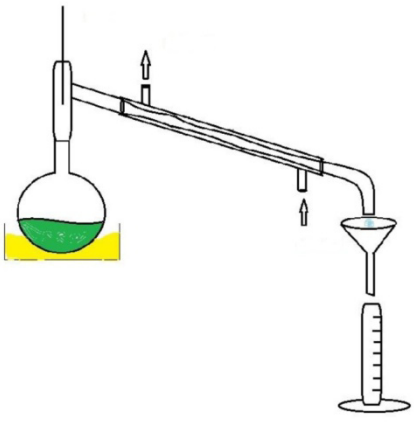

Figure4. Distillation device schematic

5. How to Run the Experiment

A round flask is placed on top of a flame on a in heating bath of Paraffin wax in order to heat all the flask evenly. A T-joint is added to its entrance and 2 thermometers, showing the scale of 0 to a 400 degrees Celsius are installed to two other ends of the flask. The condenser is attached to the other end of the T-joint and we block the other end of the T-joint in order not to allow the steam to escape. A scaled cylinder must be placed under the

condenser in order to collect the product of distillation. In joints, silicon oil must be applied to stop any leakage. Incoming water must be attached to the lower part of the condenser and the outgoing water must be installed to the top of the condenser in order to fill the condenser completely. As a result, the more we travel through the condenser, its temperature decreases and steams cool off. Before the start of the experiment, in order to prevent ultra-boiling phenomenon and to prevent foaming, some boiling stone must be placed in the flask. 35cc of thymus must be placed in the flask after installing the joints. Boiling must be awaited after the ignition of the flame. When the first drop appears in the cylinder, the displayed temperature is the boiling point of the liquid.
160°C = the degree read on the thermometer
The temperature must increase in order to allow all other components escape the steam container, and they should turn into liquid in the condenser. Collecting samples of the liquid must be done at a certain temperature and time after each 5ml is condensed in order to realize the materials that are present. The experiment must be carried out until the essence changes color.

Table3. The data of the experiment

Rate of distillation %	Volume of distillation CC	Tc in °C	Tc Out °C	Vapour Temperature °C	Vapour Liquid °C	Time Min	Observation
0	0	21.3	21.3	22	22	0	1
0.028	1	21.4	21.9	160	170	18.28	
0.142	5	21.7	22.5	199	202	31.29	
0.285	10	21.8	22.5	208	214	36.24	
0.428	15	21.8	22.5	220	224	44.17	2
0.571	20	21.9	22.6	232	235	75.41	3,4
0.714	25	21.9	22.7	240	240	81.45	5

6. The Experiment Error

1. The most important error in this experiment is the difference with the sea level, which results in a difference in pressure. We know that the boiling point of a liquid is the temperature at which the steam pressure of the liquid is the same as the atmospheric pressure. With regard to the fact that in the laboratory the air pressure is less than the sea level pressure (635 ml of mercury), the boiling point of the liquid is less than that of the sea level one.
2. Accidental errors such as clotting of the liquid in the condenser and its sudden release
3. The angle between the condenser and the distillation column affects the results of the experiment, and it is better to keep it at 45 degrees.
4. The error of reading the temperature from a mercury thermometer

7. Observations

1. The first bubble appeared at 136 degrees Celsius
2. After 56min, the flame was boosted in order to achieve more heat since the liquid temperature stays steady at 230 degrees Celsius
3. Gradually, the color of the distillation product changes to light yellow from the transparent mode. The amount of froth produced from the essence is fixed.
4. The color of the essence of the boiling pot changes to black, which indicates the decomposition of natural compounds, and this is when we stop the experiment.

8. The result of the experiment

Table4. The results of collecting samples and determining the amounts of compounds in condensed sample with GC device

Retention Time	RT=2.494	RT=3.072	RT=4.01	RT=4.633	RT=8.69
No#	1	2	3	4	5
Name	α-Pinene	α-Terpinene	P-cymene	γ-Terpinene	Thymol
Thyme Essential Oil	1.118	3.831	25.815	14.771	54.46
sample0	4.918	8.884	43.334	24.421	18.444
sample1	1.814	5.478	40.391	24	27.643
sample2	0	0	24.662	15.567	59.771
Sample3	0	0	4.876	2.904	92.22

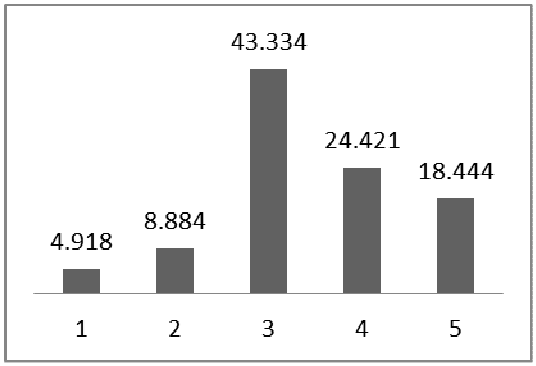

Figure5. The diagram drawn based on the results of sample 1 using GC device

According to Figure5, in 1cc volume of distillation, the most available material is P-cymene.

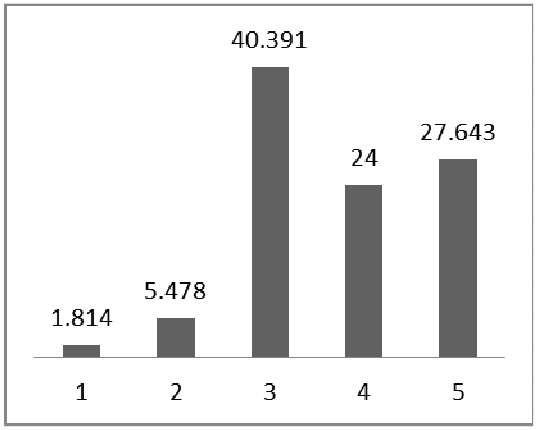

Figure6. The diagram from the results of sample2 using GC device

As it is clear in Figure6, compared to the previous image, P-cymene and Thymol in 28 percent of distillation volume are increasing.

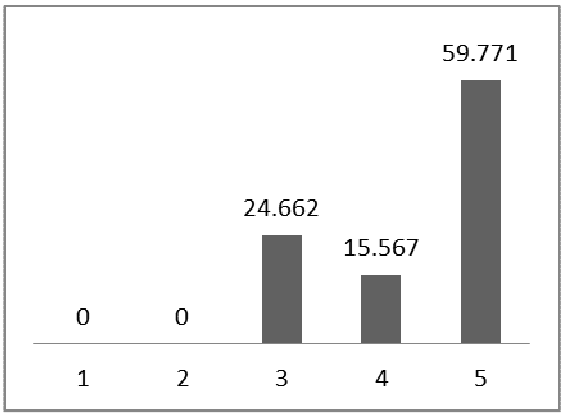

Figure7. The diagram from the results of sample3 using GC device

In Figure7, as predicted, Thymol will increase in 57 percent of distillation volume and other items will decrease.

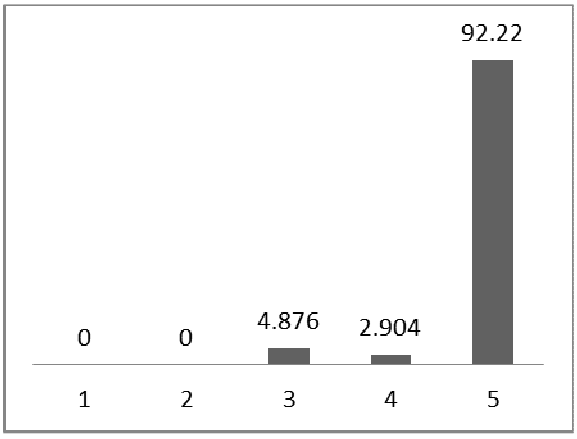

Figure8. The diagram drawn based on the results of sample 4 using GC device

In Figure8, as it is clear, Thymol is at its highest in 71 percent of distillation volume.

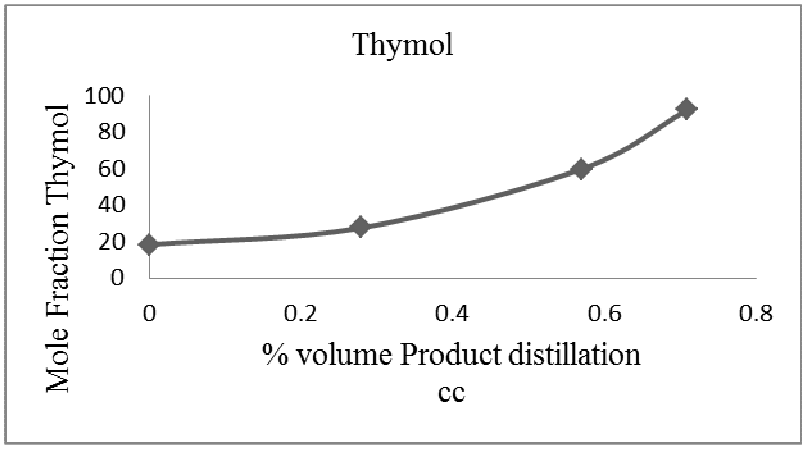

Figure9. Thymol amount changes according to distillation volume increase

As it is clear in Figure9, with an increase in amount of distillation, we can get more Thymol as it is clear in the experiment that the amount of Thymol is on an increase from 18 percent to 92 percent.

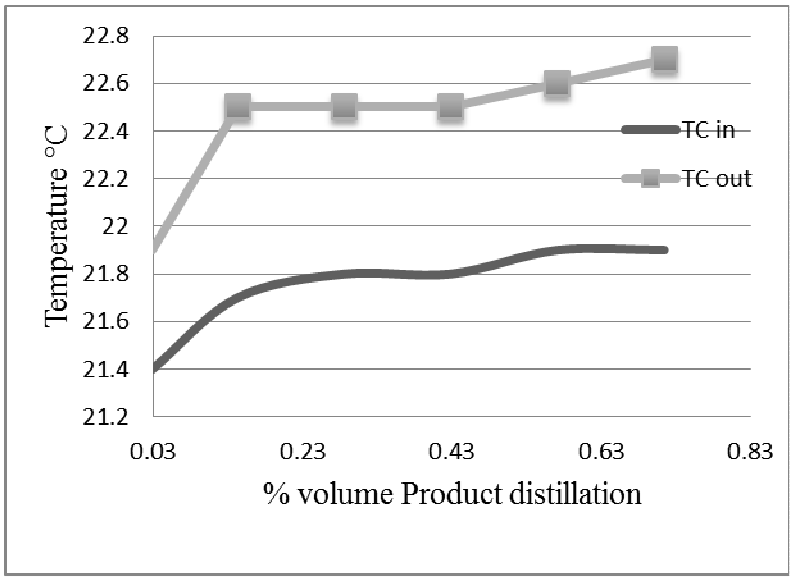

Figure10. Condenser temperature changes along with an increase in distillation volume

In Figure10, by increasing the amount of distillation product, the temperature of condenser water outflow will increase from 21.9 degrees Celsius to 22.7 degrees Celsius, and the temperature of incoming cold water will not be stable and will increase in such a way that after a while, incoming cold-water temperature has increase from 21.4 degrees Celsius to 21.9 degrees Celsius.

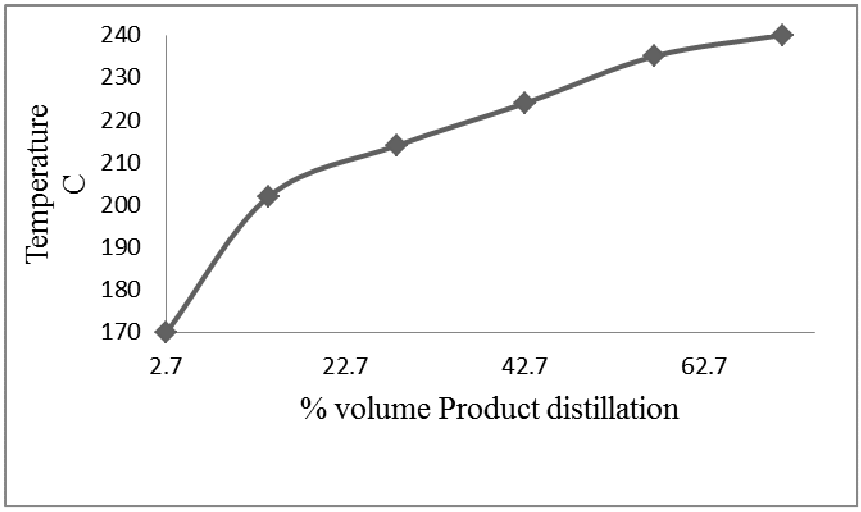

Figure11. TBP curve based on Thymus Vulgaris essence

In Figure11, as it is clear, with an increase in distillation amount from 3 percent to 72 percent, the boiling point of Thymus essence solution is on an increase from 160 degrees Celsius to 240 degrees Celsius.

9. Discussion and Conclusion

1. The first bubble was noticed at 136 degrees Celsius.
2. After 56 min, the flame was powered in order to increase the heat since the liquid temperature is stable at 230 degrees Celsius.
3. Gradually, the color of distillation product will change from no color to light yellow color.
4. The amount of froth produced by essence is stable.

5. The color of the boiling essence in the container will almost turn into black, which is an indicator of decomposition of natural compounds, and this is when we will stop the experiment.

10. References

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