Extraction of Essential Oil from Rose (*Malaysian Roses*)

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***Abstract*—The study was conducted to determine the present of essential oil in different mass quantity of roses by using hydrodistillation and steam distillation. In addition, the study reveals the characteristic of volatile component and its composition in the rose essential oil. The samples used in this study were the mixture of rose species from the Cameron Highlands. The petals were allowed to dry between 6-7 days. The moisture content of rose calculated was 77.7%. The masses quantities of petals manipulated for extraction were 30g, 40g and 50g. The extraction time was kept constant at 6 hours. The percentage by mass of essential oil yield from hydrodistillation was ranging from 0.09-0.1% while steam distillation ranging from 0.024-0.097%. The obtained oils were analyzed by using GC-MS. From the analysis result, *9-Nonadecene, á-Pinene, Eicosane* and *Heneicosane* were the major components of the rose oil. The study indicated that the hydrodistillation is more suitable for extracting the essential oils compare to the steam distillation. Hydrodistillation gave high amount of components compare to steam distillation. The main component of rose oil which is Citronellol was not found in this study. However, the others volatile component can be used in cosmetic industries instead of perfume industries.**

***Keywords*— rose, Cameron Highlands, essential oil, hydrodistillation, steam distillation.**

1. INTRODUCTION

Rose flowers (Rosa) are domestic flower that become common flower in our society, for its fragrance, but also for rose oil and rose water, and for cosmetic and medical purposes (Gustavsson 1998). Rose essential oil has a variety of practical used (Naryanand & Kumar, 2003) in the industries for the scenting and flavouring purpose. Rose oil is used essentially as an aroma ingredient in pharmaceutical (e.g. treatments and moisturizers), and is extensively used as a scent component in fragrances, creams, and cleansers. The scent of the rose is trapped by using different kind of technique.

There are a lot of method can be used to extract the essential oil from rose. Normally the methods used are traditional methods which are hydrodistillation and steam distillation. In addition of this method, solvent extraction by using Soxhlet apparatus can be used (Memarzadeh, Ghasemi Pirbalouti & AdibNejad, 2015). Nowadays with the advance technologies, another method of extraction is created in order to increase the percentage yield of the oil and also the purity of the volatile components (CO_2 extraction).

From the previous research (Younis, Khan, Khan, Riaz & Perves, 2007), four species of rosa (R. damascena, R. centifolia, R. borboniana and Rosa 'Gruss an Teplitz') have different oil content ranging from 0.03% to 0.1%. The methods used in the extraction were steam distillation and solvent extraction. By using the hydrodistillation method, essential oil extracted from Rosa damascenea Mill of South of Iran yield about 0.16% (Moein, Ghasemi, Karami & Tavallali, 2010).

Gas chromatographic analysis (Sood et al., 1994) is most advanced, ideally technique for separation of different volatile components by the perfumer. By adding the mass spectrometry analysis, GC-MS is being used. GC-MS is a hybrid analytical method that combined the separation abilities of GC with the recognition properties of MS to give a higher efficiency of sample analyses. While GC can isolate volatile components of the sample, MS can differentiate the volatile components based on the basis of their mass.

The previous study stated that more than 95 macro and micro component found in the rose oil (Loghmani-Khouzani, Sabzi Fini & Safari, 2007). The main components were *citronellol, nonadecane, geraniol and heneicosane* through the steam distillation method. Other research indicated that *Nonadecane, heneicosane, docosane, citronellol* and *9-nonadecene* were obtain through the hydrodistillation method (Moein, Ghasemi, Karami & Tavallali, 2010).

The importance of the research is to identify whether Cameron Highlands rose can be commercialized through its essential oil based on the composition of the oil. Most of the journal about the extraction of rose essential oil came from outside of Malaysia such as India, Pakistan and Iran which used Rosa damascenea Mill which is famous for commercially harvest for the rose oil.

This study is conducted to show how different quantity of mass affects the yield of essential oil by using hydrodistillation and steam distillation. Besides that, the volatile components of rose oil were determined in this study.

2. METHODOLOGY

A. Materials

The Rose flower was acquired from Cameron Highlands as the sample. Unwanted material such as leaves, buds and stem were removed, only petals were taken. The rose petals were dried by using natural method with conditions 1 atm and 25°C. The mass of the petals were weighted before the drying process take placed. The petals were weighted daily until the mass of the petals became constant. The percentage of moisture content was calculated by using equation below:

$$\%\ moisture\ content = \frac{initial\ mass - final\ mass}{initial\ mass}\ x\ 100\% \quad (1)$$

Figure 2-1 shows the fresh petals that were obtained from Cameron Highlands. The petals were allowed to dry between 6-7 days and the result was shown in Figure 2-2.

Figure 2-1: petals before dried

Figure 2-2: petals after dried

B. Hydrodistillation

30g of weighted petals was place into the Clevenger apparatus with 1L of water. The temperature constant was set 200°C along the experiment. The distillation time was 6 hours. The experiment was repeated with 40g and 50g of petals.

C. Steam Distillation

30g of weighted petals was place into the flask at the center of Clevenger apparatus. The steam was supplied by boiling the water and the steam was let to flow through the petals. The distillation time was 6 hours. The experiment was repeated with 40g and 50g of petals.

D. Analysis

The analysis of composition in rose essential oil will be conducted in the Varian 450-GC combined with Varian 240-MS. equipped with a cross-linked 5% pH ME siloxane HP-5MS capillary column (30 mm x 0.25mm in diameter, film thickness, 0.25 µm). Oven temperature used is at 60°C for 3 min and then programmed to increase gradually to 220°C at a rate of 6°C/min. The injector and detector (FID) temperatures are 290°C and the carrier gas is helium with a flow of 1 mL/min. The volume injected is 0.1 µL of the oil and the split ratio is 1:50 (Khouzani, Fini & Safari, 2007). The compounds are then being identified by comparison of their mass spectra with the GC-MS library.

In Figure 2-3, the methodology was summarized with the apparatus and equipment diagram.

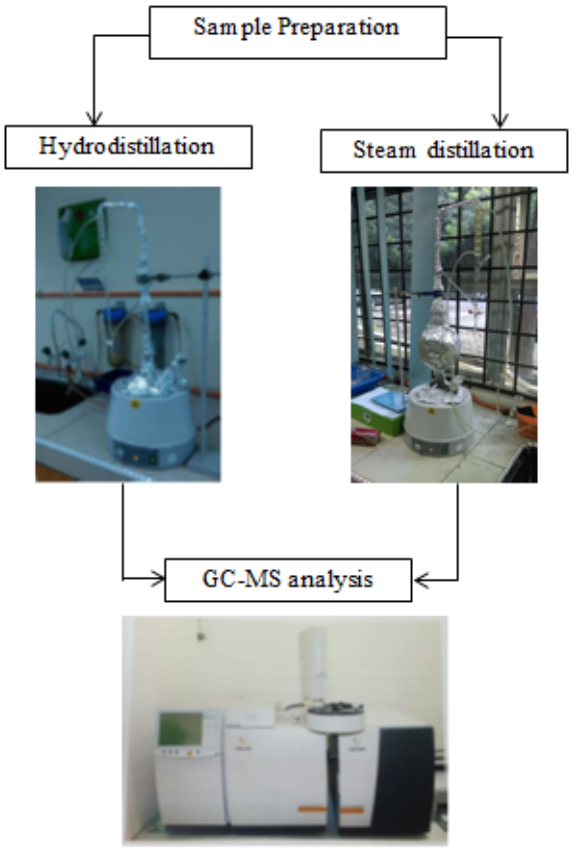

Figure 2-3: Flow Chart of the Experiment

3. RESULTS AND DISCUSSION

A. Moisture content of rose

The initial mass of petals weighted was 2000±1g. After 6-7 days when the mass of petals was constant, the final mass of petals was 446±1g. The moisture content of rose petals was 77.7%.

B. The effect of different mass of sample on the essential oil yield with different type of methods.

The mass of essential oil yield from hydrodistillation and steam distillation were recorded. The mass of essential oils yield are recorded in the Figure 3-1 and Figure 3-2.

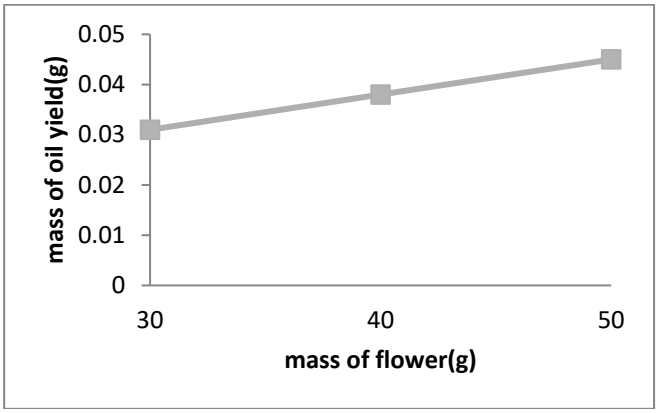

Figure 3-1: Graph of hydrodistillation mass of oil yield against mass of flower

For the hydrodistillation, the mass of essential oil yield seems increase with the mass of petals/flower. This mean the more amount of petals used, high amount of essential oils will be

recovered. From the Figure 3-1, the percentage mass of essential oil by mass was calculated. 30g and 40g of petals yield 0.1% while 50g yield 0.09% of oil.

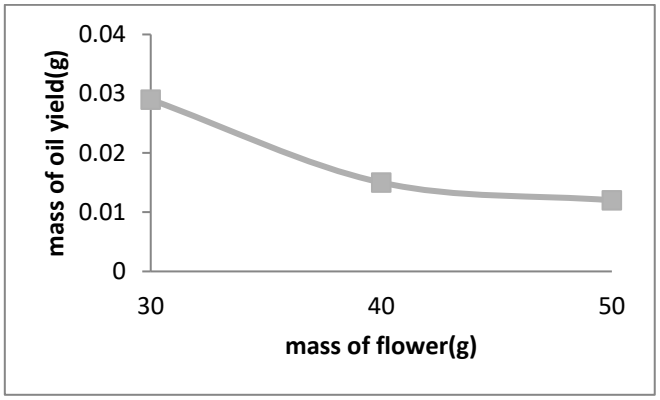

Figure 3-2: Graph of Steam Distillation mass of oil yield against mass of flower

From the calculation based on Figure 3-2, 30g of petals yield 0.097%, 40g of petals yield 0.038% and 50g of petals yield 0.024% of oils. The result of steam distillation was contradict to the hydrodistillation. The reason why the essential oils yield of steam distillation decreasing was the voidage of the petals. Below were the samples taken from the flask after the steam distillation complete.

(a) **(b)**

Figure 3-3: (a) shown the petals that at the bottom of the flask, (b) shown the petals at the center or top of the flask

With the increased of the mass, the volume of the petals also increased. The condition of petals inside the flask was compacted and blocked the flow of the steam (Rhodes, 2008). Figure 3-3(a) shows the wet petals that blocked the path of the steam flow. Some petals that steam could not reach shown in Figure 3-3(b). Hence the mass yield of essential oils decreased by the increasing the mass of the petals.

The next graph which was Figure 3-4 had shown the comparison between the hydrodistillation and steam distillation method.

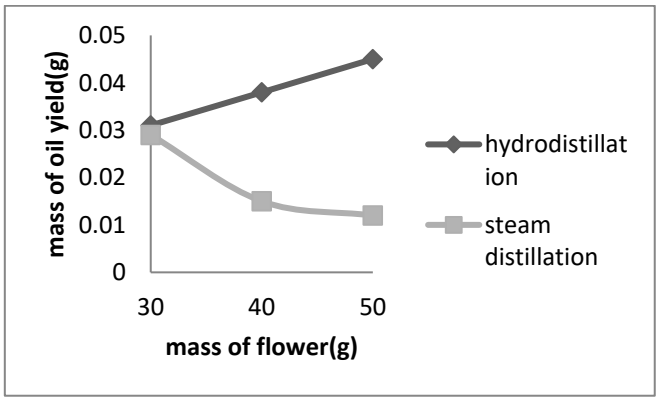

Figure 3-4: Comparison on method based on mass of oils yield

The hydrodistillation gave high yield of the essential oils compared to the steam distillation. This was due to the low voidage in the flask of steam distillation.

C. Composition of Rose Essential oil

In this study, more than 100 components were found in the essential oil of Rosa from Cameron Highlands. Figure 3-5 and Figure 3-6 shown the result obtained from the analysis by using the GC-MS. The amount of components analyzed in term of percentage was ranging from 0.0001-2.81%. Thus, the components that selected in Table 3-1 based on the amount more than 0.1% for both method of extraction. The components are listed in order of their elution from a HP-5Ms column.

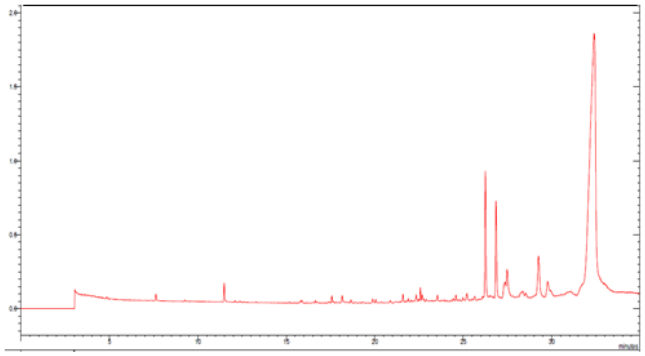

Figure 3-5: Hydrodistillation GC-MS Analysis

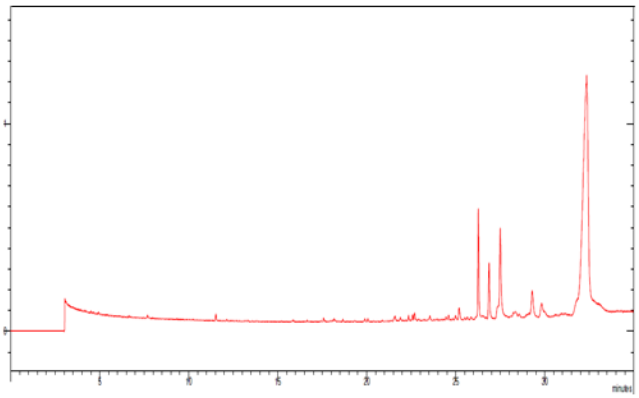

Figure 3-6: Steam Distillation GC-MS Analysis

Table 3-1: The percentage composition of the rose essential oil identify by GC-MS

No	Components	Hydro distillation	Steam Distillation
1	*Octane, 2,3,6,7-tetramethyl-*	-	0.290%
2	*á-Pinene*	0.179%	0.031%
3	*Nonadecane*	0.104%	0.005%
4	*9-Nonadecene*	1.680%	1.190%
5	*Dodecane, 2,6,11-trimethyl-*	0.152%	-
6	*Tetradecanoic acid, 10,13-dimethyl-, methyl ester*	0.394%	0.518%
7	*2-Methylundecane*	0.708%	0.001%
8	*Eicosane*	0.714%	0.294%
9	*Citronellyl propionate*	0.280%	0.120%
10	*Heneicosane*	2.810%	2.340%
11	Others	92.979%	95.211%
	Total	100.00%	100.00%

The analyzed result of hydrodistillation and steam distillation has the differences in term of amount and component present in the oil. Hydrodistillation gave slightly higher amount of component compared to steam distillation. A study proves that á-Pinene has

antibacterial properties (Wang et al, 2012). It is used in production of deodorant. *Citronellyl propionate* is a good antiperspirant which can be combined with *á-Pinene* to make deodorant product. Alkane and alkene (*Heneicosane, Eicosane, 9-Nonadecene, Nonadecane*) are used as fragrance agent in cosmetic industries.

Commercial rose oil from damask rose contains high amount of *Citronellol* component as indicated by previous study (Loghmani-Khouzani, Sabzi Fini & Safari, 2007). *Citronellol* is a sweet floral aroma of rose and owns a bitter taste. It possesses a floral rosy and fresh citrus flavor which highly contributed in perfume industries. In this study, *Citronellol* was not found in the oil. However, there are others volatile component that still can be used.

4. CONCLUSION

The objective of the research was to determine the effect of the mass quantity of petals toward the essential oil yield. It can be concluded that, for hydrodistillation method, the higher the quantity of petals, the higher the yield of essential oil. In contrast of steam distillation, the higher the mass, the lower the yield of essential oil. This problem may be overcome by increasing the size of the flask in order to increase the voidage of the petals as long the petals do not too compact in the flask. Hydrodistillation gave high amount of major components compare to steam distillation. Although *Citronellol* was not found in this rose oil, there are still other volatile components such as *á-Pinene* and *Citronellyl propionate* that can be used in cosmetic product.

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