

# Effect of Essential Oil Extraction in term of time and mass from Hibiscus *Rosa-Sinensis*

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**Abstract**— Essential oil can be extracted through many ways. It can be extracted through conventional method (hydrodistillation and steam distillation), solvent extraction, and supercritical fluid extraction (SFE). The aim of this paper is to study the effect of different operational conditions; extraction time, and extraction methods based on the yield of hibiscus obtained, and to study the compounds exist in the extracted oil. In this research, the essential oil was extracted through hydrodistillation (HD) and steam distillation (SD) method by manipulating the time of extraction; 2, 4, and 6 hours for each method. The obtained oil was then analysed by using Gas Chromatography-Mass Spectroscopy (GC-MS). Based on the result, the yield of oil is increasing when the time of extraction is extended for both HD and SD. As for the method of extraction, 6 hours of extraction by SD gave the best yield which was 0.036% compared to HD which was 0.033%. Analyses of the oil by GC-MS indicate that SD produced higher quality of chemicals than HD. Also, there were 11 major chemicals extracted by SD (*cis- $\alpha$ -Terpineol*, *Linalyl anthranilate*,  *$\alpha$ -Pinene*,  *$\beta$ -Selinene*, *Nonadecane*,  *$\alpha$ -Myrcene*, *Lavandulol*, *(E)- $\beta$ -Farnesene*, and *Isopropyl Myristate*, *Alloaromadendrene oxide-(1)*, and *Eicosane*), while 9 major chemicals extracted by HD (*cis- $\alpha$ -Terpineol*, *Linalyl anthranilate*,  *$\alpha$ -Pinene*,  *$\beta$ -Selinene*, *Nonadecane*,  *$\alpha$ -Myrcene*, *Lavandulol*, *(E)- $\beta$ -Farnesene*, and *Isopropyl Myristate*). SD is good method in extracting more chemicals, but in term of amounts, HD managed to exceed SD.

**Keywords:** *Hibiscus Rosa-Sinensis*; *Hydrodistillation*; *Steam Distillation*; *Extraction*; *Essential Oil*; *Gas Chromatography Mass Spectrometer*.

## I. INTRODUCTION

Hibiscuses that available in this world today are a hybrid from the ancestors which are scattered around the world; Madagascar, Fiji, Hawaii, Mauritius, and China (Hidden Valley Nature Arts, 2016). *Hibiscus rosa-sinensis* is one of the ancestors to the modern hibiscus. This hibiscus is commonly known as Chinese hibiscus and tropical hibiscus because it is believed to be originated from china (Sukirti & Prashant, 2011). It also can be found in several countries in Asia mostly, including Malaysia. Moreover, hibiscus *rosa-sinensis* is the national flower of Malaysia. It is a plant that is used in decoration purposes and some people grows them as a fence plant.

Essential oil has been used for centuries in such different purposes. The essences are extracted from various flowers, fruits, and plants. The term 'essential oil' was first used in the 16<sup>th</sup> century by Paracelsus van Hohenheim (Guenther, 1950). Through some researches, essential oil is proven as powerful substances with remarkable medicinal properties due to the existence of substances that are very complex in their molecular structures (Worwood, 1991). Moreover, the consumed oil does not leave any chemicals and toxics trace in the body like any other chemical drugs.

*Hibiscus rosa-sinensis* is one of glabrous shrub, widely cultivated for traditional purposes. The red flowered hibiscus is most preferred in medicinal area. Based on the previous study, the leaves and flowers are used in promoting hair growth and aiding in healing ulcers (V.M. Jadhav, R.M. Thorat, V.J. Kadam, & N.S. Sathe, 2009). In India mostly, the people there includes the extracts of various part of hibiscus *rosa-sinensis* in their herbal products which intended to treat hair problem. Meanwhile in Malaysia, the hibiscus is being used traditionally to reduce fever and expectorant by using hot water extracts of flowers and roots (V.M. Jadhav et al., 2009).

Nowadays, there are various methods to extract essential oil. These methods include hydrodistillation, steam distillation, solvent extraction, and supercritical fluid extraction. Both SD and HD are known as traditional methods to isolate the essential oil. These methods have some disadvantages which includes long extraction time and losses of volatile compound during the extraction. Despite of the disadvantages, they are known for being the simplest methods of extraction and easily available. Moreover, there is no study has been made regarding the comparison of these methods (SD and HD) in extracting essential oil from hibiscus *rosa-sinensis*.

Based on previous study in extracting essential oil, the longer the extraction time, the higher the yield of extracted essential oil (Yahya & Yunus, 2013). Meanwhile, steam distillation is said to be more efficient than hydrodistillation method (Gavahian, Farhoosh, Farahnaky, Javidnia, & Shahidi, 2015). However, no research has been conducted on these conditions towards extraction of hibiscus *rosa-sinensis* and effects of these parameters on the chemical composition of extracted hibiscus *rosa-sinensis* oil. Thus, the study embarks on the three objectives; to study best extraction time of essential oil from hibiscus *rosa-sinensis*, to study best extraction method between hydrodistillation and steam distillation, and to analyse the chemicals of the oil.

## II. METHODOLOGY

### A. Materials

Hibiscus *rosa-sinensis* flowers were collected in Kota Bharu, Kelantan. The petals of the flower were detached and naturally dried for a week. 30 gram of dried samples was used for every run of the extraction process.

### B. Moisture Content Analysis

The detached fresh petals were weighted and considered as weight of sample. After it was completely dried, the sample is weighted again. The moisture content of the sample was calculated by using both obtained data. The equation used is as follows;

$$\text{Moisture Content} = \frac{(\text{Mass of Sample} - \text{Mass of Dry Sample})}{\text{Mass of Sample}} \times 100\% \quad (1)$$

### C. Oil Extraction

#### Steam distillation (SD)

SD was performed by using heater as the heating source. In SD procedure, 30 g of dried petals of hibiscus *rosa-sinensis* was placed in the junction of flask and Clevenger type. 1 L of distilled water was heated in the apparatus flask for 2, 4 and 6 hours from initial temperature of 27°C. The distilled water was added every 1 hour as during the steam distillation, the water did not recycle back into the flask.

The extracted essential oil was collected and being kept in vials and stored in dark cupboard.

#### Hydrodistillation (HD)

HD is another method of extraction which is used in this study. Overall, HD is conducted in a similar way as SD. The volume and size of utilized container were exactly the same as that used for SD. Except, in HD the 30 g dried petals was being immersed in the distilled water inside the flask. It was then being heated using the same heating source. The extraction was done for 2, 4 and 6 hours. The storage of the essential oil was same as in SD.

### D. Percentage of oil yield

The empty vial was weighted and recorded before being used to keep the essential oil. After the oil was collected together with hexane, the vial was then being evaporated in the fume hood in order to obtain the essential oil. When the hexane was fully evaporated, the vial containing oil was then weighted again.

From the weight of oil obtained, the percentage of oil yielded is then being calculated.

$$\text{Oil Yield (\%)} = \frac{\text{Mass of Oil Obtained}}{\text{Mass of dried petals used as sample}} \times 100\% \quad (2)$$

### E. Gas chromatograph-Mass spectrometry (GC-MS)

GC-MS analysis of the obtained essential oil was being performed by using the Varian 45-GC and Varian 240-MS, operating at 70 eV ionization energy and equipped with hp-5ms column (30m x 0.25 mm, 0.25 µm film thickness). The oven temperature was set from 70°C and gradually increased

to 240°C at a rate of 5°C per minutes. Helium was used as carrier gas at the flow of 1mL/min and split ratio of 1:50. The ion source was set at 240°C. As for scanning range, ranges of 35 to 425 amu were used. The compounds were then being identified by comparison of their mass spectra with the GC-MS library.

Figure 1 shows the flow diagram of the experimental conducted in the study.

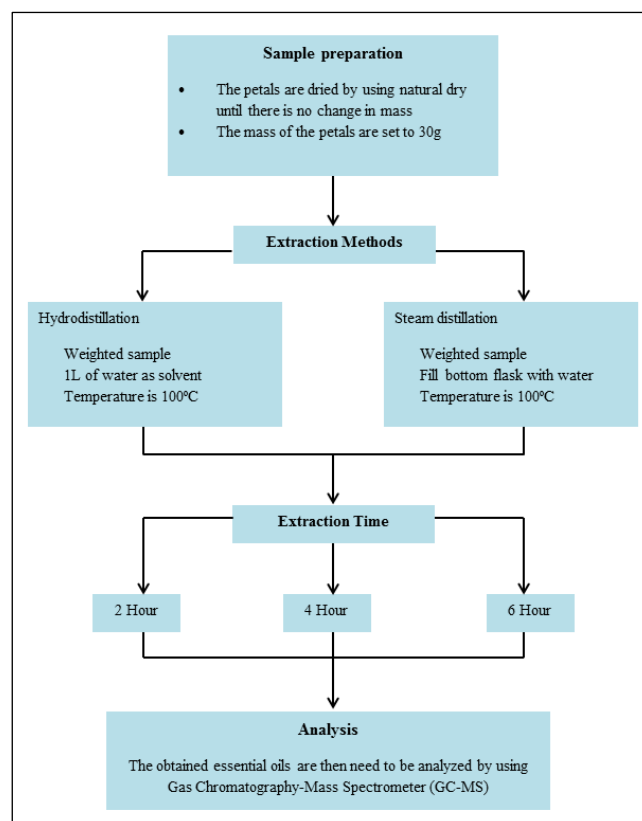


Fig. 1: Process Flow Diagram of the experimental work

## III. RESULTS AND DISCUSSION

### A. Moisture content

The initial mass of hibiscus petals weighted was 700±1 g. After 6 to 7 days, the final mass was constant at 105±1 g. The moisture content of hibiscus *rosa-sinensis* was calculated to be 85%.

### B. Effect of extraction time on percentage of oil yield

The extraction times used in this study were 2, 4, and 6 hour. Both HD and SD methods were tested in this study to see how the extraction time can affect the oil yield for hibiscus *rosa-sinensis*.

Based on figure 2 below, at 2 hour of extraction, HD yields 0.014% of oil. As the extraction conducted for 4 and 6 hours, the percentage of yield oil is increasing to 0.015% and 0.033% respectively. As for SD, 0.015% of oil is yield at 2 hour of isolation. Same with HD, oil yielded for 4, and 6 hours of SD extraction are increasing which are 0.02% and 0.036% respectively. The increasing of percentage oil yield as the

extraction time extended was due to the increasing of composition of essential oil obtained.

### C. Effect of method of extraction on percentage of oil yield

HD and SD methods of extraction were chosen in order to see the efficiency in yielding essential oil. The percentages of oil yields from both extraction methods are being compared as shown in the chart in figure 2. The oil yield from SD was higher than HD for all extraction times. For the extraction time of 2 hour, SD yields 29.17% higher than HD. For 4 and 6 hours, SD yields 28.04% and 10.63% respectively higher than HD. The percentages of difference were become smaller as the extraction time increase. This was because it had nearly reached the maximum of oil yields for both SD and HD methods. Based on research done in comparing method of extraction (HD and SD) and yield, it was proven that SD gave a better yield compare to HD (Boutekedjiret, Bentahar, Belabbes, & Bessiere, 2003).

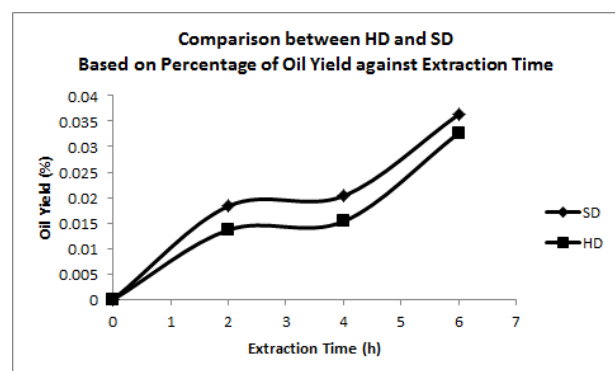


Fig. 2: Chart of Comparison between HD and SD

### D. Major volatile compounds in hibiscus rosa-sinensis

Gas chromatography- mass spectrometry was used to analyse the chemical compositions of the extracted oil. The components were identified by comparing their retention times and mass spectre with those standards, which are available in mass spectrometer library. Figure 3 and figure 4 shows the GC-MS result of oil extracted from HD and GC-MS result of oil extracted from SD respectively.

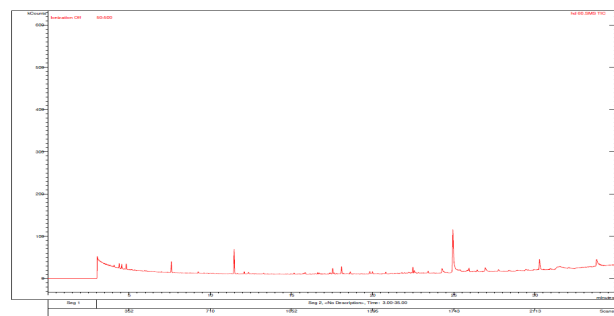


Fig. 3: GC-MS result of oil extracted from HD

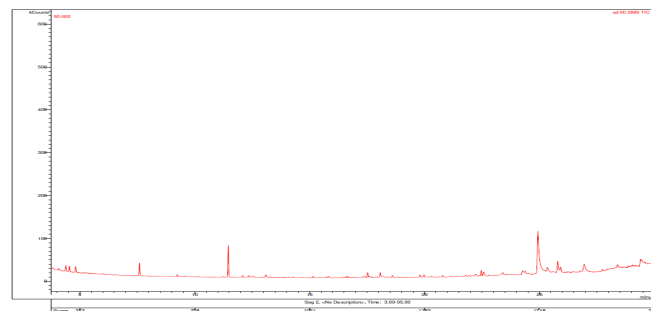


Fig. 4: GC-MS result of oil extracted from SD

From the result presented in the table 1, there are 9 major chemicals (*cis- $\alpha$ -Terpineol*, *Linalyl anthranilate*,  *$\alpha$ -Pinene*,  *$\beta$ -Selinene*, *Nonadecane*,  *$\alpha$ -Myrcene*, *Lavandulol*, *(E)- $\beta$ -Farnesene*, and *Isopropyl Myristate*) that present in hibiscus *rosa-sinensis* oil when being extracted with HD. Meanwhile, as for SD extraction as shown in table 2, there are 11 major chemicals (*cis- $\alpha$ -Terpineol*, *Linalyl anthranilate*,  *$\alpha$ -Pinene*,  *$\beta$ -Selinene*, *Nonadecane*,  *$\alpha$ -Myrcene*, *Lavandulol*, *(E)- $\beta$ -Farnesene*, *Isopropyl Myristate*, *Alloaromadendrene oxide-(1)*, and *Eicosane*) existed in the extracted oil. The chemicals found in hibiscus *rosa-sinensis* oil analysis have some similarity with the chemicals found in hibiscus *sabdariffa* in previous study (Ebije, Oladipupo, AbdulRazaq, & A. Ogunwande, 2014).

It can be seen that the compounds existed in the hibiscus *rosa-sinensis* that was extracted using SD were higher in terms of quality compared to the oil that was extracted by HD. For example, *Cis- $\alpha$ -Terpineol* was extracted by HD with 89% of quality. However, the quality was increase to 90% when it was extracted by SD. The quality of  *$\alpha$ -Pinene* also increases from 76% to 90% when SD was used instead of HD. Extraction of essential oil is more suitable to be extracted with SD in order to get better quality of chemicals in the oil (Boutekedjiret et al., 2003).

Table 1: Analysis of hibiscus *rosa-sinensis* quality extracted by HD

| GC-MS Hydrodistillation                    |            |             |
|--------------------------------------------|------------|-------------|
| Components                                 | CAS-number | Quality (%) |
| 1 <i>cis-<math>\alpha</math>-Terpineol</i> |            | 89          |
| 2 <i>Linalyl anthranilate</i>              | 7149-26-0  | 71          |
| 3 <i><math>\alpha</math>-Pinene</i>        | 7785-70-8  | 76          |
| 4 <i><math>\beta</math>-Selinene</i>       | 17066-67-0 | 69          |
| 5 <i>Nonadecane</i>                        | 629-92-5   | 62          |
| 6 <i><math>\alpha</math>-Myrcene</i>       | 1686-30-2  | 35          |
| 7 <i>Lavandulol</i>                        | 1845-51-8  | 29          |
| 8 <i>(E)-<math>\beta</math>-Farnesene</i>  | 18794-84-8 | 25          |
| 9 <i>Isopropyl Myristate</i>               | 110-27-0   | 85          |

Table 2: Analysis of hibiscus *rosa-sinensis* quality extracted by SD

| GC-MS Steam Distillation |                                          |             |    |
|--------------------------|------------------------------------------|-------------|----|
| Components               | CAS-number                               | Quality (%) |    |
| 1                        | <i>cis-<math>\alpha</math>-Terpineol</i> |             | 90 |
| 2                        | <i>Linalyl anthranilate</i>              | 7149-26-0   | 85 |
| 3                        | <i><math>\alpha</math>-Pinene</i>        | 7785-70-8   | 90 |
| 4                        | <i><math>\beta</math>-Selinene</i>       | 17066-67-0  | 66 |
| 5                        | <i>Nonadecane</i>                        | 629-92-5    | 70 |
| 6                        | <i><math>\alpha</math>-Myrcene</i>       | 1686-30-2   | 75 |
| 7                        | <i>Lavandulol</i>                        | 1845-51-8   | 75 |
| 8                        | <i>(E)-<math>\beta</math>-Farnesene</i>  | 18794-84-8  | 70 |
| 9                        | <i>Isopropyl Myristate</i>               | 110-27-0    | 76 |
| 10                       | <i>Alloaromadendrene oxide-(1)</i>       |             | 60 |
| 11                       | <i>Eicosane</i>                          | 112-95-8    | 55 |

Table 3: Analysis of hibiscus *rosa-sinensis* oil extracted from HD

| GC-MS Hydrodistillation                  |            |          |          |          |
|------------------------------------------|------------|----------|----------|----------|
| Components                               | CAS-number | 2 hr (%) | 4 hr (%) | 6 hr (%) |
| <i>cis-<math>\alpha</math>-Terpineol</i> |            | 0.316    | 0.768    | 0.0427   |
| <i>Linalyl anthranilate</i>              | 7149-26-0  | 0.0453   | 0.03     | 0.139    |
| <i><math>\alpha</math>-Pinene</i>        | 7785-70-8  | 0.0959   | 0.0846   | 0.359    |
| <i><math>\beta</math>-Selinene</i>       | 17066-67-0 | 0.00895  | 0.0712   | 0.0969   |
| <i>Nonadecane</i>                        | 629-92-5   | -        | 0.0691   | 0.0497   |
| <i><math>\alpha</math>-Myrcene</i>       | 1686-30-2  | 0.00438  | -        | 0.0237   |
| <i>Lavandulol</i>                        | 1845-51-8  | 0.00179  | -        | 0.0162   |
| <i>(E)-<math>\beta</math>-Farnesene</i>  | 18794-84-8 | 0.0108   | -        | 0.0146   |
| <i>Isopropyl Myristate</i>               | 110-27-0   | 0.0278   | 5.56     | 1.23     |
| Others                                   |            | 99.48908 | 93.4171  | 97.7822  |
| Total                                    |            | 100      | 100      | 100      |

Table 4: Analysis of hibiscus *rosa-sinensis* oil extracted from SD

| GC-MS Steam Distillation                 |            |          |          |          |
|------------------------------------------|------------|----------|----------|----------|
| Components                               | CAS-number | 2 hr (%) | 4 hr (%) | 6 hr (%) |
| <i>cis-<math>\alpha</math>-Terpineol</i> |            | -        | 0.2580   | 0.6700   |
| <i>Linalyl anthranilate</i>              | 7149-26-0  | 0.0182   | 0.4180   | 0.1220   |
| <i><math>\alpha</math>-Pinene</i>        | 7785-70-8  | -        | 1.1200   | 0.3330   |
| <i><math>\beta</math>-Selinene</i>       | 17066-67-0 | 0.0184   | 0.2080   | 0.0599   |
| <i>Nonadecane</i>                        | 629-92-5   | -        | 0.0269   | 0.0468   |
| <i><math>\alpha</math>-Myrcene</i>       | 1686-30-2  | -        | 0.0436   | 0.0128   |
| <i>Lavandulol</i>                        | 1845-51-8  | 0.4390   | 0.0532   | 0.0170   |
| <i>(E)-<math>\beta</math>-Farnesene</i>  | 18794-84-8 | 0.0095   | 0.0325   | 0.0034   |
| <i>Isopropyl Myristate</i>               | 110-27-0   | 0.0188   | 0.7500   | 0.9250   |
| <i>Alloaromadendrene oxide-(1)</i>       |            | 0.0143   | 0.1880   | -        |
| <i>Eicosane</i>                          | 112-95-8   | -        | 0.4480   | 0.0131   |
| Others                                   |            | 99.4819  | 96.4538  | 97.7970  |
| Total                                    |            | 100      | 100      | 100      |

In table 3 and table 4, it can be noticed that some chemicals are decreasing in value as the extraction time increase, vis a vis, some of them are increasing with increasing extraction time. Leakage of vapour during hydrodistillation and steam distillation or because of improper decantation may be the cause of the declination (Kumar, 2010). For example, the chemical that showed decrement for both HD and SD was  *$\alpha$ -Pinene*. The others chemical that was showing amount of 90 % above did not be considered as they contained low quality of chemicals in hibiscus *rosa-sinensis* essential oil.

*Isopropyl Myristate* was the chemical with highest amount can be seen based on the analyses for both methods. It has been used as flavoring agents in food additives, odor agents in industry uses, air care products, cleaning and furnishing care products, personal care products, and laundry and dishwashing products ("*Isopropyl Myristate*," n.d.). *Cis- $\alpha$ -Terpineol* was also among the highest chemical in term of amount that can be found in the hibiscus *rosa-sinensis* essential oil. It has been used as odor agents, intermediates, processing aids and solvents for cleaning or degreasing in industry uses. As for consumer uses, this chemical is used as air care products, cleaning and furnishing care product, laundry and dishwashing products, and personal care products ("*Alpha-Terpineol*," n.d.).

#### IV. CONCLUSION

In conclusion, extending the extraction time does increase the hibiscus *rosa-sinensis* essential oil yields. Higher yield of oil by SD prove that SD is a better than HD in term of quantitative. Analyses of the oil by GC-MS indicate that SD is more efficient than HD in term of qualitative. SD is good method in extracting more chemicals, but in term of amounts, HD managed to exceed SD.

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