

---

# Effect of Microwave Irradiation Power on The Bioactive Compound of Essential Oil from Indigenous Herb, *Physalis Angulata L.* Using Solvent Free Microwave Extraction

Muhammad Faiz Bin A Razak<sup>a</sup>, Nurhaslina Binti Che Radzi<sup>a</sup>,

<sup>a</sup>Faculty of Chemical Engineering, Universiti Teknologi MARA, Selangor, Malaysia

---

## Abstract

*Physalis angulata L.* is an herbal plant from the *Solanaceae* plant family and it have been used mainly in the medical purpose. The demand for a natural source to replace the usage of synthetic source for antibiotic. The antibiotic come from the bioactive components of the plant is obtained by using extraction process and irradiation microwave power is one of the affecting factors to the composition of the extracted product. The objective of this study is to investigate the effect of different irradiation power and extraction time on the bioactive compound. The essential oils from *Physalis angulata L.* was extracted by using Solvent Free Microwave Extraction (SFME). The essential oil of *Physalis angulata L.* was obtained with five differences power (270W, 360W, 450W, 540W, 630W) of irradiation power using a domestic microwave oven with wave frequency of 2.45GHz. The result from GC-MS show the present of 23 compound in the essential oil and the suitable irradiation microwave power for the extraction is 450W with the extraction yield of 0.4787%. These results indicated that the used of high irradiation microwave power more than the suitable value will affect the yield of *Physalis angulata L.* essential oil.

---

## Article Info

Article history:

Received date: DD Month 20XX

Accepted date: DD Month 20XX

---

Keywords:

*Physalis Angulata L.*  
Solvent Free Microwave  
Extraction  
Essential Oils  
Bioactive Compound  
Irradiation Power

---

## 1.0 Introduction

Plant especially herbal plants have been used to treat many infectious diseases since very ancient times. Scientific investigations that have been done on the plant materials clearly demonstrated the therapeutic efficacy of the plants over time[1]. *Physalis angulata L.* was a plant from *Solanaceae* botanic family and this herbaceous plant is native on North America, South America and Asian[2][3]. All parts of this plant can be used for many purposes especially for medical and the fruit of this plant is edible where it have been gathered traditionally. *Physalis angulata L.* has many many medical applications, including pain relievers, diuretics and antipyretics; this plant also can be used to cure malaria, gonorrhoea, asthma, diabetes, liver disorders, constipation, rheumatism, skin problems, wounds, digestive and intestinal disorders [4][5][6]. Based on the previous study that have been done, the withasteroids have been identified as the secondary metabolites which come from this plant[7]. This study

have uncovered a substantial variation related to chemical structure of the approximately 60 synthesized of withasteroids, including withanolides, physalins, withaphysalins, and withanolides with altered carbon skeletons[4][5][8]. Besides, *Physalis angulata L.* also shows several biological effects such as immunomodulatory, anti-inflammatory and trypanocidal effects[9][10]. Nowadays, numerous countries utilized the full benefit of the plants to treat diverse illnesses counting irresistible infections of the respiratory, gastrointestinal, urinary and biliary systems [11]. Even though the present chemical used for the antibiotics which comes from synthetic sources is very effective, but it still poses a harmful effect to human. Demand for natural sources was increase and the research on the essential oil has become the important focus for the researcher[12]. Therefore, an appropriate extraction method using green technology is believed to reduce the harmful effect and safe for human consumption.

Currently, the conventional and traditional method such as hydrodistillation, steam distillation and solvent extraction have been used in the extraction of the essential oils. However, these methods is time consuming, high energy consumption, low quality, low efficiency and yield[13]. The other method which is innovative and promising such as ultrasonic assisted extraction, microwave assisted extraction and supercritical fluid extraction also being used in the essential oil's extraction [14][15]. The quantity and the composition of the bioactive compound in the essential oil is might be varies with different extraction method applied[16]. For a couple of years, the change in state of mind has advanced progressively to a "green" tendency and there have been an expending request for modern greener methods for the extraction of essential oil. These modern green methods will more ecofriendly with lesser energy and organic solvent consumption, shorter time of extraction, low carbon dioxide and waste emissions, whereas keeping up a high quality of the extracts [17][18][19]. Nowadays, the utilization of the microwave technology such as Solvent free microwave extraction (SFME) have become desirable and recognized as the suitable alternatives for the conventional method that has been used and this extraction method have been applied by many researcher in several studies [17][20].

In the process of natural product, three analytical procedure was followed which are extraction, evaporation and lastly analysis. The duration for analysis just take a few minutes or second to be completed. The longest duration is taken by extraction or distillation process where it takes at least a few hours, or even days[21][22]. This process then is followed by a prolonged heating and stirring process in order to remove the solvent. The conventional method in solvent extraction procedures comprises 80% of the total process. Other than that, it need 90% of energy and need up to 99% of solvent used in order to complete the analysis procedures[15]. Solvent free microwave extraction (SFME) have been introduced as a key sustainable technology in reaching the green chemistry objective to overcome the drawback of the solvent extraction. This green technology was implemented to reduce or eliminate the use of solvent and consumption of energy. Moreover, SFME not only can reduce the emissions of carbon dioxide, it also can reduce the cost, increase the quantity and quality of the product[23][24]. By utilizing the help of microwave, the extraction process can be completed in just a few

minutes without using any solvent. The notable advantages in SFME is the low energy consumption, high reproducibility, shorter process time and high purity of final product produced[25]. Some classes of compound such as antioxidants and essential oil can be efficiently separated from different kind of raw materials. The objective of this study is to investigate the effect of different irradiation microwave power on the essential oil extracted and to determine the optimum power for the highest yield.

## 2.0 Methodology

### 2.1 Material

*Physalis angulata* L. leaves was collected from the Peninsular of Malaysia. The voucher specimen (054-19) was deposited at Forest Research Institute Malaysia (FRIM). The dried leaves was cutted (Approximately 1 mm) and stored at room temperature.

### 2.2 SFME apparatus and procedures

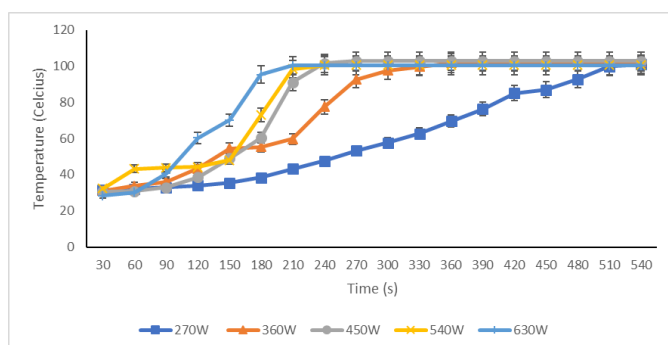
Modified domestic microwave oven with 900W maximum delivered power and the wave frequency of 2450MHz was used to setup solvent free microwave extraction. The modification has been made to the microwave oven by drilling a hole at the top of the microwave oven. A round bottom flask was used with the 500 mL capacity of the flask. It was placed inside the oven. The flask was put through the hole that have been drill and it was connected to the Clevenger apparatus. The hole then was closed by using an aluminum foils to prevent any loss of heat to the surroundings. In the standard solvent-free microwave extraction, the extraction procedure was performed at the atmospheric pressure where the plant samples was prepared. 50 g of the plant samples was soaked with 1000 ml of distilled water 60 minutes. Later, the excess water was removed, and the wetted plant was placed inside the 500 ml round bottom flask. The microwave oven was operated at different level of power (270W 360W, 450W 540W, 630W) until the plant material is dried. The extracted oils then are collected and in was stored at low temperature until it was used for the analysis[26]. The extraction yield of the essential oils was calculated by using by using the following equation [27]:

$$\text{Yield (\%, w/w)} = \frac{\text{Mass of extracted oils}}{\text{Mass of dried material}} \times 100 \quad (1)$$

### 3.0 Results and discussion

#### 3.1 Effect of irradiation microwave power

The function of irradiation power in the extraction is to control the amount of energy that can be received by the plant or samples where it will be converted into heat energy. This heat energy will help the release process of essential oil from the plant materials. The microwave power or the irradiation power that have been used in the solvent-free microwave extraction is more related to the temperature where the increase in the microwave power will cause the rapid increase in system temperature. Microwave power have become vital aspects as the power acts as the driving force in order to break the plant cell structure membrane and it will cause the essential oil to be diffused out and dissolved in the solvent. Therefore, the increase in the microwave power generally will speed up the extraction process and increase the yield of essential oil[26].

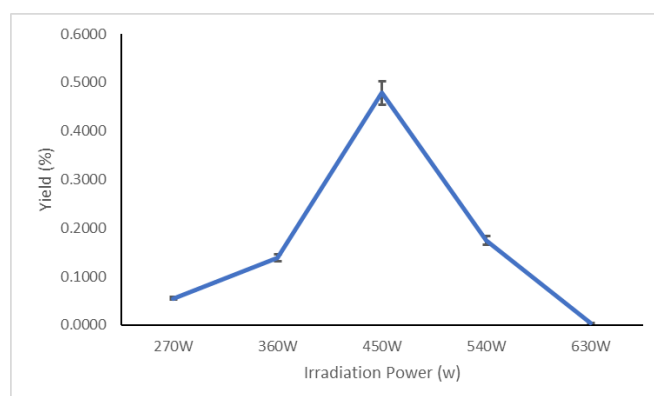


**Figure 1** The temperature profiles for each microwave power used on the extraction of *Physalis Angulata L.*

The rising speed of temperature for each of the microwave power can be measured and represented in the graph. Figure 1 shows the rise of the temperature for each irradiation power used in the extraction of *Physalis angulata L.* where based on the figure above, the higher irradiation power used, the shorter time for water to reach the boiling point [23]. When the irradiation power was set at the 270W, the time taken for the microwave oven to reach boiling point of water is 505 s while at 630 W takes only 180 s to reach the target temperature. This is because as more microwave power used, the higher the energy release by the radiation. The correct microwave power is important

because for the process to reach the water boiling point is depending on the sample plant size and weight.

From figure 1, the rapid increase in the temperature can be observe when 630W of microwave power input was use. However, in the utilization of solvent free microwave extraction technique, the extraction process is mainly affected by a single characteristic factor. Because of this factor causes the extraction by using the highest irradiation microwave power of 630W will not exactly give the highest yield of the extracted material.



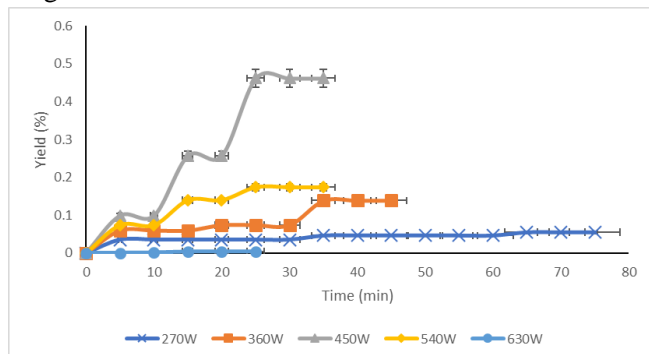
**Figure 2** The effect of irradiation microwave power to yield of *Physalis Angulata L.* essential oil by using different power

Based on Figure 2, it can be observed that the highest yield of 0.4787% for the *Physalis angulata L.* essential can be obtained at the irradiation microwave power of 450W. The moisture content in the plant cell is activated to rotate under the irradiation microwave and a rapid internal change cause by the resulting increase of pressure occur in the plant cells. Violent water vaporization and rapid decompression have caused the mechanical strain to the plants cell where this condition is affected by dehydrating effect and change in the glandular wall surface tension[23]. Based from the previous study, high yield of the essential oil is obtain when irradiation microwave power of 450W is utilize[26]. Based on the Figure 2 also, it can be observed that the lowest yield is obtain at 630W. The factor that effecting this result is the degradation of the plant matrix and compound of essential oil. The other that effecting low yield of essential oil is the carbonization of the plant sample occur because of the overheating when high microwave power is use. This was supported with research done by [20] where the microwave power up until 540W able to obtained high

yield of essential oil.

### 3.2 Effect of extraction time on yield of essential oil

Generally, there are three important stages involve in the process of extraction which are equilibrium phase, transition phase, and diffusion phase. During the phase of equilibrium, on the outer layer of the matrix there is the displacement of substrate occurs and this displacement of substrate takes place at the constant rate. After the equilibrium phase, the transition phase takes place where mass transfer occurs because of the convection and diffusion that happen in this stage. The diffusion phase is the last phase that occurs during the extraction process where at this stage the rate of extraction is slow and the release of the extracts during the extraction process through the diffusion mechanism is occurs[26]. In the *Physalis angulata L.* extraction process by using solvent-free microwave extraction techniques, extraction time is one of the important factors that need to be considered. Generally, the increase in the extraction time will cause the increase in the extraction yield but the yield of the essential oils may become lower due to increasing length of the extraction time.



**Figure 3** The effect of irradiation microwave time to yield of *Physalis Angulata L.* essential oil on different microwave power

Based on Figure 3, the effect of extraction time on the yield of *Physalis angulata L.* can be observed. At the microwave power input of 270W, the value of yield obtained is at 0.055%. According to the report by (Z. Liu et al.) [20], irradiation time is the important factor as the loss of volatile compound and degradation of plant samples will occur as longer time is applied.

### 3.3 Essential oil analysis

The chemical composition of the *Physalis angulata L.* essential oil was determined by using Gas-Chromatograph-Mass Spectrometer (GC-MS) and Table 1 shows the result from the GC-MS. Based on

the result, the GC-MS analysis of the essential oil shows 23 compounds was detected representing 31.05% of the total oil extracted. The main compound contains in the essential of were n-Hexadecanoic acid (14.032%), Z-10-Pentadecen-1-ol (7.02%), 9,17-Octadecadienal, (Z)- (1.41%), 9-Octadecenal (1.14%) and Cyclohexanol, 5-methyl-2-(1-methylethyl)-, (1à,2á,5à)-(ñ)- (0.882%). The other constituents present in the essential oil was less than 0.8%. The chemical compound present in the essential is depending on the method of extraction used and the parameter involve in the process. The amount of monoterpene and sesquiterpene present in the essential oil was small and most of the compound was categorized as others compound. Monoterpene was a chemical compound with 10 carbon atoms inside the molecules and sesquiterpene was a compound with 15 carbon atoms. Most of the chemical constituent detected in the essential oil consist of 16 carbon atom and 18 carbon atoms.

**Table 1** Chemical compound of *Physalis angulata L.* by GC-MS (450W, 35min)

| Retention Time | Compounds  | Amounts |
|----------------|--|---------|
| 31.2561        | Cyclohexanol, 5-methyl-2-(1-methylethyl)-, (1à,2á,5à)-(ñ)- | 0.882%  |
| 37.1076        | Cyclohexene, 1-(1,1-dimethylethoxy)-2-methyl-              | 0.240%  |
| 38.717         | 2-Cyclohexen-1-ol, 2,6,6-trimethyl-, acetate               | 0.104%  |
| 38.7276        | n-Hexadecanoic acid  | 14.032% |
| 38.7557        | Heptane, 1-(ethenylthio)-                                  | 0.233%  |
| 44.3843        | Cyclopentane, 2-isopropyl-1,3-dimethyl-                    | 0.551%  |
| 44.3911        | 1,15-Pentadecanediol                                       | 1.410%  |
| 44.3921        | 9,17-Octadecadienal, (Z)-                                  | 0.530%  |
| 44.4222        | 6-Octenoic acid, 3,7-dimethyl-, methyl ester               | 0.106%  |
| 37.0791        | 2-Nonanol  | 0.450%  |
| 38.7463        | Trans-3,5-dimethylthiane                                   | 0.189%  |
| 38.7763        | Trimethyl-(non-1-enyloxy)-silane                           | 0.127%  |
| 42.7174        | 5,10-Pentadecadien-1-ol, (Z,Z)-                            | 0.316%  |
| 42.7434        | 13-Tetradecene-11-yn-1-ol                                  | 0.161%  |
| 44.3589        | 6,6-Dimethyl-1,3-heptadien-5-ol                            | 0.319%  |
| 44.3696        | 1-Methyl-3,3-pentamethylenediaziridine                     | 0.264%  |
| 44.3793        | 9-Octadecenal  | 1.140%  |
| 44.388         | Z-10-Pentadecen-1-ol                                       | 7.020%  |
| 44.3967        | 6-Octadecenoic acid, (Z)-                                  | 0.592%  |
| 21.1451        | Hexadecanoic acid, ethyl ester                             | 0.122%  |
| 37.0707        | Undecanoyl chloride  | 0.803%  |
| 38.7291        | 4-Pentadecanol   | 0.634%  |
| 44.3933        | 9,12-Octadecadienal  | 0.825%  |

#### 4.0 Conclusions

Solvent free microwave extraction was utilized for the essential oil extraction from *Physalis angulata* L. and based on the result shows that the irradiation microwave power was the main factor affecting the overall process. Based on the extraction process that have been done, the suitable irradiation microwave power for the extraction is 450W and the extraction time needed to complete the process is 35 minutes with the extraction yield of 0.4787%. the observation has been made regarding to the irradiation power used in the experiment where low extraction obtained when the power more than 450W was used. It can be concluded that the irradiation microwave power is one of the main factors that effecting the overall efficiency of the solvent free microwave extraction. The parameter is not only affecting the yield but also the number of bioactive components present in the essential oil.

#### Acknowledgment

The author would to thanks Universiti Teknologi Mara (UiTM) and Faculty of Chemical Engineering for the guidance and facility provided for the research.

#### References

- [1] C. S. Meira, E. T. Guimarães, J. A. F. Dos Santos, D. R. M. Moreira, R. C. Nogueira, T. C. B. Tomassini, I. M. Ribeiro, C. V. C. De Souza, R. Ribeiro Dos Santos, and M. B. P. Soares, "In vitro and in vivo antiparasitic activity of *Physalis angulata* L. concentrated ethanolic extract against *Trypanosoma cruzi*," *Phytomedicine*, vol. 22, no. 11, pp. 969–974, 2015.
- [2] R. da Silva Leite, M. N. do Nascimento, T. T. Tanan, L. P. Gonçalves Neto, C. A. da Silva Ramos, and A. L. da Silva, "Alleviation of water deficit in *Physalis angulata* plants by nitric oxide exogenous donor," *Agric. Water Manag.*, vol. 216, no. November 2018, pp. 98–104, 2019.
- [3] V. Kumar, D. K. Singh, S. Mohan, R. K. Gundampati, and S. H. Hasan, "Photoinduced green synthesis of silver nanoparticles using aqueous extract of *Physalis angulata* and its antibacterial and antioxidant activity," *J. Environ. Chem. Eng.*, vol. 5, no. 1, pp. 744–756, 2017.
- [4] E. Maldonado, N. E. Hurtado, A. L. Pérez-Castorena, and M. Martínez, "Cytotoxic 20,24-epoxywithanolides from *Physalis angulata*," *Steroids*, vol. 104, no. June 2012, pp. 72–78, 2015.
- [5] G. C. Moreira and F. de Souza Dias, "Mixture design and Doehlert matrix for optimization of the ultrasonic assisted extraction of caffeic acid, rutin, catechin and trans-cinnamic acid in *Physalis angulata* L. and determination by HPLC DAD," *Microchem. J.*, vol. 141, no. April, pp. 247–252, 2018.
- [6] C. C. Hsu, Y. C. Wu, L. Farh, Y. C. Du, W. K. Tseng, C. C. Wu, and F. R. Chang, "Physalin B from *Physalis angulata* triggers the NOXA-related apoptosis pathway of human melanoma A375 cells," *Food Chem. Toxicol.*, vol. 50, no. 3–4, pp. 619–624, 2012.
- [7] Y. Zhang, C. Chen, Y. L. Zhang, L. Y. Kong, and J. G. Luo, "Target discovery of cytotoxic withanolides from *Physalis angulata* var. *villosa* via reactivity-based screening," *J. Pharm. Biomed. Anal.*, vol. 151, pp. 194–199, 2018.
- [8] C. N. Casero, J. C. Oberti, C. I. Orozco, A. Cárdenas, I. Brito, G. E. Barboza, and V. E. Nicotra, "Withanolides from three species of the genus *Deprea* (Solanaceae). Chemotaxonomical considerations," *Phytochemistry*, vol. 110, pp. 83–90, 2015.
- [9] B. J. M. Da Silva, S. W. G. Pereira, A. P. D. Rodrigues, J. L. M. Do Nascimento, and E. O. Silva, "In vitro antileishmanial effects of *Physalis angulata* root extract on *Leishmania infantum*," *J. Integr. Med.*, vol. 16, no. 6, pp. 404–410, 2018.
- [10] L. a. Pinto, C. S. Meira, C. F. Villarreal, M. a. Vannier-Santos, C. V. C. De Souza, I. M. Ribeiro, T. C. B. Tomassini, B. Galvão-Castro, M. B. P. Soares, and M. F. R. Grassi, "Physalin F, a seco-steroid from *Physalis angulata* L., has immunosuppressive activity in peripheral blood mononuclear cells from patients with HTLV1-associated myelopathy," *Biomed. Pharmacother.*, vol. 79, pp. 129–134, 2016.
- [11] A. Sharma, R. D. C. Flores-Vallejo, A. Cardoso-Taketa, and M. L. Villarreal, "Antibacterial activities of medicinal plants used in Mexican traditional medicine," *J.*

- Ethnopharmacol.*, vol. 208, pp. 264–329, 2017.
- [12] J. J. Guo, Z. P. Gao, J. L. Xia, M. a. Ritenour, G. Y. Li, and Y. Shan, “Comparative analysis of chemical composition, antimicrobial and antioxidant activity of citrus essential oils from the main cultivated varieties in China,” *Lwt*, vol. 97, no. August, pp. 825–839, 2018.
- [13] X. J. Li, W. Wang, M. Luo, C. Y. Li, Y. G. Zu, P. S. Mu, and Y. J. Fu, “Solvent-free microwave extraction of essential oil from *Dryopteris fragrans* and evaluation of antioxidant activity,” *Food Chem.*, vol. 133, no. 2, pp. 437–444, 2012.
- [14] Y. Wang, R. Li, Z. T. Jiang, J. Tan, S. H. Tang, T. T. Li, L. L. Liang, H. J. He, Y. M. Liu, J. T. Li, and X. C. Zhang, “Green and solvent-free simultaneous ultrasonic-microwave assisted extraction of essential oil from white and black peppers,” *Ind. Crops Prod.*, vol. 114, no. October 2017, pp. 164–172, 2018.
- [15] F. Chemat, A. S. Fabiano-Tixier, M. A. Vian, T. Allaf, and E. Vorobiev, “Solvent-free extraction of food and natural products,” *TrAC - Trends Anal. Chem.*, vol. 71, pp. 157–168, 2015.
- [16] M. Boukroufa, C. Boutekedjiret, L. Petigny, N. Rakotomanomana, and F. Chemat, “Bio-refinery of orange peels waste: A new concept based on integrated green and solvent free extraction processes using ultrasound and microwave techniques to obtain essential oil, polyphenols and pectin,” *Ultrason. Sonochem.*, vol. 24, pp. 72–79, 2015.
- [17] M. Abdelhadi, A. Meullemiestre, A. Gelicus, A. Hassani, and S. A. Rezzoug, “Intensification of *Hypericum perforatum* L. oil isolation by solvent-free microwave extraction,” *Chem. Eng. Res. Des.*, vol. 93, no. April, pp. 621–631, 2015.
- [18] A. Filly, X. Fernandez, M. Minuti, F. Visinoni, G. Cravotto, and F. Chemat, “Solvent-free microwave extraction of essential oil from aromatic herbs: From laboratory to pilot and industrial scale,” *Food Chem.*, vol. 150, pp. 193–198, 2014.
- [19] G. Khalili, A. Mazloomifar, K. Larijani, M. S. Tehrani, and P. A. Azar, “Solvent-free microwave extraction of essential oils from *Thymus vulgaris* L. and *Melissa officinalis* L.,” *Ind. Crops Prod.*, vol. 119, no. April, pp. 214–217, 2018.
- [20] Z. Liu, B. Deng, S. Li, and Z. Zou, “Optimization of solvent-free microwave assisted extraction of essential oil from *Cinnamomum camphora* leaves,” *Ind. Crops Prod.*, vol. 124, no. August, pp. 353–362, 2018.
- [21] C. Zhao, X. Yang, H. Tian, and L. Yang, “An improved method to obtain essential oil, flavonols and proanthocyanidins from fresh *Cinnamomum japonicum* Sieb. leaves using solvent-free microwave-assisted distillation followed by homogenate extraction,” *Arab. J. Chem.*, 2018.
- [22] M. E. Lucchesi, F. Chemat, and J. Smadja, “Solvent-free microwave extraction of essential oil from aromatic herbs: Comparison with conventional hydro-distillation,” *J. Chromatogr. A*, vol. 1043, no. 2, pp. 323–327, 2004.
- [23] Y. Li, A. S. Fabiano-Tixier, M. A. Vian, and F. Chemat, “Solvent-free microwave extraction of bioactive compounds provides a tool for green analytical chemistry,” *TrAC - Trends Anal. Chem.*, vol. 47, pp. 1–11, 2013.
- [24] O. O. Okoh, a. P. Sadimenko, and a. J. Afolayan, “Comparative evaluation of the antibacterial activities of the essential oils of *Rosmarinus officinalis* L. obtained by hydrodistillation and solvent free microwave extraction methods,” *Food Chem.*, vol. 120, no. 1, pp. 308–312, 2010.
- [25] H. Benmoussa, A. Farhat, M. Romdhane, and J. Bouajila, “Enhanced solvent-free microwave extraction of *Foeniculum vulgare* Mill. essential oil seeds using double walled reactor,” *Arab. J. Chem.*, 2015.
- [26] H. S. Kusuma, A. Altway, and M. Mahfud, “Solvent-free microwave extraction of essential oil from dried patchouli (*Pogostemon cablin* Benth) leaves,” *J. Ind. Eng. Chem.*, vol. 58, pp. 343–348, 2018.
- [27] X. L. Qi, T. T. Li, Z. F. Wei, N. Guo, M. Luo, W. Wang, Y. G. Zu, Y. J. Fu, and X. Peng, “Solvent-free microwave extraction of essential oil from pigeon pea leaves [*Cajanus cajan* (L.) Millsp.] and evaluation of its antimicrobial activity,” *Industrial Crops and Products*, vol. 58, pp. 322–328, 2014.