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Extraction methods of essential oil from kaffir lime (*Citrus hystrix*): A review

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Abstract

Essential oil is a valuable product in various industries but unfortunately, it is scarce. Various extraction methods currently being used to obtain the essential oil from kaffir lime leaves (C*itrus hystrix*), however, to date, best extraction methods are yet to be concluded. This review paper highlights the production yield, pros and cons of various kaffir lime leaves essential oil extraction methods available in present research papers albeit limited to lab scale. Each methodology has their own advantages and disadvantages, so it is not possible to say that one method is the best. This review also covers the pre-treatment processes as since they can significantly affect the performance of other important processes as well as the production yield. In conclusion, each of the extraction and pre-treatment method has its own pros and cons, hence selecting a suitable method depend heavily on the producer's requirements.

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1.0 Introduction

Kaffir lime or "limau purut" is a citrus fruit found locally in Southeast Asia countries such as Malaysia, Indonesia and Thailand. Essential oil derived from plants are in very high demand in the modern age as it is an important component for various products ranging from food and aromatics to medicine and agriculture, especially so for essential oils extracted from kaffir lime. Aside from their lovely fragrance, they are often used to combat depression and anxiety (Ades, 2009; Awang, 2007; Guzmán-Gutiérrez et al., 2015; Russo, 2011). In the medical field, they are used as antimicrobial, analgesic, sedative, anti-inflammatory, spasmolytic, and anaesthetic remedies (Bakkali et al., 2008; Lertsatitthanakorn et al., 2006; Luangnarumitchai et al., 2007). It can even be used to combat the growth of malignant cells and prevent cancer (Anindito et al., 2015). In agriculture, it can be used to protect grains due to its ability to kill rice weevil and this ability may be attributed to the β-citronellal in the oil (Adedire & Ajayi, 1996; Buatone & Indrapichate, 2011; Loh et al., 2011; Md Othman et al., 2016). In pisciculture, bacterial fish disease can severely cause economic losses and it was discovered that lime nano-emulsion was found to be effective in treatment of bacterial infection in Tilapia fish and can be used for treating bacterial infection in fish farm (Thomas et al., 2014).

The essential oil composition and content depend on various reasons. In the study by Ghafar (2013), it was found that citronellal is the main volatile compound that can be found in kaffir lime leaves. The essential oil from fresh kaffir lime leaves particularly are rich in β-citronellal which was the major compound present with 66.85% of total oil followed by β-citronellol (6.59%), linalool (3.90%) and citronellol (1.76%) in the research conducted by Loh et al. (2011). Meanwhile, for dried kaffir lime leaves, the major compounds have slightly different concentrations such as 69.96% for β-citronellal, 6.67% for β-citronellol and 3.86% for linalool (Ismail, 2016). In contrast to the findings of Loh et al. (2011), it was found that essential oil from kaffir lime leaves also contain α-farnesene and camphor (Mansor & Sauid, 2015). Because of phytochemical polymorphism, the essential oil composition often changes in different parts of the plant and sometimes even between different oil glands of a single leaf albeit rarely (Johnson et al., 2004). The growth phase of the plant also affects the essential oil yield such as flowering stage, vegetative stage and fruiting stage (Johnson et al., 2004; Novak et al., 2006; Slavkovska et al., 2013). Besides that, the composition and oil yield are also dependent on the location or region they were taken (Dardioti et al., 2012). Hence, minor discrepancies may occur even though different studies were carried out using the same method.

Extraction methods are what majorly affect the production yield and composition. Conventional methods such as Soxhlet extraction and hydro-distillation have been used for a long time for the extraction of bioactive compounds from plants and herbs. With the advancement of technology, more techniques were invented, and studies were made on the performances of the new techniques.

2.0 Methodology

2.1 Extraction methods

Hydro-distillation

Hydro-distillation is relatively cheap, simple, eco-friendly, and produce great oil quality (Kusuma & Mahfud, 2015). Hydro-distillation is suitable for preventing damage to the leaves. Charring and degradation of the leaves could be prevented because the leaves are not directly heated. It is still important not to put too much water as this will lead to increase in energy consumption to raise and maintain the temperature at optimum temperature as well as causing hydrolytic effect that decreases oil yield and quality (Kusuma & Mahfud, 2016). The drawback of using hydro-distillation is the slow extraction process. Furthermore, it usually requires four hours to extract the essential oil as it took three hours to obtain the peak yield percentage (Bousbia et al., 2009; Chanthaphon et al., 2008; Md Anjazi & Sauid, 2015) as exhibited in Table 1.

Table 1: The hydro-distillation period effects on yield (Md Anjazi & Sauid, 2015).

Hydro-distillation period (h)	Yield (%)
2.59	1.14
3.00	1.75
4.00	1.5
5.00	1.3
5.41	1.23

Steam distillation

For steam distillation, the leaves are not submerged in the water, instead held above the water in a packed bed. The water is boiled, and steam produced will pass through the leaves, breaking down the cell structure and releasing the volatile compounds. The vapor will then condense into a flask and the condensate contains both water and oil. Water-soluble compounds will dissolve at a lower extent in the condensate (Tongnuanchan & Benjakul, 2014).

Steam distillations took at most two hours to obtain maximum yield (Kasuan et al., 2013; Mohd Yusoff et al., 2013). The higher the steam temperature, the richer the oil extracted (Kasuan et al., 2013). The hypothesis is supported by the findings of Mohd Yusoff et al. (2013), in which, increasing the temperature of the steam increased the yield of essential oil. The highest yield was obtained at 95 °C with 2.94%.

Supercritical fluid extraction (SFE)

Supercritical CO_2 extraction has advantages over other extraction techniques such as low operating temperatures as well as its non-toxic and inert properties make SFE an excellent choice for the recovery of valuable compounds from medicinal herbs (Roldán-Gutiérre et al., 2008). CO_2 also allows low operating pressure and temperature parameters setting (Reverchon & Marco, 2006). The extraction period is also quite short (Wetwitayaklung et al., 2009). It is a rapid, selective and convenient method for separating and fractionating active compounds (Norulaini et al., 2004). In comparison with Soxhlet extraction, supercritical fluid carbon dioxide extraction saves time and provide high extraction rate (Zhang et al., 2011). However, the yield was obtained from SFE.

Pressurised liquid extraction (PLE)

PLE is a method of extraction using liquid solvents at elevated pressure and temperature. This method improves the extraction performance when compared to room temperature and atmospheric pressure. The efficiency of the extraction is dependent on the solubility of the analyte in the extraction solvent as well as the partitioning of the target-compound between the water and the extraction solvent (Acta et al., 2011). The advantages of using this technique of extraction are higher extraction yield, short processing time, clean extracts and low solvent consumption (Acta et al., 2011; Delgado-Zamarreño et al., 2004). Organic solvents such as hexane, methylene chloride, isopropanol, and ethanol work more efficiently as solvent since the desired product is organic. Water is better for the extraction of polar compounds but less favourable extraction of non-polar compounds, so it will provide less satisfactory results (Marriott et al., 2001; Ong, 2004).

PLE can produce significant amount of oil with the percentage yield of 47.27% on dry weight basis. This was done at 1000 psi and 100 °C for 30 minutes using n-hexane (Haiyee & Winitkitcharoen, 2012). The yield may look fantastic, but this is from only 2 g of sample. This is due to the limitation of the PLE itself in which the maximum amount of sample that can be loaded on the extraction thimble at laboratory scale is merely 10 g (Suchan et al., 2004). In the study by Ghafar (2013), the oil yield obtained from optimised PLE was 56.16%. The equipment itself is quite expensive, but there has been a successful custom-made PLE system used in the study of Sanagi et al., (2005). The extraction recovery efficiency is approximately 100% relative to conventional Soxhlet extraction.

Soxhlet extraction

Soxhlet extraction is a very common technique for obtaining volatile components from raw materials. It is done by heating up a solution with a solute of limited solubility in a percolator until it boils, then condensing and collecting the condensate from a reservoir in which the concentrated solute can be obtained. The yield of essential oil from Soxhlet extraction with ethanol solvent at 81 °C to 96 °C for 25.5 hours was 13.39% (Munawaroh & Astuti, 2010). For 2 g of kaffir lime leaves, a Soxhlet extraction with n-hexane for 16 hours yield up to 22.80% oil yield (Haiyee & Winitkitcharoen, 2012). Even though the yield is high, the processing time is very long (Ong, 2004; Pourmortazavi & Hajimirsadeghi, 2007).

Microwave hydro-diffusion and gravity (MHG)

Microwave hydro-diffusion and gravity was founded by Bousbia, Vian, Ferhat, Meklati, and Chemat in 2009. It applies the concept of physical and chemical phenomena that are fundamentally different from those applied in conventional solvent extraction and distillation techniques. Without adding solvent or water, extraction can simply be done with modified microwave and cooler. The method for MHG is relatively simple. Without drying the plant sample, it is microwaved directly without adding any solvent and the oil within the sample will diffuse out along with natural water inside the sample in a physical phenomenon known as hydro-diffusion. The oil will be continuously extracted when it drops out into a cooler connected to the bottom of the microwave. The advantages of this method are high product purity, does not require any solvent or post-treatment of waste water and the consumption of only a fraction of the energy normally required by conventional food extraction methods. Munawaroh & Astuti (2010) noted that for hydro-distillation, the essential oil has an odour of terpenes hydrocarbons fresh, pungent but different from fresh fruit and with a persistent boiled odour. However, MHG method offers the possibility for a better reproduction of natural aroma of the fruit essential oil comparable to cold press method but more than the hydro-distilled essential oil. It also yields oil faster than normal hydro-distillation in which 15 minutes of MHG produces the same amount as 3 hours of hydro-distillation (Bousbia et al., 2009). The yield of the essential oil from this method is comparable to that of hydro-distillation which is 1.0% to 1.1%.

Solvent extraction

Common solvents used for bioactive compounds extraction are ether, methanol and hexane. It is often used for heat sensitive and fragile material such as flowers which would not be able to handle the heat of high temperature extraction such as steam distillation.

It is one of the simplest methods for isolating volatile compounds, but it uses a large amount of solvent. In the study of Tinjan and Jirapakkul (2007), solvent extraction method was used to isolate free and glycosidically bound volatile compounds from fresh kaffir lime leaves. Free and glycosidically bound volatile compounds extracted by solvent method had the kaffir lime leaf characteristic odour. Unfortunately, the use of solvent extraction has multiple drawbacks. Using great amount of synthetic chemical solvents may influence the ecological equilibrium and render the oil unsuitable for pharmaceutical and food usage (Bakkali et al., 2008). Furthermore, solvent extraction also complicates the downstream processes to concentrate and purify the product. In the study of Zaibunnisa et al. (2009), it was found that in the downstream process, emulsion formation was a major problem and the complete removal of the compounds was not successful. A compilation of various extraction methods indicating the advantages and disadvantages as well as the yield of essential oil obtained is summarised in Table 2.

2.2 Pre-treatment methods

Pre-treatment methods are techniques used in order to compliment the extraction process. In this case, it is not directly involved with extracting the oil, but improving the materials for the extraction processes. The effects of pre-treatment vary depending on the method of treatment and types of raw materials. It may increase final product purity, decrease other process workload, increase the efficiency of other processes, and prevent equipment damage.

Ultrasound

Ultrasonic pre-treatment is a non-thermal technique that can help modify the material structure without posing any problem to the desired heat-sensitive bioactive compounds (Pongmalai et al., 2013). Ultrasound treatment can greatly influence the drying kinetics (Musielak et al., 2016). In the research of Śledź et al. (2014), it was indicated that an ultrasound pre-treatment resulted in up to 56% reduction of drying period while maintaining or only slightly decreasing total phenolic content. It was also noted in the same study that ultrasound modifies the tissue microstructure of leaves and can increase the heat and mass transfer.

In their study, longer session of ultrasound application contributed to greater reduction of the drying time although not indefinitely. Since the microstructure plays an important role in determining the effectiveness of sonication on the material, it is important to adjust the parameters of sonication to different materials separately.

Table 2: Overview of oil yield, advantages and disadvantages of various extraction methods.

Methods	Essential Oil Yield (%)	Advantages	Disadvantages
Hydro-distillation	1.1 (Bousbia et al., 2009) 0.82 (Md Anjazi & Sauid, 2015; Zakaria & Sauid, 2016) 0.83 (Yusoff & Sauid, 2016) 0.78 (Ismail & Sauid, 2016)	Can prevent charring and degradation. Simple to configure.	Slow process and requires a lot of heat energy and water.
Steam Distillation	4.26 (Kasuan et al., 2013) 3.11 (Mohd Yusoff et al., 2013)	Pressure can be manipulated to handle heat sensitive materials. The higher the steam temperature, the richer the oil yields.	May char the leaves and requires great amount of heat energy to produce steam continuously.
Supercritical Fluid Extraction	1.24 (Trabelsi et al., 2016) 0.33 (Norkaew, Pitija, Pripdeevech, Sookwong, & Wongpornchai, 2013)	Low operating temperatures. Uses non-toxic and inert substances. Using ethanol-water co-solvent can reduce solvent volume needed.	High installation costs. Requires solvents in addition to raw materials.
Pressurized Liquid Extraction	47.27 (Haiyee & Winitkitcharoen, 2012) 56.16 (Ghafar, 2013)	Higher extraction yield, short processing time, clean extracts and low solvent consumption.	Expensive initial cost. Using water may yield less-satisfactory results.
Soxhlet Extraction	13.39 (Munawaroh & Astuti, 2010) 22.80 (Haiyee & Winitkitcharoen, 2012)	Common and conventional method which can produce high yield.	Very long process and can be very expensive due to the usage of large amount of costly solvents.
Microwave Hydro-diffusion and Gravity (MHG)	1.0 (Bousbia et al., 2009)	Does not require any solvent and has lower energy consumption compared to other high temperature methods. Retains the organoleptic qualities. Yields oil faster than normal hydro-distillation.	Yield of oil is comparable but lesser than other conventional methods.
Solvent Extraction		Suitable for heat-sensitive materials. Simple to setup. Effective at separating free and glycosidically bound volatile compounds.	Consume large amount of solvent. Can negatively affect the ecological equilibrium. Complicates downstream process.

Table 3: The effects of ultrasonic pre-treatment on essential oil yield for longer period (Md Anjazi & Sauid, 2015)

Ultrasonic Pre-treatment (min)	Yield (%)
0	1.3
60	1.5
120	1.75
145	0.82

Ultrasound can improve the quality and yield of oil because the implosions of cavitation bubbles cause fissions at cell walls, hence aiding in solvent penetration and rapid exudation of components (Chemat et al., 2016; Santos et al., 2014; Zhang et al., 2008). Ultrasound-assisted extraction can obtain valuable compounds from plants without some of the cons of conventional techniques like losses and degradation of volatile and thermolabile compounds. In multiple situations, it is faster and more efficient than conventional extraction (Roldán-Gutiérrez et al., 2008). Ultrasound application can reduce colour changes, lower water activity and reduce the loss of some nutrient elements (Fan et al., 2017). It was found that the longer the pre-treatment period, the greater the average yields (Zakaria & Sauid, 2016). Although, the duration of the ultrasound treatment must be controlled since the energy released from the popping bubbles increase the temperature (Sandra & Ashokkumar, 2011). Hence, prolonged period of ultrasound treatment may vaporise some of the volatile compounds before the extraction process itself, and hence decreasing the yield as exhibited by Md Anjazi & Sauid (2015) in Table 3. The optimal time period for ultrasound treatment of fresh kaffir lime leaves is 120 minutes at frequency of 53 kHz (Rosli & Sauid, 2016).

Drying

Drying is a very important process of pre-treatment because it removes water from the samples. Drying increases the concentration of volatile components in the leaves compared to fresh, undried leaves (Raksakantong et al., 2011). Moderate temperature is usually used for drying herbs because at high temperature exposure such as 70 °C, the oil glands may break. This significantly reduced the amount of oil yield as proven in studies (Kumar et al., 2016).

Compounds get dragged to the leaf surfaces and are lost during drying by the evaporating water, so herbs must have some moisture content before analysis to avoid damage to the active chemical constituents.

Table 4: The result of ultrasonic pre-treatment on oil yield from fresh and dried kaffir lime leaves.

Time (min)	Average of Yield from fresh kaffir lime leaves (%) (Rosli & Sauid, 2016)	Average of Yield (%) from dried kaffir lime leaves (Zakaria & Sauid, 2016)
0	0.265	0.537
60	0.295	0.818

Hence, dried sample will give higher yield than fresh sample (Kamal et al., 2011; Singh et al., 2014). Table 4 shows a comparison of the yield of oil for fresh and dried kaffir lime leaves extraction using hydro distillation.

Physical size alteration

Cutting or grinding the leaf samples improves the extraction of essential oils by reducing distillation time, lowering energy and increasing the efficiency of the distillation process. It was noted in the study of Singh et al., (2014) that the yield increased with decreasing particle size regardless the type of extraction methods. This can be attributed to the increase in surface area when the leaves are chopped. As the surface area increases, heat transfer becomes more efficient and mass transfer resistance decreases. Inversely, if particle size increases, the resistance to mass transfer increase and this inhibits the transportation of oil from the cell to the solvent (Singh et al., 2014).

Particle size is an important factor in extraction and decreasing the diameter enhances the mass transfer leading to a better extraction yield (Ouzzar et al., 2015). Furthermore, decreasing the internal mass resistance causes equilibrium conditions to control the extraction, hence shortening the distillation period (Kusuma & Mahfud, 2016). The same authors also found that the oil yield for chopped leaves were higher than the intact full leaves, whereby the highest yield is 2.3% and 2.0%, respectively. The physical structure of the materials is critical in determining the extraction efficiency as it is related to the ability of the water vapour to diffuse within the materials (Pourmortazavi & Hajimirsadeghi, 2007).

Effective microbe enzymes

Efficient recovery of oil enclosed in the cell results from cell walls disruption. It may be done by specific enzymes that breakdowns the individual types of

Table 5: The yield of essential oils from kaffir lime leaves with different pre-treatment time (Ismail & Sauid, 2016; Yusoff & Sauid, 2016)

Effective Microbe Pre-treatment Period (h)	Yield of oil from dried leaves (%) (Yusoff & Sauid, 2016)	Yield of oil from fresh leaves (%) (Ismail & Sauid, 2016)
0	0.196	0.09
12	0.833	0.78
18	0.750	0.52
24	0.596	0.47
30	0.459	0.46

polysaccharides in the cell wall structure (Najafian et al., 2009). Effective microbe enzymes refer to using biological waste as catalysts for improving essential oil extraction. The biological waste may come from various sources such as pineapple, apple and other fruit peels. These wastes contain multiple reusable components with high value and potential (Upadhyay et al., 2013).

Bacterial and fungal culture can be isolated from fruit wastes and all the culture would produce pectinase enzyme albeit in varying amount (Geetha et al., 2012). The effective microbe produces enzymes pectinase which can degrade the pectin substances in the leaves to release the oil. This is the reason for the oil yield increases and extraction time can be shortened (Kashyap et al., 2001; Perez et al., 2013). Pectinase can be used to hydrolyse these substances and they can be found in agricultural and food commodities (Jayani et al., 2015). Theoretically, enzyme assisted extraction can increase the yield of essential oil without changing much of its chemical and physical properties (Chandran et al., 2012).

In the research of Yusoff & Sauid (2016) it was found that the optimum effective microbe pre-treatment period for dried kaffir lime leaves was 12 hours. Their experiment involved growing microbes from pineapple peels for three months anaerobically to obtain the enzymes before spraying it onto kaffir lime leaves as pre-treatment process. Using hydro-distillation, they managed to successfully extract essential oil from kaffir lime leaves and concluded that the destruction of the cell walls by the effective microbe enzymes contributed to higher essential oil yield from hydro-distillation. Furthermore, their findings were supported by another similar research done by Ismail & Sauid (2016). They also found the highest yield for fresh kaffir lime leaves resulted from 12-hour pre-treatment with effective microbe. The comparison of the two studies is shown in Table 5.

Ismail & Sauid (2016) also reported that the leaves were still green at 12 hours of treatment but when the

Table 6: The oil yield of kaffir lime leaves with different pre-treatment (Mansor & Sauid, 2015).

Pre-treatment	Maximum Oil Yield (%)
None	1.1942
Ultrasonic	2.9780
Effective Microbe	2.4550
Ultrasonic + Effective Microbe	6.9942

effective microbe treatment was prolonged to 30 hours, the leaves turned to brownish in colour and this could contribute to the lower amount of oil yield obtained. Collectively, the pre-treatment methods synergistically increase the maximum oil yield, and this was proven by the research conducted by Mansor & Sauid (2015). The results of their findings are tabulated in Table 6.

3.0 Conclusions

A review of the collective techniques of extraction and pre-treatment was presented in this article. The yield of the essential oil differs between extraction and pre-treatment methods. Methods like PLE and Soxhlet extraction boast high yield production but the trade-offs are low material loading and extremely long extraction period respectively. The new MHG method yields oil quickly but at total yield is lower than conventional methods. Each of the method has its own pros and cons. Hence, selecting a method depends heavily on the requirements of the producer.

It should also be noted that pre-treatments should be used whenever possible since they can effectively improve the extraction process and increase yield for low cost. Drying, chopping or grinding, ultrasound and effective microbes are all very useful pre-treatment methods for improving productivity and they can all be used together in a process line.

With the passing of time and development of technology, there will be more tools for the invention of new novel processes that can further improve on the current methods like the MHG. This review only serves to act as an overview and compare the current methods available.

However, the discussion on suitable extraction techniques is still open and new researches are conducted each year. There is still hope in improving the current methodologies and achieving a breakthrough that can shape the future of various industries.

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