

Product Development of Diffusion Solution from *Citrus reticulata* Blanco Essential Oil

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Abstract

The aim of this study was to develop a diffuser solution product from *Citrus reticulata* Blanco essential oil, which was recovered by hydrodistillation. The hydrodistillation process was influenced by factors such as: raw material grinding time (grinding for 40s); raw material/solvent ratio (1/3 g/mL); raw material moisture content (70%), and extraction time (120 min) with an essential oil content of 0.254 mL/g dw. Using gas chromatography-mass spectrometry (GC-MS), 21 chemical components in *Citrus reticulata* essential oil were identified, of which the main compound was limonene, accounting for 94.64%. Thanks to its biological activities, *Citrus reticulata* essential oil is widely used in the field of developing household care products, including diffuser solutions. The results showed that with the IPM base oil content of 25%, the essential oil content of 40%, and the emulsifier PEG-40 of 20% combined with 96% ethanol at a content of 5%, preserved in a dark glass bottle to bring a product with good sensory scores in terms of uniformity, scent, and volatility. The diffuser solution product created helps to utilize the by-product material from *Citrus reticulata* peel to produce essential oil, contributing to increasing the usage value of the *Citrus reticulata* tree. This study is novel in that it combines hydrodistillation-process selection with the formulation of a passive diffuser solution from *Citrus reticulata* peels by-products, thereby connecting essential-oil recovery with aroma-delivery design in a single product-oriented workflow.

Keywords

Citrus reticulata, Essential Oil, Diffusion Solution, Limonene, Hydrodistillation

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1. INTRODUCTION

Essential oils (EOs) have been known since ancient civilizations. Essential oils are volatile liquid substances naturally found in aromatic plants, especially herbs (Abers et al., 2021; Sadgrove et al., 2022). They represent concentrated aromatic fractions that contribute to the characteristic scent of plant materials. EOs are often used in perfumes, cosmetics, shower gels, soaps, foods and beverages, and household cleaning products (Guzmán and Lucia, 2021; Sharma et al., 2023). EOs are also used to flavor foods and beverages and in aromatherapy to promote well-being, mental relaxation, and personal care (Getachew et al., 2019; Maleš et al., 2022; Tsitlakidou et al., 2023). Currently, essential oils can be recovered from plants using conventional methods (e.g., steam distillation, hydrodistillation, and mechanical pressing) as well as modern methods (e.g., microwave-assisted distillation, ultrasonic-assisted extraction, and supercritical CO₂ extraction) (Sethunga et al., 2022). Each method has its own advantages and limitations; however, hydrodistillation is considered simple, easy to perform, and capable of providing relatively high essential-oil recovery

efficiency (Hamid et al., 2021).

Citrus reticulata Blanco essential oil is a popular essential oil of the Citrus family (Cao et al., 2024). *Citrus reticulata* EOs are extracted mainly from cells containing volatile aromatic compounds, which are found mostly in the peels, a by-product of the fruit, and to a lesser extent in the leaves and flowers (Salvatore et al., 2022). *Citrus reticulata* EO is a light-yellow liquid with the characteristic aroma of fresh *Citrus reticulata* and is insoluble in water. According to many previous studies, the main component of *Citrus reticulata* EOs is limonene (Geraci et al., 2024; Job et al., 2024; Nguyen et al., 2024). It also contains other compounds such as geraniol, neral, geranyl acetate, geraniol, β -geryophyllene, nerol, and neryl acetate, as analyzed by gas chromatography mass spectrometry (GC-MS) method. Limonene is present in the ingredients of many types of fragrances, perfumes, and scented soaps. Besides its aromatic value, limonene also has biological activities such as antibacterial and antioxidant properties (Gupta et al., 2021; Meryem et al., 2023). In addition, limonene is used for metal degreasing in the paint industry, has cleaning applications in

the electronics and printing industries, and acts as a solvent in paint production.

Essential-oil diffuser solutions are products used to disperse natural fragrances into the air. The diffusion process is based on fragrance diffusion through capillary action and evaporation. Essential oil diffusers are known for their combination of base oils, emulsifiers, ethanol, antioxidants, and essential oils (Ngan et al., 2020). Of which, essential oils provide the fragrance. Base oils are neutral oils that combine with essential oils to help dilute the essential oils so that the scent is not too strong and keep the essential oils from evaporating too quickly. Meanwhile, ethanol enters the mixture of oils and essential oils to help dilute the oils and bind them all together so that they can move up the diffuser stick more effectively. Emulsifiers help the essential oils and oils blend with ethanol to keep the essential oils in suspension (Pavoni et al., 2020). Finally, antioxidants work to prevent the oxidation of essential oils and base oils. Using a diffuser solution from essential oils makes the space have a fragrant aroma that has a spiritual therapeutic value, helps reduce stress, creates a feeling of relaxation and comfort, and increases labor productivity. In addition, it also has the effect of eliminating moldy odors, bringing a gentle fragrance to clothes. The diffuser solution is also a decorative accessory to make the space more beautiful.

The objective of this study was to develop a formulation and establish a technological process for the production of a diffusion solution from *Citrus reticulata* EOs, which is produced by distillation. The study investigated the factors affecting the extraction process of mandarin essential oil. After the extraction of the essential oil, the factors affecting the formulation of the diffusion solution were also evaluated through factors such as external inspection, turbidity and volatility. Compared with earlier reports that mainly described the composition or bioactivity of *Citrus reticulata* essential oil, the present work specifically addresses how extraction conditions, base oil, emulsifier, and ethanol jointly affect the formation of a usable diffuser system.

2. EXPERIMENTAL SECTION

2.1 Raw Materials

The raw material used for the experiment was the peel from *Citrus reticulata*, harvested at UKT farm, Loc Ninh district, Binh Phuoc province, Vietnam (11°50'35.700"N 106°35'27.700"E, 67000 Vietnam). *Citrus reticulata* are about 8-10 months old from flowering to harvest. The raw material selected after harvest has a uniform, fresh green color, the inner peel is white, not bruised, moldy, or rotten.

Chemicals used in this study included: Sodium sulfate anhydrous (Na_2SO_4), distilled water, Paraffin oil, Hydrogenated castor oil (PEG-40), 96% ethanol, Polysorbate 20 (Tween 20), Polysorbate 80 (Tween 80), Isopropyl myristate (IPM), Dipropylene glycol (DPG). All were purchased from Nguyen Ba Production and Trading Company Limited (Trung Lang Street, Ward 12, Tan Binh District, Ho Chi Minh City, 70000 Vietnam).

The equipment used is a laboratory hydrodistillation system, moisture balance (Ohaus model MP90, USA), analytical balance (Ohaus model PA214, USA), magnetic stirrer with heating (Velp, AREX-6 Digital PRO, Italy), refractometer (Hanna, HI 96800, Romania), pH meter (Hanna HI5221-02, Romania).

2.2 Hydrodistillation of Essential Oil from *Citrus reticulata* Peels

The process of distilling essential oil from *Citrus reticulata* peels is carried out through many steps to obtain pure essential oil products. The process was carried out according to the study of Job et al. (2024) and slightly adjusted to suit actual condition. First, *Citrus reticulata* peels is selected with a sufficient amount, then washed to remove dirt and impurities. Next, the raw material is ground for a certain period of time to release the essential oil-containing sacs in the plant cells. This mixture is then distilled with water at a ratio of 1:3 – 1:9 (g/mL) in a Clevenger distillation device, maintained at a humidity of 20-70% and distilled for 30-180 (min) at a fixed temperature of 110°C. The steam carrying the essential oil after leaving the device is cooled to condense into a liquid consisting of essential oil and water. This mixture is extracted, collecting the essential oil layer floating on top. Finally, the essential oil is dried with sodium sulfate (Na_2SO_4) to completely remove the remaining water, obtaining pure tangerine peel essential oil, which can be used in cosmetics, pharmaceuticals and foods.

For clarity, the obtained product should be understood as a solvent-free hydrodistilled essential oil fraction rather than an analytically pure single substance. No organic solvent was used during the extraction step itself; n-hexane was employed only later during GC-MS sample preparation. The content of *Citrus reticulata* essential oil is calculated according to Equation (1):

$$Y = \frac{V}{(100 - W_b) \times m} \quad (1)$$

When:

Y : Essential oil content (mL/g dw)

V : Volume of essential oil obtained (mL)

W_b : Moisture content of raw materials (%)

m : Mass vovf raw material (g)

2.3 Technological Process of Developing Diffused Essential Oil Products

Technological process of developing diffused essential oil products from *Citrus reticulata* essential oil is carried out by Ngan et al. (2020) and and slightly adjusted. First, adding emulsifier (PEG – 40, or TWEEN 20, or TWEEN 80), and *Citrus reticulata* essential oil in turn, stirring for 30 minutes or until the mixture is homogeneous to obtain mixture 1. Next, slowly add ethanol to mixture 1, continue stirring for 10 minutes to obtain mixture 2. Then slowly add base oil (IPM, or DPG, or Paraffin) to mixture 2 and continue stirring for 10 minutes to obtain the product.

2.4 Methods of Analysis of Essential Oils

The physical and chemical parameters of the essential oil after collection were analyzed according to Vietnamese standards (TCVN) and were conducted according to previous studies and with slight adjustments. Determine the relative density at 20°C according to TCVN (2010c), the refractive index according to TCVN (2010c), the optical rotation according to TCVN (2010b), the residue on evaporation according to TCVN (2010d), the acid index of the essential oil according to TCVN (2010a), the ester index of the essential oil according to TCVN (2010d), and the saponification index of essential oils according to TCVN (2015).

2.5 GC-MS Analysis

GC-MS using a GC Agilent 6890N coupled with an Inert MS 5973 (USA) was used to analyze the composition of *Citrus reticulata* EOs. First, 25 µL of EO was added to 1.0 mL of n-hexane (Sigma -Aldrich), followed by dehydration by using Na₂SO₄. The head pressure is 9.3 psi. GC-MS was performed such as He carrier gas; injection temperature 250°C; flow rate 1.0 mL/min; injection volume 1.0 µL; division 1:100; oven temperature progression 50°C for 2 min, increased to 80°C at 2°C/min, then to 150°C at 5°C/min, to 200°C at 10°C/min and finally maintained at 300°C at 20°C/min for 5 min.

Compound assignments in the present study were made from GC-MS data obtained under the stated operating conditions and should be regarded as tentative identifications. Retention -index confirmation with an n-alkane series and authentic reference standards was not included in this work and should be incorporated in future analyses, especially for minor constituents.

2.6 Method for Assessing the Volatility of Diffusion Solutions

This method is performed according to the research of (Ngan et al., 2020). First, drop 2 g of sample solution onto a petri dish and monitor the evaporation time. Weigh the diffusion solution sample before and after leaving it at room temperature for 1, 2 and 3 h. The volatility of the sample is the change in mass before and after the survey time.

Accordingly, the volatility test should be interpreted as a comparative gravimetric screening of formulation-level mass loss rather than as a direct measurement of vapor-phase composition. Future studies should combine gravimetric monitoring with headspace GC-based analysis to better describe compound-specific fragrance release.

2.7 Data Analysis

The experiments were run three times to ensure consistent results, with data presented as mean ± standard deviation (SD) to show the average and spread. A p-value below 0.05 was used to flag significant differences. Calculations and stats were handled using Microsoft Excel 2019 and Origin software. Excel probably covered simple stuff like averages, SD, and t-tests through its Data Analysis Toolpak, while Origin tackled more complex

tasks like t-tests, ANOVA, regression, and visualizations such as error bar or contour plots for optimizing conditions.

The present study used a one-factor-at-a-time experimental design as an initial screening strategy, and the reported values represent triplicate measurements for each investigated condition. Although this approach was sufficient for selecting practical operating windows, multivariate optimization would provide a stronger evaluation of factor interactions in future work.

3. RESULTS AND DISCUSSION

Prior to distillation, the *Citrus reticulata* peel was characterized by a moisture content of 70.284 ± 0.960%, ash content of 0.936 ± 0.051%, cellulose content of 14.376 ± 2.024%, total fat content of 1.350 ± 0.303%, and pectin content of 2.691 ± 0.218%. These basic physicochemical data support the interpretation of extraction behavior, although oil-gland density was not measured in the present study.

Table 1. Physical and Chemical Parameters of *Citrus reticulata* Essential Oil

Essential oil index	Value
Relative density (g/mL)	0.850 ± 0.014
Residue on evaporation (%)	3.978 ± 0.690
pH	4.383 ± 0.001
Refractive index	1.475 ± 0.001
Optical rotation (°)	124.314
Color	Transparent to pale yellow
Aroma	Natural aroma slightly strong
Taste	Spicy and slightly bitter
Acid Index (mg KOH/g)	1.866 ± 0.323
Ester Index (mg KOH/g)	5.337 ± 0.167
Saponification Index (mg KOH/g)	7.053 ± 0.530

3.1 Factors Affecting the *Citrus reticulata* Essential Oil Hydrodistillation Process

During the hydrodistillation of essential oils from *Citrus reticulata* Blanco peel, many technical factors directly affect the content of recovered essential oils. Based on Figure 1, it can be seen that the influencing factors include the time of grinding the raw materials, the ratio of raw materials to water, the extraction time, and the moisture content of the raw materials.

First, the influence of the time of grinding the raw materials is equivalent to the size of the raw materials presented in Figure 1a. The raw materials were ground with a grinder (Philips HR1393, Netherlands) for the surveyed time from 10 - 60 s. The results showed that the higher the grinding time, the higher the content of essential oils obtained (Tischer et al., 2017). Specifically, at 10 s, the content reached 0.171 (mL/g dw) and at 50 s, the content reached 0.242 (mL/g dw). However, after

60 s, the content tended to decrease slightly with 0.238 (mL/g dw). This is explained by the fact that the longer the material is ground, the finer it is crushed, and the more the essential oil-containing cells are broken, making it easier for the essential oil to escape and come into contact with water vapor (Tran et al., 2019). In addition, the larger contact surface area helps enhance the diffusion of essential oils. On the contrary, whole or chopped materials still have intact cell structures, causing many essential oil sacs to not be broken, limiting recovery efficiency (Dao et al., 2020). However, it should be noted that if ground too finely, some essential oil may be adsorbed onto the surface of the plant residue or washed away with the condensate, leading to loss. Therefore, a moderate grinding time of 50 s is optimally chosen.

Table 2. Chemical Composition of *Citrus reticulata* Blanco Essential Oil

Retention time (min)	Components	Content (%)
7.797	(1R)-2,6,6-Trimethylbicyclo[3.1.1]hept-2-ene	0.58
9.756	Bicyclo[3.1.0]hexan, 4-methylene-1-(1-methylethyl)	0.21
9.946	Bicyclo[3.1.1]heptene, 6,6-dimethyl-2-methylene-(1S)	0.58
10.793	β -Myrcene	1.85
11.560	Octanal	0.19
13.102	Limonene	94.64
17.739	Linalool	0.32
18.071	Nonanal (CAS) n-Nonanal	0.11
19.001	2-Cyclohexen-1-ol, 1-methyl-4-(1-methylethenyl)-, trans-	0.04
19.688	cis-(1)-2-Epoxy-p-menth-8-ene	0.28
19.974	7-Oxabicyclo[4.1.0]heptane, 1-methyl-4-(1-methylethenyl)	0.23
23.079	α -Terpineol	0.12
23.744	Decanal (CAS) n-Decanal	0.16
24.182	trans-Carveol	0.08
25.152	Carvone	0.14
29.391	2-Cyclohexen-1-ol, 1-methyl-4-(1-methylethenyl)-, trans-	0.10
28.063	Carveol	0.12
29.391	cis-p-mentha-1(7),8-dien-2-ol	0.14
29.924	(2R,4R)-p-Mentha-6,8-diene, 2-hydroperoxide	0.03
36.760	aR-Turmerone	0.04
38.329	Benzyl benzoate	0.03

The effect of the ratio of raw materials: water on efficiency hydrodistillation is shown in Figure 1b. Figure 1b clearly shows that the essential oil recovery efficiency is highest when the raw

material-to-water ratio is 1/4 (0.247 mL/g dw) and lowest at the ratio of 1/9 (0.220 mL/g dw). This can be explained by the fact that when heating the mixture of pureed *Citrus reticulata* peel and water, water will penetrate the cell membrane and penetrate into the cells containing essential oil, causing them to swell, at some point the essential oil sacs will break and release the essential oil. On the contrary, when the ratio of water and raw material is small, meaning that the amount of water used is small, it will not be enough to dissolve the amount of colloid on the cell membrane, reducing the rate of water vapor penetration and the diffusion of essential oil (Dao et al., 2020). This is the reason why the extraction efficiency of essential oils at a ratio of 1/3 is lower than that of 1/4 (g/mL).

Based on Figure 1c, it can be seen that with each different moisture content of raw materials, different essential oil contents will be produced. The essential oil content will decrease as the moisture content in the *Citrus reticulata* peel gradually decreases from 70% (fresh raw materials) to only 20% moisture content when dried. According to Figure 1c, the essential oil content obtained at 70% moisture content is the highest (0.22 mL/g dw) and the lowest is at 20% moisture content (0.175 mL/g dw). This can be explained that when the raw materials are kept fresh, the essential oil content in the *Citrus reticulata* peel is completely preserved and when the *Citrus reticulata* peel is dried, in addition to the moisture content in the *Citrus reticulata* peel, it also contains essential oil bags. When the *Citrus reticulata* peel is dried, the essential oil bags in the peel evaporate (Wang et al., 2022). This significantly reduces the efficiency of recovering essential oil from the *Citrus reticulata* essential oil hydrodistillation method. In addition, with a raw material moisture content of 70%, the essential oil content obtained is almost equivalent to that of raw material moisture content of 50% and 60% (0.210 and 0.220 mL/g dw). However, to obtain a high essential oil content without having to dry the raw material, a raw material moisture content of 70% (fresh raw material) is chosen as the appropriate moisture content for the following experiments.

In addition, the longer the extraction time, the more the essential oil content obtained increases as shown in Figure 1d (Ngo et al., 2020). Especially from the time starting from the first drop of essential oil falling, that is, from 0 min to 30 min, the essential oil content obtained increases rapidly from 0 mL/g dw to 0.22 mL/g dw. This can be explained because during this time, the water has reached the boiling temperature and the *Citrus reticulata* peel cells have swollen to the maximum and burst, releasing essential oil particles, causing the essential oil content obtained to increase dramatically in the first 30 min of hydrodistillation. But from 30 min to 150 min (0.254 mL/g dw), the essential oil content obtained began to increase slowly until 150 min when it stopped increasing. This is because in the first 30 min, the essential oil sacs in the peel were released and recovered almost completely by hydrodistillation, so after 30 min, the essential oil content in the peel was no longer much, leading to the distilled essential oil content being insignificant. Finally, from 150 to 180 min

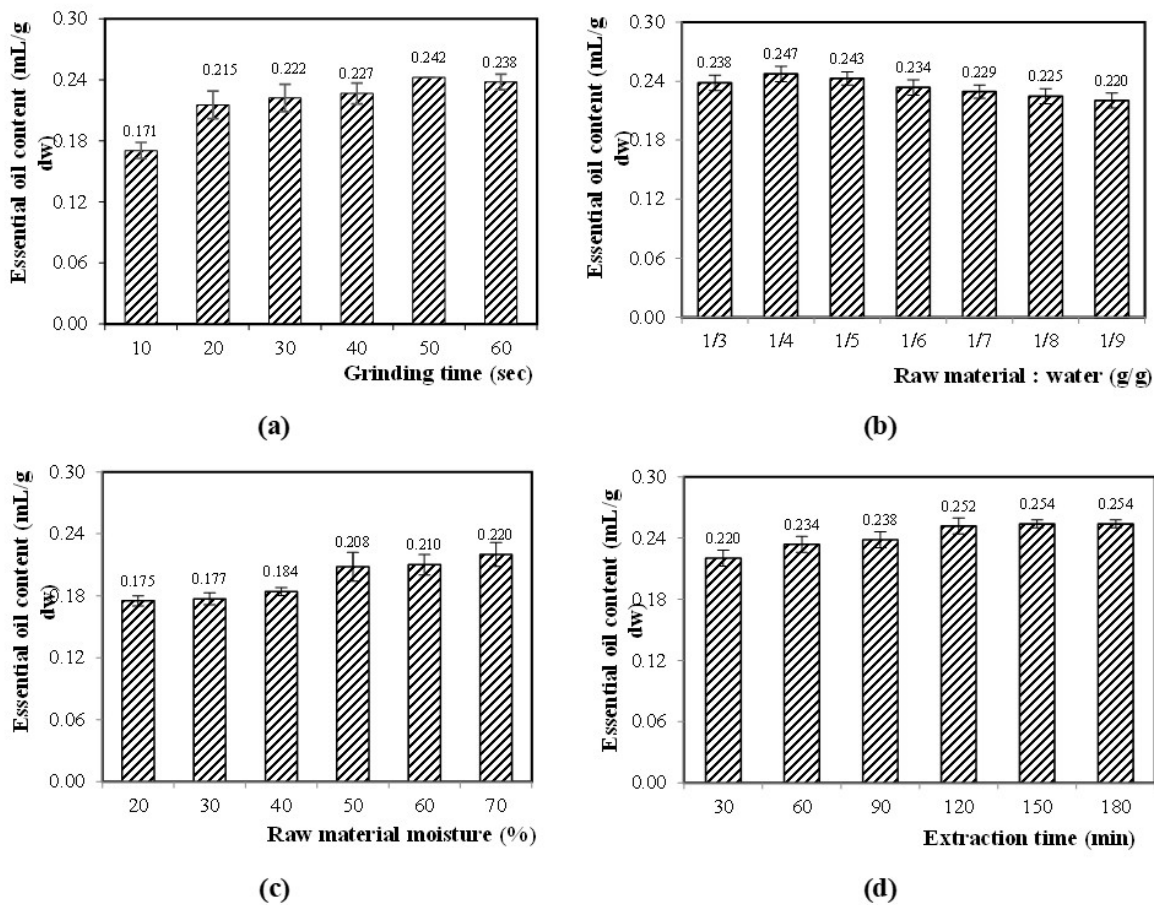


Figure 1. Factors Affecting the Distillation Process of *Citrus reticulata* Essential Oil (a) Grinding Time; (b) Raw Material to Water Ratio; (c) Moisture; (d) Extraction Time

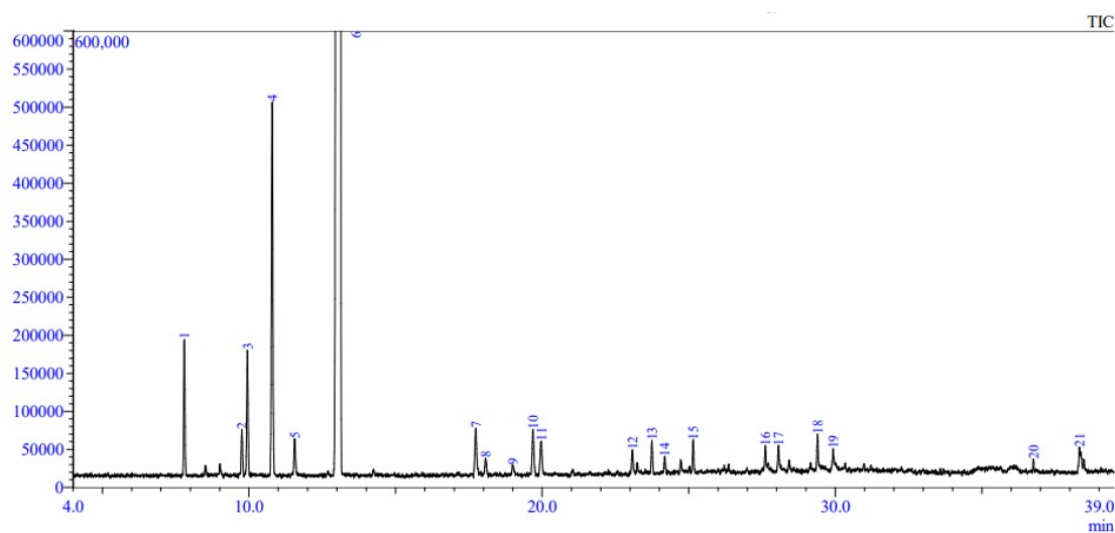


Figure 2. Chromatographic Diagram of *Citrus reticulata* Blanco Essential Oil

(0.254 mL/g dw), the essential oil content obtained no longer increased, proving that the essential oil content in the *Citrus reticulata* peel had been completely distilled. If the experiment continues, the essential oil content obtained will likely decrease due to the sensitivity of the essential oil to temperature, light and long-term heat exposure. From this, it can be seen that with a raw material distillation time of 120 min (0.252 mL/g dw), the essential oil content obtained is almost equivalent to the raw material extraction time of 150 min and 180 min. Therefore, to obtain a high essential oil content while saving extraction time, 120 min was chosen as the appropriate extraction time.

3.2 Physicochemical Parameters and Chemical Composition of *Citrus reticulata* Essential Oil

Table 1 presents the physicochemical parameters of the essential oil was obtained from the above survey parameters. The results show that the density of *Citrus reticulata* essential oil is 0.85 ± 0.014 g/mL, which is similar to the result in India (Bhuyan et al., 2015), which is 0.84 g/mL. Regarding the non-volatile components, the residue on evaporation in *Citrus reticulata* essential oil is $3.978 \pm 0.690\%$. The remaining non-volatile residues in the essential oil are derivatives of terpenes containing oxygen such as fatty acids and esters. There is no significant difference when compared with the international standard (International Organization for Standardization, 2012). For the pH index of essential oil in this study is 4.383 ± 0.001 , showing that the essential oil is slightly acidic, under the significant influence of limonene, which is the largest component in mandarin essential oil. For the refractive index of *Citrus reticulata* essential oil is 1.475, which is lower when compared with the refractive index of essential oil grown in India is 1.4788 (Bhuyan et al., 2015). For the optical rotation of mandarin essential oil is 124.314, which depends on the experimental conditions, equipment and method of determination, storage conditions of essential oil, nature of essential oil, etc.

These physicochemical indices are useful quality- characterization parameters for the hydrodistilled oil, but they should not be interpreted as proof of absolute chemical purity. Instead, they indicate that the recovered oil met the expected characteristics of *Citrus reticulata* essential oil obtained without organic-solvent extraction.

The essential oil sample after collection was determined for chemical composition by GC/MS method. The results on the chromatogram had different retention times, corresponding to this, the obtained essential oil sample contained 21 components, corresponding to 21 compounds shown in the chromatogram of *Citrus reticulata* essential oil shown in Figure 2 and Table 2. Based on Table 2, the GC-MS analysis results of *Citrus reticulata* essential oil show that there are 21 compounds accounting for 99.99%, with 97.86% being monoterpene hydrocarbons and 2.13% being oxygenated monoterpene hydrocarbons, and 0.01% being unidentified substances. Of which, there are 2 main compounds including limonene (94.64%), β -myrcene (1.85%). The limonene content in this study was

higher when comparing the results of this analysis with the study of (Nguyen et al., 2024) with limonene (80.7%), much higher than 77.63% in the study of Job et al. (2024), and 85.7% of Oliveira et al. (2023), while the study of Cao et al. (2024), the limonene content only accounted for 62.35% to 76.72% for fresh and dried materials and of (Geraci et al., 2024) accounted for $70.74 \pm 2.91\%$ to $86.80 \pm 3.38\%$ depending on the regional conditions of the plant.

Because limonene accounted for 94.64% of the total chromatographic area, the volatility and sensory behavior of the diffuser system are expected to be governed largely by a limonene-rich phase. Nevertheless, the minor oxygenated monoterpenes and trace oxidation-related compounds detected in the profile may still contribute to aroma complexity, perceived persistence, and storage-related changes.

3.3 Factors Affecting the Production Process of Diffusion Solution from *Citrus reticulata* Essential Oil

3.3.1 Effect of Base Oil Type and Base Oil Content

The effects of 3 base oils including paraffin oil, isopropyl myristate (IPM), dipropylene glycol (DPG) on the external inspection of the diffusion solution are presented in Table 3. The results show that when using isopropyl myristate (IPM) with a content of 25%, the best results are obtained in terms of turbidity and uniformity of the solution. When the IPM content is reduced to 15% and 20%, the solution becomes cloudy and separates, the oil floats on the surface of the solution. Therefore, the higher the IPM content, the lower the turbidity of the solution. For dipropylene glycol (DPG) and paraffin oil, at all 3 contents (15%, 20%, and 25%), there is a clear phenomenon of turbidity and separation in the solution. This phenomenon may be due to the inappropriate base oil, leading to an unstable system, turbidity and separation (Tian et al., 2022). On the other hand, when considering the volatility factor, the samples were measured for volatility at 1 h, 2 h, and 3 h in Figure 3. The IPM content increased from 15-25%, the product volatility decreased from 46.30% to 38.54% after 3 hours (Figure 3c). Similarly, for paraffin (Figure 3a) and DPG (Figure 3b), the volatility decreased after 3 h to 45.95-41.81% and 45.50-39.22%, respectively. However, the base oils had clear differences in turbidity and uniformity. When using DPG and paraffin, the product volatility was higher than IPM but made the solution cloudy and separated. On the other hand, when using IPM at 25% content, the volatility was lowest (38.54%) but gave good results in terms of transparency and uniformity in the solution. Therefore, IPM base oil with 25% content was chosen to investigate the following factors. From a formulation perspective, the superior performance of IPM is consistent with its better lipophilic compatibility with the limonene-rich essential oil phase. In the presence of PEG-40 and ethanol, this compatibility likely improves miscibility and optical clarity while the relatively low volatility of IPM helps moderate the overall evaporation rate of the diffuser solution.

Table 3. Effect of Base Oil on Turbidity and Homogeneity

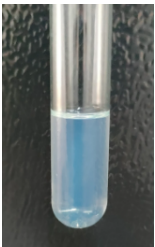
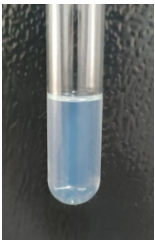
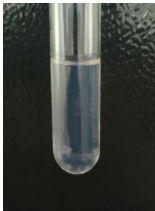
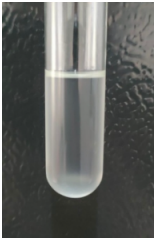
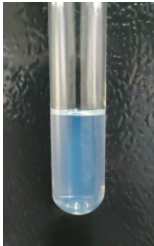
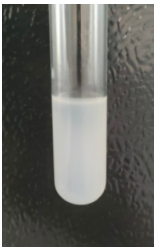
Content	Turbidity and Homogeneity of Base Oil Type		
	Paraffin	DPG	IPM
15%			
External inspection	Cloudy, separates at the bottom	Cloudy, separates at the bottom	Cloudy, separates on the surface of the solution
20%			
External inspection	Cloudy, separates at the bottom	Cloudy, separates at the bottom	Slightly cloudy, uniform
25%			
External inspection	Slightly cloudy, oil layer separates at the bottom	Cloudy, oil layer separates at the bottom	Transparent, homogeneous

Table 4. Effect of *Citrus reticulata* Essential Oil Content on Turbidity and Homogeneity

Essential oil content (%)	35	40	45	50
Turbidity and homogeneity				
External inspection	Opaque, separates into two separate layers	Transparent, uniform	Slightly opaque, separates into two separate layers	Opaque white, separates into two separate layers

3.3.2 Effect of *Citrus reticulata* Essential Oil Content

The effect of *Citrus reticulata* essential oil content on turbidity, uniformity and volatility is shown in Table 4 and Figure

4 shows that the *Citrus reticulata* essential oil content is pro-

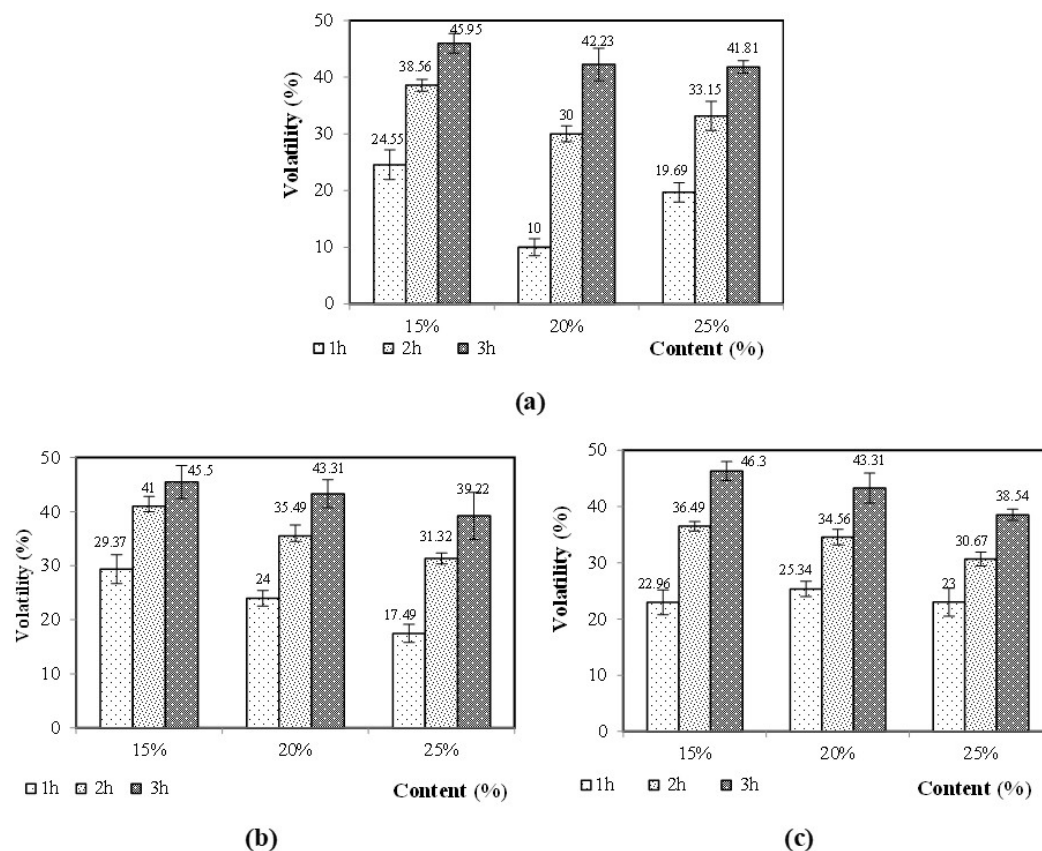


Figure 3. Effect of Base Oil on the Volatility of Diffusion Solution a) Paraffin Oil; b) DPG; c) IPM

portional to the turbidity of the product. In addition, using *Citrus reticulata* essential oil at too high a concentration also affects the product's odor when it causes an overly strong odor when used. In addition, the *Citrus reticulata* essential oil content at 35% has the highest volatility after 3 h of 45.77% and the lowest with 50% essential oil content of 40.30%. Meanwhile, the volatility after 3 h of the essential oil content at 40% and 45% is 43.07% and 42.29% respectively. However, considering all aspects of the product's appearance, the essential oil content at 45% gives a homogeneous, transparent diffusion solution. Although 45% essential oil produced acceptable transparency under the screened condition shown in Table 4, the final formulation used for product development was fixed at 40% essential oil because it provided a better balance among appearance, odor acceptability, and volatility in the overall system. This observation also supports the view that increasing the loading of a limonene-dominant oil can intensify odor but at the same time makes phase stabilization more difficult.

3.3.3 Effect of Emulsifier Type and Emulsifier Content

The results of the survey of turbidity, homogeneity and volatility of the product when using different types of emulsifiers are presented in Table 5 and Figure 5, showing that TWEEN 20

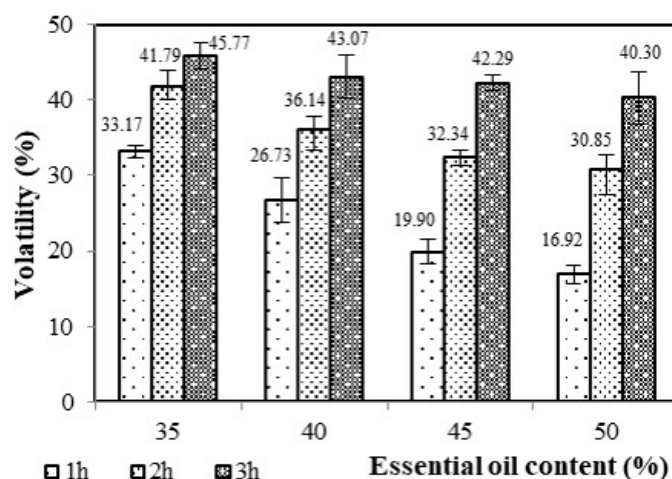
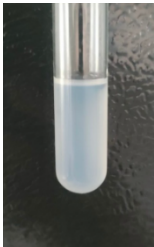
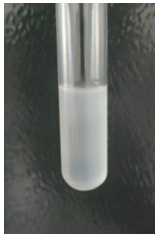
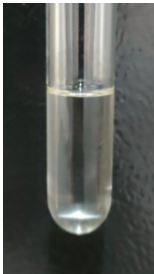
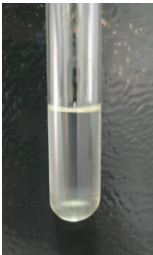


Figure 4. Effect of *Citrus reticulata* Essential Oil Content on the Volatility of Diffusion Solution

with all 3 concentrations (15, 20, 25%) has turbidity from low to high, along with TWEEN 80 with concentrations of 15 and

Table 5. Effect of Emulsifier Type and Emulsifier Content on Turbidity and Homogeneity

Content (%)	Turbidity and homogeneity of emulsifier type		
	PEG-40	TWEEN 20	TWEEN 80
15%			
External inspection	Opaque, separation, light citrus scent	Opaque, no separation, light fragrance, light emulsifier smell	Opaque, separation, light fragrance, light emulsifier smell
20%			
External inspection	Clear, homogeneous, light citrus scent	Slightly opaque, no separation, light fragrance, strong emulsifier smell	Opaque, no separation, light fragrance, strong emulsifier smell
25%			
External inspection	Clear, homogeneous, light citrus scent	Slightly opaque, no separation, light fragrance, very strong emulsifier scent	Clear, homogeneous, light fragrance, very strong emulsifier scent

20% also gives a cloudy solution. In contrast, PEG-40 (20, 25%) and TWEEN 80 (25%) create a clear, homogeneous and stable solution. However, TWEEN 80 is an emulsifier with a characteristic odor when used at high concentrations, creating an unpleasant scent for the product. Unlike the other 2 substances, the PEG-40 emulsifier has a characteristic odor but is lighter and more pleasant, creating a pleasant scent for the product with all 3 concentrations as above. This phenomenon of turbidity and separation may be due to the use of low emulsifier content or unsuitable emulsion type leading to unstable system. Besides, another cause may be due to insufficient ethanol concentration to dissolve the solution. The emulsifier content increased from 15-25% of TWEEN 80, the volatility in 3 h

decreased from 43.7-32.24% (Figure 5a), similar to TWEEN 20 and PEG-40, respectively 43.34-33.62% (Figure 5b) and 44.96-30.11% (Figure 5c). The reason is that TWEEN 80, TWEEN 20 and PEG-40 have viscosities of 425 mPa.s, 400 mPa.s and 1500 mPa.s, the higher the viscosity, the lower the volatility of the product. The volatility of PEG-40 at 20% concentration was 41.38%, which gave higher results than when using 25% concentration of 30.11%. Therefore, PEG-40 at 20% concentration was chosen as the emulsifier for the diffusion product preparation process. The behavior observed here also illustrates that emulsifier selection affects more than only phase appearance. Because PEG-40 has a substantially higher viscosity than Tween 20 and Tween 80, increasing its concentration

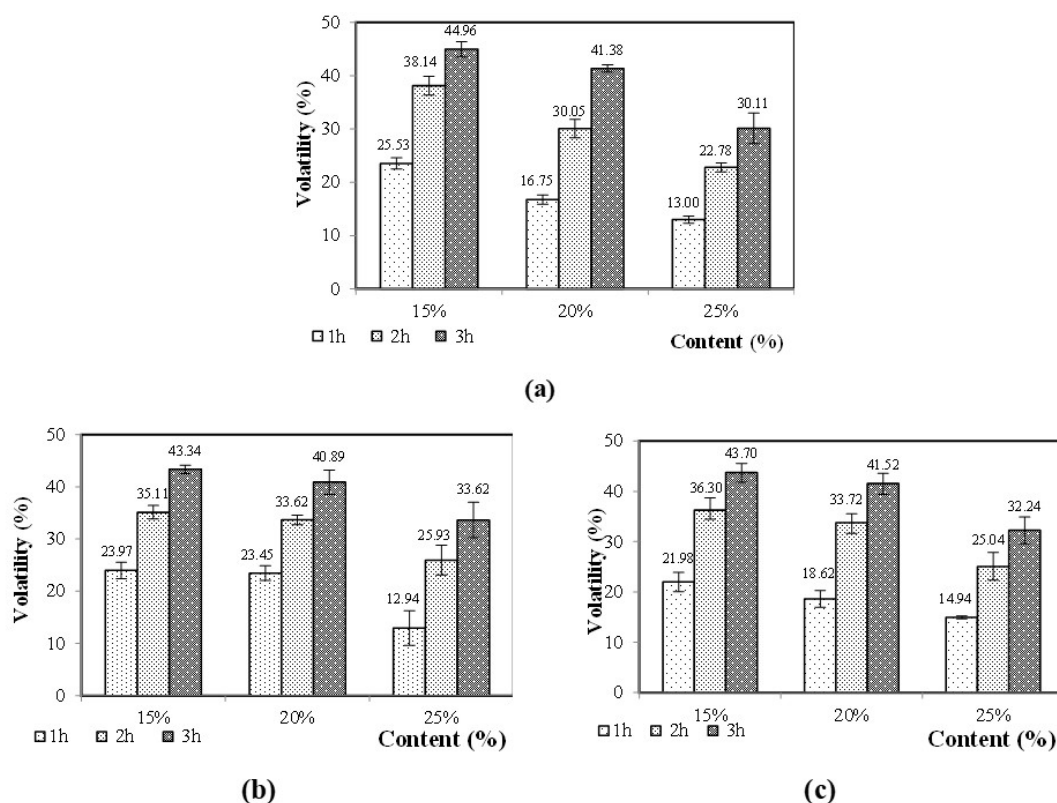


Figure 5. Effect of Emulsifier Content on the Volatility of Diffusion Solution a) PEG-40; b) TWEEN 20; c) TWEEN 80

can reduce mass transfer and slow evaporation; however, an excessively high emulsifier content may also suppress fragrance release. Therefore, the chosen 20% PEG-40 level represents a compromise between transparency, odor quality, and controlled volatilization.

In an additional storage observation over 4 weeks, the optimized diffuser solution remained visually stable without obvious phase separation or turbidity. Dark packaging, especially amber glass, reduced color change more effectively than light-colored packaging and was therefore considered more suitable for minimizing light-induced deterioration during storage at ambient conditions.

4. CONCLUSIONS

This study identified suitable conditions for extracting *Citrus reticulata* Blanco essential oil, with limonene as the main active ingredient at a relatively high content. In addition, the study also provides physicochemical parameters of the essential oil. The results show that relative density reaches 0.850 g/mL, the residue on evaporation accounts for 3.978%, pH 4.383, the refractive index reaches 1.475, and the optical rotation reaches 124.314°. The acid, ester and saponification values of the essential oil account for: 1.866; 5.337; 7.053 (mg KOH/g), respectively. The obtained *Citrus reticulata* essential

oil is the basis for testing to create a diffused solution product from tangerine essential oil for daily use. Through research results, it has been shown that with the IPM base oil content of 25%, the essential oil content of 40%, and the emulsifier PEG-40 of 20%, the product is preserved in a dark glass bottle, combined with 96% ethanol with a content of 5% to create a diffused solution product from tangerine essential oil. Beyond the practical product obtained, the present results also have broader implications for the design of aroma-delivery systems based on terpene-rich natural oils. Specifically, the study shows that release behavior and physical stability must be tuned simultaneously through the combined selection of carrier oil, emulsifier, volatile co-solvent, and light-protective packaging. The main limitations of the present work are the absence of retention-index confirmation for GC-MS identifications, the lack of headspace compositional analysis during evaporation, and the absence of a systematic ethanol-gradient study; these points should be addressed in future investigations.

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