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Evaluation of the quality of pomelo (*Citrus maxima*) essential oil products during storage

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Abstract

The main disadvantage of the pomelo essential oil storage process is the change in colour and composition over time, which significantly affects the quality of essential oils and limits their usability. Thus, this study aimed to determine the effect of packaging type and storage conditions (temperature, time) on the quality of pomelo essential oil products. Pomelo essential oil was preserved in 3 types of packaging (brown glass, white glass, and plastic) at two temperatures of 30°C and 15°C for eight weeks. The parameters used to evaluate the quality of essential oil samples include density, colour index, acid value, chemical compositions, and bioactive activities. The optimal storage conditions of pomelo essential oil were determined as follows: storing in brown glass jars at 30°C for eight weeks. The results showed that after eight weeks, the pomelo essential oil samples were stored in brown glass jars at room temperature (30°C), still retaining their characteristic odour, unchanged colour, density index as 0.856 (g/mL), acid index as 0.748 mg KOH/g, IC_{50} value as 96.271 (mg/mL), limonene content as 75.99%, indicating that the above storage condition would maintain quality and improve the commercialisation of pomelo essential oil products in the Vietnamese market.

1. Introduction

Essential oils are volatile, aromatic liquids obtained from plants through distillation and extraction (Ríos, 2016). Essential oils are usually extracted using several advanced technologies, such as supercritical fluid extraction, solvent-free microwave extraction and conventional methods, such as water, steam, and solvent extraction (Aziz *et al*., 2018). Pomelo is a plant belonging to the genus *Citrus*. Therefore, the main component of pomelo essential oil is limonene (over 90%), which is quite similar to other essential oils of trees in the genus *Citrus* such as orange (89.7-94.7%), lemon (83.9-85.9%), kumquat (74.11%). In addition, pomelo essential oil also contains substances such as a-pinene, linalool, geraniol and citral (Dosoky and Setzer, 2018).

Pomelo essential oil has been shown to have biological activities such as anti-inflammatory, antifungal, and antioxidant. Furthermore, pomelo essential oil is widely used in the treatment of diseases such as cancer, lowering blood lipids, reducing stress, repelling insects and preserving food (Visakh *et al*., 2022). Depending on the conditions and purposes of use, pomelo essential oil is usually preserved in different types of packaging (glass, plastic). In addition, to limit the quality change, pomelo essential oil is also stored in low-temperature conditions (from 5-20°C) and away from direct light (Riquelme *et al*., 2017).

Most previous studies only focused on considering the change in chemical compositions of essential oil at different temperatures without evaluating the effect of types of packages. This caused some disadvantages in preservation as well as maintenance quality. The use of packaging could prolong the preservation of essential oils through their ability to avoid light, oxygen, and water from the surrounding environment. This study contributes to solving the above shortcomings by finding the appropriate packaging and storage conditions (time, temperature) to help maintain the quality of pomelo essential oils.

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2. Materials and methods

2.1 Materials

Pomelo peels *(Citrus maxima)* were obtained from Tan Uyen district, Binh Duong province. Pomelo peels were preliminarily processed, chopped and stored at a temperature of 25°C. The initial moisture content of pomelo peels was 74% (w/w).

2.2 Process of distillation and storage of pomelo essential oils

Pomelo peels, after being pureed, were distilled in a pilot-scale steam distillation machine (25 kg/batch). Pomelo peels (25 kg) was distilled at 110°C for 90 minutes. After distillation, pomelo peel essential oil was anhydrous (using Na_2SO_4) and stored in the refrigerator for 14 days before conducting storage experiments.

The essential oil (240 mL) was divided into three different types of packaging (plastic, brown glass, and white glass). Each type of packaging included 40 bottles (2 mL/1 bottle) which were stored at two temperatures: room temperature (30°C) and refrigerator (15°C). All types of packaging were stored for eight weeks (85% humidity) to evaluate the chemical composition and physicochemical parameters of the essential oils inside.

2.3 Measurement of physicochemical properties

2.3.1 Sensory and color assessment

Pomelo essential oil was conducted for a sensory evaluation (smell, colour observation). The colour of pomelo peel essential oil was measured with a Lab colourimeter (Konica Minolta, Chroma Meter CR-400).

2.3.2 Evaluation of density and acid index

The density of pomelo peel essential oils was evaluated according to Vandegehuchte *et al*. (2013). The density of pomelo peel essential oils was calculated by Equation (1) (Vandegehuchte and Steppe, 2013).

$$d = \frac{m2 - m}{m1 - m} \quad (1)$$

Where d: density of pomelo essential oils (g/mL), m: mass of pycnometer (g), m1: mass of pycnometer and water (g) and m2: volume of pycnometer and essential oil (g).

The acidity index of pomelo peel essential oils was evaluated according to Kawashima *et al*. (2000). The acidity index of pomelo peel essential oil was calculated by Equation (2) (Kawashima, 2000).

$$Acidity\ acid = \frac{5.61 \times C \times V}{P} \quad (2)$$

Where V: volume of 0.1 M KOH used (mL), P: mass of pomelo essential oil to be tested (g), 5.61 is the molar mass of the KOH solution and C: concentration of KOH standard solution (mol/L)

2.3.3 Determination of antioxidant activity

The antioxidant activity of the pomelo essential oil was evaluated according to Dawidowicz *et al*. (2012). The percentage of free radical inhibitory was determined by Equation (3) (Dawidowicz *et al*., 2012)

$$IC\% = \left(\frac{OD_C - OD_t}{OD_C}\right) \times 100\% \quad (3)$$

Where ODc: optical density of control sample, ODt: optical density of test sample and IC%: the percentage of free radical inhibitor DPPH.

2.3.4 Determination of lab index

A Konica Minolta colourimeter (Konica Minolta, Chroma Meter CR-400) was used to determine essential oil colour indexes. 10 mL of pomelo essential oil samples were placed in cuvettes, and the values of L, a, and b were determined. The value of E was determined via Equation (4).

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2\ (\Delta b^*)^2} \quad (4)$$

Where L: colour transition from lighter to darker, a: colour change from red (+) to green (-) and b: colour change from yellow (+) to blue (-)

2.3.5 Determination of chemical compositions of pomelo essential oils

Pomelo essential oils before and after storage were identified in chemical composition using gas chromatography-mass spectrometry (GC-MS). The procedure was 30 µL of pomelo essential oil mixed with 1.0 mL of n-hexane. A machine Agilent 6890 N GC was paired with the MS 5973 probe. The column head pressure was set at 9.31 psi. The device was operated with the following specific parameters: the flow rate of helium as a carrier gas was 1 mL/min, the split ratio was 1/100, and the sample injection volume was 1 µL at 270°C. The oven temperature was initially kept at 50°C for 2 mins, then increased to 2°C/min until 80°C was reached, increased to 5°C/min until 150°C was reached, and then increased to 10°C/min until 200°C and 20°C/min until 300°C in 5 mins.

2.4 Statistical analysis

Each experiment was repeated three times. Microsoft Office Excel software (version 2016) was used to analyze the obtained data. The statistical software Statgraphics Centurion Version 20 (Statpoint

Technologies, Inc., USA) was utilized to perform a number of statistical tests with a significance of 0.05.

3. Results and discussion

3.1 Physicochemical properties and biological activities of pomelo essential oil

Pomelo essential oil, after distillation, was analyzed by GC-MS (Gas Chromatography-Mass Spectrophotometry). The chemical composition of essential oils is shown in Table 1.

The main chemical components in pomelo essential oil included d-limonene (90.084%), β-myrcene (1.571%), o-cymene (1.446%), α-pinene (1.342%). In addition, pomelo essential oil also contained α-terpineol (0.707%), caryophyllene (0.589%) and linalool (0.417%). This result was similar to the results of some other studies conducted on species of *Citrus*, in which limonene was the main component and accounted for the highest percentage. The results obtained in this study were quite similar to previous studies by Li *et al*. (2022), with limonene accounting for 87.86% of pomelo essential oil. Another study by Deng *et al*. (2020) also analyzed limonene as the main component, accounting for 93.33%. According to Uysal *et al*. (2011), the limonene content in pomelo essential oil reached 88.6% when using the steam distillation method to extract essential oil.

Table 2 shows the initial physicochemical parameters of pomelo essential oil before storage. Regarding sensory properties (colour, odour, density), the essential oil was colourless, had a cool aroma typical of pomelo peel, a density of 0.859 g/mL, acid index of 0.749 mgKOH/g. This result was similar to the study of Subramanian *et al.* (2014) studied pomelo essential oil with a density and acid index of 0.853 g/mL and 1.275 mg KOH/g, respectively. . The IC_{50} value in this study was 96.271 mg/mL, which was relatively low compared to Ozkan *et al*. (2011), with an IC_{50} value of 85.13 mg/mL.

Table 2. Physicochemical parameters of pomelo essential oil (before storage).

No.	Physicochemical criteria	Description
1	Color (sensory)	Liquid, transparent solution
2	Odor	Fragrant, characteristic of pomelo essential.
3	Density (g/mL)	0.859± 0.002
4	Acid index (mg KOH/g)	0.749±0.002
5	Lab index	L= 51.47 a= 2.40 b= -3.26
6	IC_{50} value (mg/mL)	96.271

3.2 Color change of pomelo essential oil during storage

Figure 1 shows the change of ΔE value under different storage conditions. At both room and refrigerator temperatures, the L-value had little change from the original colour, indicating that the brightness had little or no change, which the naked eye cannot detect. However, the L-value at room temperature was a difference in the direction of darkening (decreasing L-value) when compared to storage in the refrigerator. In this experiment, the temperature and light could change the essential oil in the direction of gradually darkening colour, but not significantly (p>0.05). According to ΔE colour data in all 3 types of brown, white and plastic glass packaging, with brown glass packaging, there was little colour change, almost no change compared to the colour of initial samples before storage. Therefore, essential oils stored in brown glass jars were the most suitable, in accordance with TCVN 9650:2016 (Essential oils – general principles of packaging, packaging and storage conditions).

Table 1. Chemical composition of pomelo essential oil before storage.

No	Retention time (mins)	Compound name	Molecular formula	Ratio (%)
1	8.08	α-pinene	$C_{10}H_{16}$	1.342
2	9.932	4-methylene	$C_{10}H_{16}$	0.305
3	10.908	β-Myrcene	$C_{10}H_{16}$	1.571
4	11.506	α-Phellandrene	$C_{10}H_{16}$	0.379
5	12.659	o-Cymene	$C_{10}H_{14}$	1.446
6	12.987	Limonene	$C_{10}H_{16}$	90.084
7	14.676	γ-Terpinene	$C_{10}H_{16}$	0.542
8	17.331	Linalool	$C_{10}H_{18}O$	0.417
9	19.108	Limonene oxide	$C_{10}H_{16}O$	0.152
10	21.627	2-Cyclohexen-1-one	$C_9H_{14}O$	0.653
11	21.859	α-Terpineol	$C_{10}H_{18}O$	0.707
12	23.07	trans-Carveol	$C_{10}H_{16}O$	0.271
13	23.524	2-Cyclohexen-1-ol	$C_{10}H_{16}O$	0.593
14	24.041	(-)-Carvone	$C_{10}H_{14}O$	0.652
15	25.097	2,6-Octadienal	$C_{10}H_{16}O$	0.065
16	29.902	Caryophyllene	$C_{15}H_{24}$	0.589

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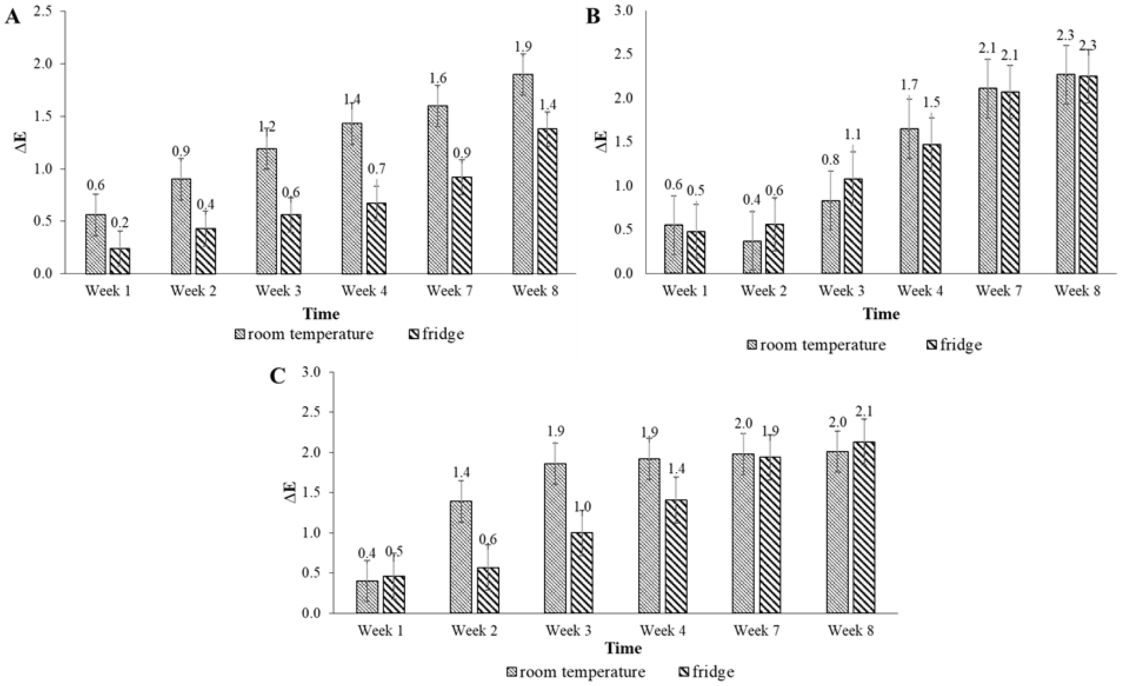

Figure 1. ΔE value of pomelo essential oil in different packaging. A: dark glass jar, B: clear glass jar, C: plastic jar.

3.3 Changes in physicochemical properties of pomelo essential oil during storage

Tables 3, 4 and 5 show the changes in physicochemical properties of pomelo essential oil stored at two temperatures (room and refrigerator) in three types of packaging (plastic bottles, white glass and brown glass) after eight weeks of storage. During storage, physicochemical properties such as odour, taste and density did not change significantly ($p>0.05$), and colour had little change. However, in the brown glass jar, the pomelo essential oil had little difference in colour and taste at both temperature levels. The results showed that brown glass packaging was suitable for essential oil preservation. The storage at 15°C and 30°C did not seem to differ in physicochemical properties among samples significantly. Therefore, storing at room temperature was more convenient for production and utilization and did not consume fuel and storage equipment.

3.4 Changes in antioxidant activity of pomelo essential oil before and after storage

According to the experiment results from section 3.3, pomelo essential oil was optimally stored at room temperature and in brown glass jars. Then, the above sample of pomelo essential oil was analyzed for antioxidant activity after eight weeks of storage (Table 6). Before storage, pomelo essential oil had an IC_{50} value of 96.271 mg/mL. When the concentration of essential oils increased, the DPPH free radical inhibitory percentage gradually increased in both samples before and after eight weeks of storage. These results were in line with the research of Li *et al*. (2022) on the antioxidant activity of pomelo essential oils (IC value as 144 mg/mL).

3.5 Changes in chemical compositions of pomelo essential oil during storage

After storage time, pomelo essential oil samples were best stored in brown glass jars at room temperature. Therefore, these samples were analyzed for chemical composition (Figure 2) at 2 weeks, 4 weeks, 6 weeks and 8 weeks. The components in Table 7 showed that the chemical components in pomelo essential oil have changed but not significantly. The main components of pomelo essential oil, such as limonene, beta-myrcene, and terpinene, were retained during storage, but the content has slightly changed. However, after storage time, pomelo essential oil still retained a limonene content of 75.99%, which was consistent with quality standards for pomelo essential oil according to TCVN 11423:2016. Nevertheless, the number of chemical compounds gradually decreased, and their structure changed. This could be due to the fact that unsuitable storage temperatures led to some compounds absorbing light and changing their chemical structure (Turek and Stintzing, 2013). During storage, limonene content changed; however, limonene was still a compound that accounted for a large proportion of the composition of pomelo essential oils after preservation. This proved that the antioxidant activity of essential oils after storage has almost no significant change compared to essential oils before storage (Table 6). The results showed that pomelo essential oil, after being stored for eight weeks at 30°C and in a brown glass jar, still maintained its stable quality compared to the original essential oil and conformed to Vietnamese standards TCVN-11423:2016.

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Table 3. Changes of physicochemical properties of essential oils in dark glass jar.

Targets physicochemical	Before preservation	After storage	
		Room temperature	Fridge
Color (ΔE)	0	1.90	1.38
Odor	The characteristic aroma of pomelo peel.	Retains the distinctive pomelo scent. More fragrant.	Keep the original scent.
Density (g/mL)	0.859±0.002[a]	0.856±0.001[a]	0.853±0.001[a]
Acid index (mg KOH/g)	0.749±0.002[a]	0.751±0.001[a]	0.748±0.002[aB]

Values are presented as mean±SD. Values with different superscripts within the same row are statistically significantly different.

Table 4. Changes of physicochemical properties of essential oils in clear glass jar.

Targets physicochemical	Before preservation	After storage	
		Room temperature	Fridge
Color (ΔE)	0	2.27	2.25
Odor	The characteristic aroma of pomelo peel.	Retains the distinctive pomelo scent.	Keep the original smell.
Density (g/mL)	0.859±0.002[a]	0.853±0.001[a]	0.852±0.001[a]
Acid index (mg KOH/g)	0.749±0.002[a]	0.752±0.002[a]	0.754±0.002[a]

Values are presented as mean±SD. Values with different superscripts within the same row are statistically significantly different.

Table 5. Change of physicochemical properties of essential oils in plastic jar.

Targets physicochemical	Before preservation	After storage	
		Room temperature	Fridge
Color (ΔE)	0	2.01	2.13
Odor	The characteristic aroma of pomelo peel.	Retains the distinctive pomelo scent.	Keep the original smell.
Density (g/mL)	0.859±0.002[a]	0.895±0.001[a]	0.862±0.001[a]
Acid index (mg KOH/g)	0.749±0.002[a]	0.750±0.002[a]	0.749±0.001[a]

Values are presented as mean±SD. Values with different superscripts within the same row are statistically significantly different.

Table 6. IC_{50} values of pomelo peel essential oil and Vitamin C.

Sample	IC_{50}
Pomelo essential oil before storage	IC_{50} = 96.271 (mg/mL)
Pomelo essential oil after storage	IC_{50} = 95.268 (mg/mL)
Vitamin C	IC_{50} = 2.541 (µg/mL)

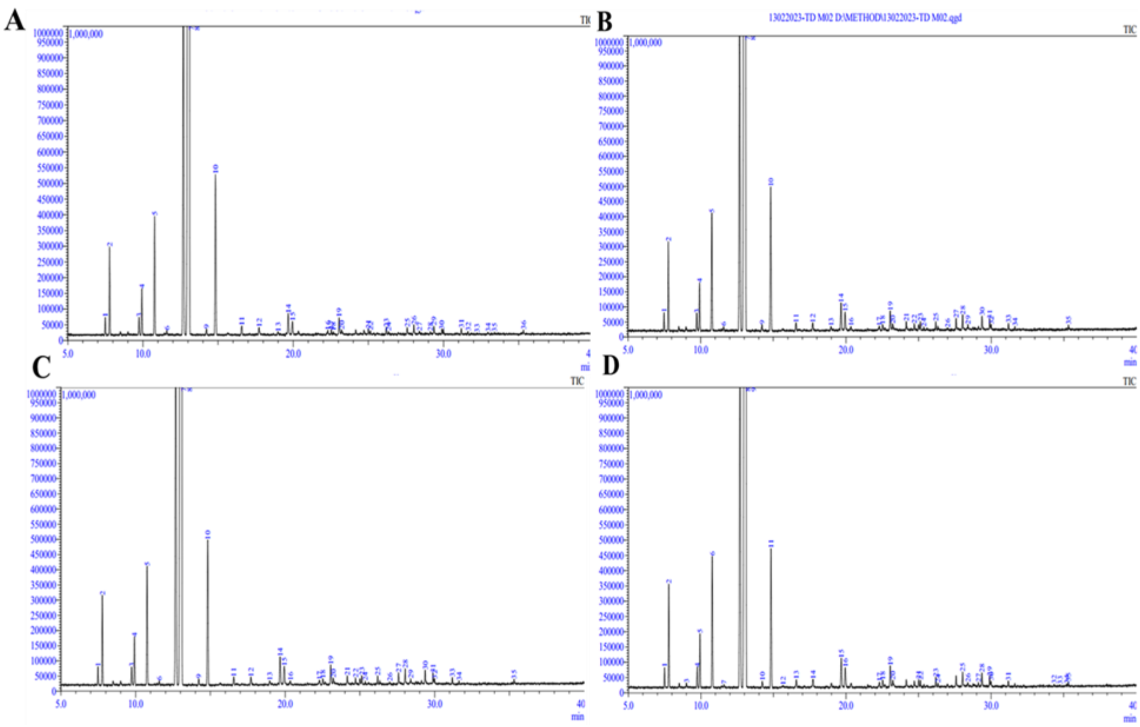

Figure 2. Chromatography of pomelo essential oil preserved in dark glass jar. A: 2 weeks, B: 4 weeks, C: 6 weeks and D: 8 weeks.

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Table 7. Chemical composition comparison at storage weeks dark glass jar.

No	Compound name	Ratio of substances (%)			
		2 weeks	4 weeks	6 weeks	8 weeks
1	Bicyclo[3.1.0]hex-2-ene	0.41	0.43	-	0.42
2	Alpha-pinene	2.07	2.05	-	2.20
3	Bicyclo[3.1.0]hexane	0.39	0.41	0.40	0.43
4	Bicyclo[3.1.1]heptane	1.08	1.12	1.12	1.14
5	Beta-Myrcene	2.75	2.72	2.85	2.79
6	1-Phellandrene	0.10	0.09	0.09	-
7	Benzene	8.32	8.89	8.40	9.68
8	L-Limonene	77.54	75.98	76.74	75.99
9	1,3,6-Octatriene,3,7-dimethyl-(Z)	-	0.13	0.15	0.13
10	Gamma-terpinene	3.75	3.33	3.84	2.98
11	Linalool	0.16	0.20	0.18	0.17
12	3-Cyclohexen-1-ol	0.09	0.09	0.11	0.10
13	(-)-Carvone	0.07	0.21	0.10	0.13
14	Citral	0.16	0.19	-	-
15	Carveol	0.16	0.20	0.18	0.29
16	Apha Sinensal	0.10	0.24	0.14	0.20
17	trans-Caryophyllene	0.14	0.15	0.17	0.11
18	Caryophyllene oxide	0.09	0.11	0.07	0.09

4. Conclusion

Brown glass packaging was chosen as the appropriate packaging for storing pomelo essential oil. After preserving pomelo essential oils in brown jars at room temperature (30°C) for eight weeks, the quality of essential oils remained stable with antioxidant value as IC_{50} = 96.256 mg/mL, density as 0.856±0.002 g/mL, acid index as 0.748±0.002 mg KOH/g. Pomelo essential oils stored in brown glass bottles did not seem to change the main components of the essential oils. The chemical compounds of pomelo essential oil after preservation were recorded as follows: limonene (75.99%), alpha-pinene (2.20%), gamma-terpinene (2.98%), benzene (9.68%), beta-myrcene (2.79%). Pomelo essential oils, after preservation, still ensured quality criteria according to TCVN 9650:2016 (Essential oils-General Packaging Principles). The above results would help preserve pomelo essential oil conveniently and quickly and improve the commercialisation of pomelo essential oil products in the Vietnamese market.

Conflict of interest

The authors declare there is no conflict of interest.

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