JURNAL REKAYASA PROSES
VOLUME 19, NUMBER 1, 2025, 42–49

RESEARCH ARTICLE

Optimization of D-limonene and phenolic compounds extraction from local Indonesian orange peel using ultrasound-assisted extraction

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Received 08 November 2024; revised 16 February 2025; accepted 19 February 2025

OBJECTIVES Malang Regency is one of the orange plantation centers in East Java, Indonesia, and has been named Indonesia's orange agrotourism area. Orange peel waste in Indonesia has not been utilized, even though orange peel contains valuable compounds, such as D-limonene and polyphenols. METHODS . To date, studies on the extraction of D-limonene and total phenolic compounds (TPC) from Baby Java Pacitan orange (Citrus sinensis L.) and Keprok Batu 55 oranges (Citrus reticulata Blanco) has not been investigated. In this work, several factors affecting the extraction of D-limonene and total phenolic compounds from local Indonesian orange peels with ultrasonic assistance were investigated and optimized. RESULTS The results showed that drying using a vacuum oven and agitation significantly increased the yield of D-limonene and TPC. The optimum condition for extracting D-limonene was a solid-solvent ratio (SSR) of 1:10 with an ethanol concentration of 96% for 40 minutes. Meanwhile, the optimum condition for extracting polyphenol compounds was a solid-solvent ratio of 1:10 with an ethanol concentration of 96% for 80 minutes. CONCLUSIONS Under optimum conditions, Baby Java Pacitan orange peel produced D-limonene of 130.5 mg/g dry biomass, which was 2.8 times higher than Keprok Batu 55 orange peel. Meanwhile, the TPC for Baby Java Pacitan orange peel and Keprok Batu 55 orange peel were 46.1 mgGAE/g dry biomass and 43.9 mgGAE/g dry biomass, respectively.

KEYWORDS Baby java Pacitan orange; D-limonene; keprok Batu 55 orange; orange peel waste; phenolic compounds

1. INTRODUCTION

Orange is one of the horticultural crops that are widely cultivated in Indonesia. Malang Regency is one of the orange plantation centers in East Java and has been named Indonesia's citrus agritourism area (Agrofarm 2019). In 2019, orange production reached 135,489.4 tons in Indonesia, reflecting a 2% increase from the previous year. There are several varieties of local orange cultivated in Malang, including Siam Madu (*Citrus nobilis*), Baby Java Pacitan (*Citrus sinensis* L.), Rimau Gerga Lebong (*Citrus nobilis* L. Var RGL) and Keprok Batu 55 (*Citrus reticulata* Blanco) (Agrofarm 2019; Jawa Pos Radar Malang 2022).

All orange varieties cultivated in Malang are sold directly to traders, MSMEs (micro, small and medium enterprises), and industry. Its use is limited to consumption as a fruit or in squeezed drinks and juice. Meanwhile, the peel of the fruit is discarded (personal interview). However, orange peel, which makes up 50-60% of the total fruit weight, contains valuable components, such as D-limonene and phenolic compounds (Ozturk et al. 2019).

D-limonene, also known as (+)-limonene or R-(+)-limonene, is one of the secondary metabolites found in orange peel and belongs to the terpene group. The content of D-limonene in the essential oil of orange peel ranges from 68% to 98%, while its yield ranges from 0.3 to 32.9 mg/g dry biomass depending on the variety of the orange and extraction method (Prasad et al. 2016; Siddiqui et al. 2022; Zuckerman 1995). It is widely used as flavor and fragrance additives in food, beverage, perfume, pharmaceuticals, and cosmetic industry. It is also used as a preservative, green solvent, and antioxidant. Besides, it has analgesic, anticancer, anti-inflammatory, neuroprotective, cardioprotective, gastroprotective, antimicrobial effects, etc. Its valuable role in preventing several chronic and degenerative diseases has received extensive attention from researchers (Anandakumar et al. 2021; Lin et al. 2024). The global market value of D-limonene reached USD 312.1 million in 2022 and is expected to grow at a compound annual growth rate (CAGR) of 6.0% from 2023 to 2030 (Grand View Research 2022).

Besides terpenes, other secondary metabolites found in the orange peel are phenolic compounds (polyphenols), such

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as phenolic acids (caffeic acid, chlorogenic acid, ferulic acid, gallic acid, and sinapinic acid) and flavonoids (hesperidin and naringin). These compounds have significant biological activities, such as anti-inflammatory, anti-cancer, antimicrobial, and antioxidant (Lü et al. 2016). The global market value of polyphenols was USD 1.68 billion in 2022 and is expected to grow at a CAGR of 7.4% from 2023 to 2030. The demand for the product is attributed to its various health benefits including anticancer, antidiabetic, antiaging, etc (Grand View Research 2022).

Several methods have been explored to isolate D-limonene and phenolic compounds from orange peel, including conventional methods (cold pressing, distillation, steam explosion, soxhlet extraction) and advanced methods (high-pressure high-temperature extraction (HPHTE), ultrasound-assisted extraction (UAE), supercritical fluid extraction (SFE), microwave-assisted extraction (MAE), enzyme assisted hydro distillation (EAHD). Conventional processes are simple but require a long extraction time and high energy consumption. Advanced methods have been shown to increase productivity, reduce time and energy consumption, and achieve the highest possible yield. The utilization of HPHTE may damage heat-labile target compounds such as D-limonene (Siddiqui et al. 2022). Compared to SFE and MAE, UAE requires lower equipment cost. SFE has a complex system configuration and requires high capital investment. Uneven heating and/or overheating of samples in MAE may reduce the extraction efficiency or cause thermal degradation of target compounds. Although EAHD has been shown to increase yield, however, enzymes are expensive and require control of medium pH and temperature for optimal enzyme action. To overcome some limitations of other advanced methods, UAE is a promising technique for isolating targeted components from biomass because it has relatively low equipment cost compared to SFE and MAE, and can be operated at low temperatures, thereby reducing thermal degradation of target compounds (Picot-Allain et al. 2021).

UAE is an extraction method that utilizes ultrasonic waves. It aids the extraction of target components from the matrix of biomass by breaking cell walls through the formation and rupture of bubbles during expansion and compression cycles, thereby facilitating the diffusion of the solvent into the biomass cells. This phenomenon is known as cavitation. As a result, UAE method requires a relatively shorter time (Lavilla and Bendicho 2017). Xhaxhiu et al. (2013) reported that 30 min of UAE yielded a higher D-limonene content (90%) than 6 h of soxhlet extraction (85%). In recent research related to D-limonene extraction from lime, Khandare et al. (2021) found that UAE reduced the extraction time. D-limonene obtained from 20 min of UAE, 30 min of stirred batch extraction, and 185 min of Soxhlet extraction was 32.9 mg/g, 21.8 mg/g, and 33.78 mg/g, respectively. In research related to the extraction of phenolic compounds from orange peel, Saini et al. (2019) reported that the phenolic content in the extract of Citrus reticulata using 30 min of UAE method (28.30 mg GAE/g) was higher compared to the maceration method (23 mg GAE/g).

Currently, the application of UAE in the extraction of polyphenols from orange peel has been extensively studied (Siddiqui et al. 2022). However, the study on the application of UAE in extracting polyphenol compounds and D-limonene simultaneously from Indonesian local orange peel has not been investigated. The objective of this study is to evaluate the effects of extraction parameters on the yield of D-limonene and phenolic compounds from Indonesian local orange peel using the Ultrasonic-Assisted Extraction (UAE) bath-type method. Two varieties of orange peel were used, namely Baby Java Pacitan orange (*Citrus sinensis* L.) and Keprok Batu 55 orange (*Citrus reticulata* Blanco). The parameters observed were the effect of drying methods, agitation, extraction time, solid-solvent ratio (SSR), and solvent concentration on the yield of D-limonene and total phenolic contents. This research is expected to be a solution for managing and valorizing orange peel waste in Malang, East Java, Indonesia.

2. RESEARCH METHODOLOGY

2.1 Materials

Two different varieties of orange peel (Baby Java Pacitan and Keprok Batu 55) were obtained from Selorejo Village, Malang, East Java. Folin-Ciocalteu reagent and sodium carbonate anhydrous were purchased from Sigma Aldrich (Sternheim, Germany). Ethanol 96% was purchased from a local supplier.

2.2 Raw material and preparation of biomass

The orange peel was washed with water to remove impurities before cutting it into 0.5 cm pieces. Subsequently, it was dried in the oven at 40 °C until it reached a constant weight. The dried orange peel was milled using a blender (Maspion MT 1569, Indonesia) to pass through a 50-mesh screen sieve. Then, the resulting orange peel powder was stored in an airtight container.

2.3 Ultrasound-assisted extraction (UAE)

The UAE system consisted of a round bottom glass reactor that was placed in the ultrasonic bath (Elmasonic S 60 H, Elma Schmidbauer GmbH, Germany) (Figure 1). The ultrasonic bath was operated at a constant frequency (37 kHz) with a total output power of 400 W. The solvent used in this work was ethanol 96%, except for the variable that used different concentrations of ethanol. The temperature of the sample was maintained at 30±5 °C. To highlight the effect of agitation, the extraction was conducted using UAE without agitation and with agitation. The experiments were performed in duplicates.

2.4 Preliminary study

In the preliminary studies, the effect of the drying method and agitation were examined to determine the appropriate pretreatment and extraction method to obtain a high extraction yield of D-limonene and total phenolic compounds (TPC). To simplify the experiments, the observation was conducted using Baby Java Pacitan orange peel. Two different drying methods were observed: a convection oven (Binder ED 53, Germany) and a vacuum oven (Memmert V029, Germany) at 40 °C. To observe the effect of agitation on the extraction yield of D-limonene and TPC, the UAE was performed without agitation and with agitation at 70 rpm. The solvent used was ethanol 96%, with a solid-solvent ratio of 1:10 for 60 min. The

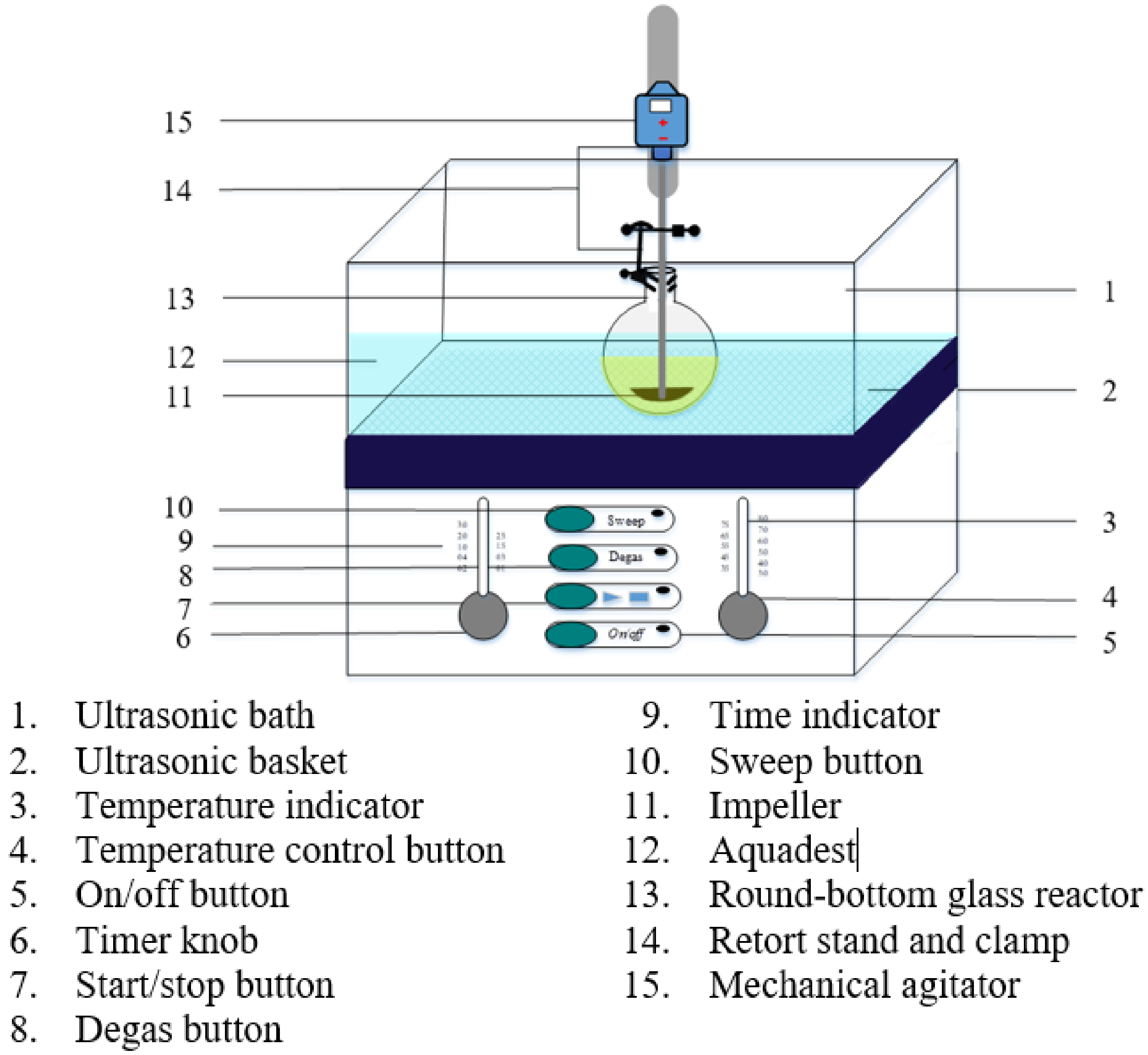

FIGURE 1. Scheme of ultrasound-assisted extraction (UAE) system.

optimum conditions obtained in the preliminary study were then used to optimize the extraction process (Figure 1.

2.5 Optimization of extraction of D-limonene and phenolic content from orange peel

The following parameters were examined for their effect on the extraction yield of D-limonene and TPC: extraction time (20, 40, 60, 80, 100 min), solid-solvent ratio (1:5; 1:10; 1:15), and solvent concentration (70%, 80%, 96% v/v). These variables were tested on the two orange peel varieties used in this study.

2.6 Determination of D-limonene content in the extract

D-limonene content was determined using a Gas Chromatography-Flame Ionization Detector, GC-FID (Shimadzu GC-2014, Japan). The analysis was carried out on a Rtx-Wax column with dimensions of 30 m × 0.32 mm, and 0.25 μm film thickness. The carrier gas used was H2 with a flow rate of 2 mL/min. The oven temperature was 100 °C (hold 0.5 min) to 250 °C at 16 °C/min (hold 10 min). The detector and injection temperature were set at 250 °C and 230 °C, respectively. The make-up gas used was N2 at 52 mL/min with the flow rates of H2 and air were 40 and 400 mL/min, respectively. An appropriate amount of D-limonene standard dissolved and diluted with ethyl acetate to prepare a calibration curve of D-limonene. The determination of D-limonene yield is performed through calculations using a linear regression equation derived from the calibration curve of D-limonene. D-limonene yield is expressed in milligrams of D-limonene per gram of dry biomass (mg/g DW).

2.7 Determination of total phenolic compounds (TPC) in the extract

Total phenolic compounds (TPC) in orange peel extracts were determined using the Folin-Ciocalteu colorimetric method and gallic acid as standard as described by Genwali et al. (2013) with a slight modification. An extract concentration of 1000 ppm was prepared. In a test tube, 0.5 mL of extract solution was added with 2.5 mL of Folin-Ciocalteu reagent (10%) and incubated at room temperature for 2 min. A 2 mL of 7,5% sodium carbonate (Na_2CO_3) was then added, mixed, and incubated at room temperature for 30 min. The absorbance of samples was measured using a UV/VIS spectrophotome-

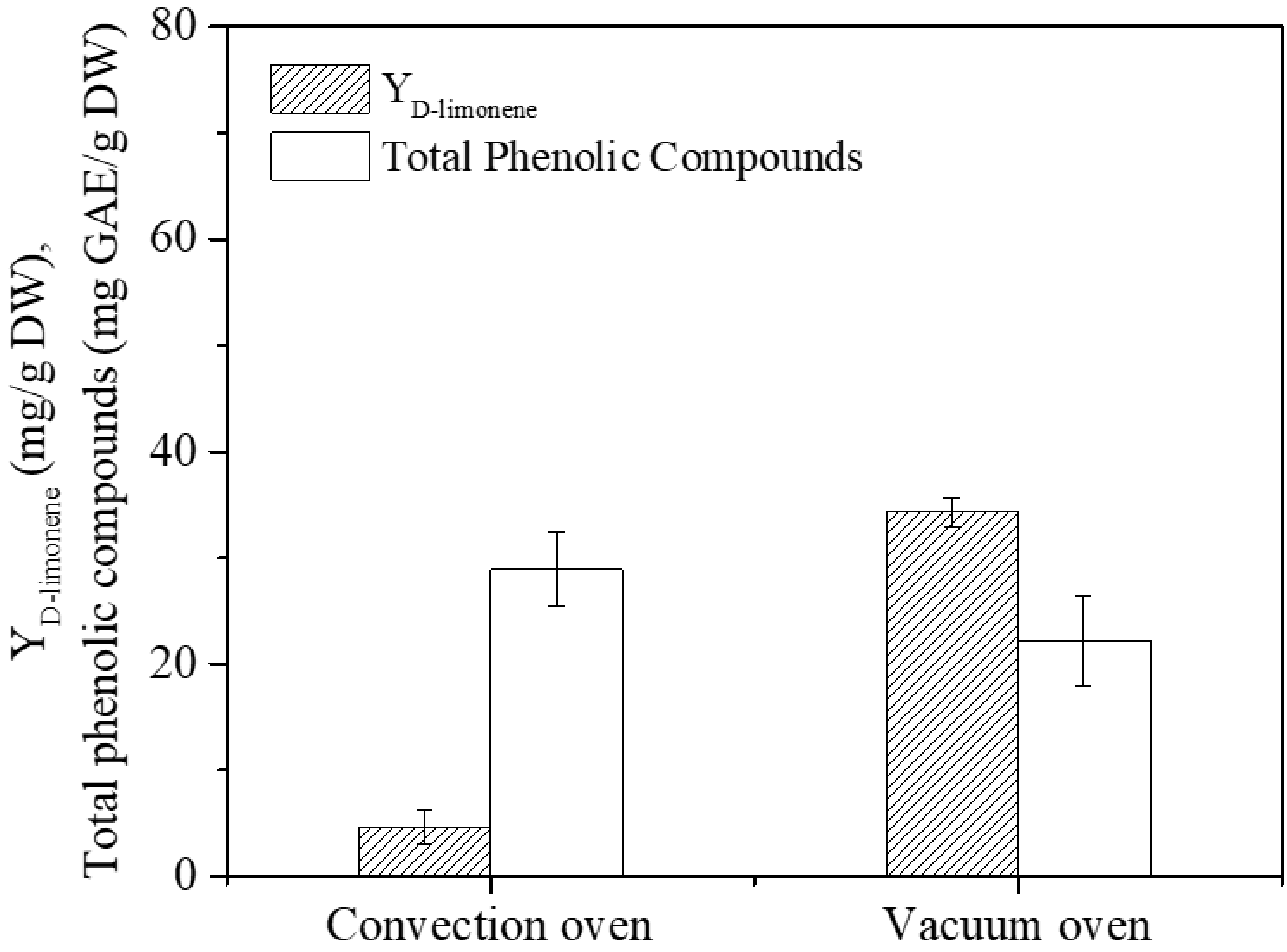

FIGURE 2. Effect of drying method on the yield of D-limonene and total phenolic compounds of orange peel of Baby Java Pacitan and Keprok Batu 55.

ter (Mecasys Optizen Pop, Korea) at 745 nm. The experiments were carried out in duplicates. The total phenolic content of extracts was expressed as mg gallic acid equivalents (GAE) per gram of dry biomass (mg GAE/g DW).

2.8 Statistical analysis

All data were analyzed using Minitab statistical software version 18. Statistical analysis was performed using analysis of variance (ANOVA), and differences between groups were assessed using the least significant difference test (p-values < 0.05 were considered statistically significant).

3. RESULTS AND DISCUSSION

3.1 Effect of drying method

Two methods of drying were investigated, namely using a convection oven and a vacuum oven. The results showed that using a convection oven required 8–9 hours to dry the orange peel until it reached a constant weight. While using a vacuum oven significantly shortened the drying time to 5 h. As depicted in Figure 2, observation of the yield of D-limonene ($Y_{D\text{-limonene}}$) showed that a vacuum oven was more favorable to obtain a high yield of D-limonene. It succeeded in increasing the yield of D-limonene by 7.5 times compared to drying using a convection oven ($p < 0.01$). It indicates that drying method significantly affects the yield of D-limonene. According to Mohapatra (2006), D-limonene is a thermolabile compound that can be degraded at a temperature of 50 °C (Mohapatra 2006). Although the temperature of drying in this study was lower than 50 °C (40 °C), however the longer time of drying might also contribute to the degradation of D-limonene.

Observation on the phenolic content (TPC) showed that drying using a convection oven produced a relatively higher phenolic compound content (1.3 times higher) compared to using a vacuum oven. However, statistical analysis showed that drying method did not significantly affect TPC ($p > 0.05$) (Figure 2). Although the TPC of biomass dried in a convection oven was relatively higher than in a vacuum oven, the drying time was much longer. For process efficiency reasons and a relatively high yield of D-limonene, a vacuum oven was considered more favorable as a pretreatment of orange peel to obtain a relatively high concentration of D-limonene and phenolic compounds. Therefore, in the next experiments, drying using a vacuum oven was used as a pretreatment to remove water content in the biomass.

3.2 Effect of agitation

To optimize the extraction process, the effect of agitation was investigated. The results showed that ultrasonic-assisted extraction equipped with mechanical agitation increased the yield of D-limonene and TPC by 57.0 % ($p < 0.05$) and 31.4 % ($p > 0.05$), respectively, compared to extraction without agitation (Figure 3). It implies that agitation significantly contributed to the extraction of D-limonene but did not significantly affect the extraction of phenolic compounds. In a solid-liquid

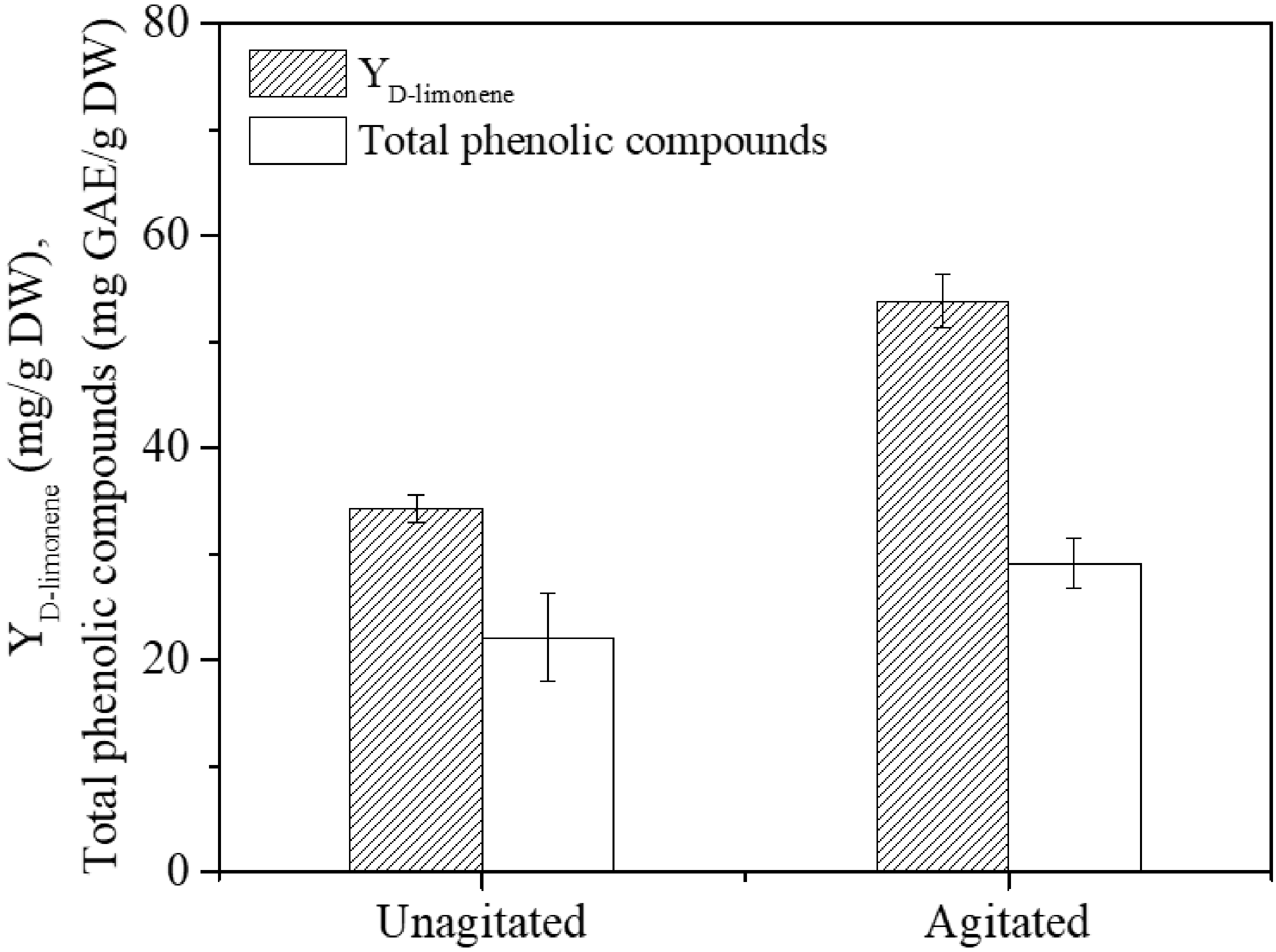

FIGURE 3. Effect of agitation on the yield of D-limonene and total phenolic compounds of orange peel of Baby Java Pacitan and Keprok Batu 55.

system, agitation provides good mixing thereby decreasing external mass transfer resistance that allows the exposure of particle surface to solvent. In the presence of ultrasound, agitation helps to keep the particles in suspended form and assists in intensifying the external mass transfer (Khandare et al. 2021). As a result, the yield of D-limonene and TPC increased.

3.3 Effect of extraction time

By applying agitation, the effect of extraction time on the yield of D-limonene and TPC for both varieties of orange peel was then investigated, and the results are presented in Figure 4. Time of extraction significantly affects the yield of D-limonene and TPC ($p < 0.001$). In general, the longer the extraction time, the higher the yield of D-limonene and TPC obtained. After a certain period of time, the content of D-limonene and phenolic compounds in the extract declined. This may be due to changes in the target compounds due to long-term exposure to ultrasonication (Carreira-Casais et al. 2022). As in the work of Shang et al. (2019), long-term ultrasonic extraction can destroy the molecular chains of polysaccharides and degrade polysaccharide molecules. This phenomenon may also occur in D-limonene and phenolic compounds when long-term extraction is applied, resulting in the degradation of the desired products.

The highest yield of D-limonene ($Y_{D\text{-limonene}}$) was achieved at the extraction time of 40 min for both varieties of orange peel. Under this condition, the orange peel of Baby Java Pacitan generated D-limonene 130.5 mg/g DW or 2.8 times higher than Keprok Batu 55. Prolonging the time of extraction to 60 min caused D-limonene to diminish by 58.7% and 92.6% to 53.8 mg/g DW and 3.5 mg/g DW, respectively, for Baby Java Pacitan and Keprok Batu 55 orange peel. The same trend was also observed in phenolic content; however, it needed a longer time to reach the maximum value. These results confirm that the extraction rate of each component may be different. The highest phenolic compound content for both varieties of orange peel was achieved at the extraction time of 80 min. Under this condition, the TPC for both varieties were not significantly different i.e. 46.1 and 43.9 mg GAE/g DW for Baby Java Pacitan and Keprok Batu 55 orange peel, respectively.

The increase in extracted D-limonene and TPC to a certain value can occur because the cavitation effect of the ultrasound enhanced the swelling, hydration, fragmentation, and pore formation of the plant tissue matrix. As a result, it increases the exposure of the target compounds and the solvent and aids their release into the solvent. The decrease of D-limonene and TPC with prolonged extraction time may be caused by the degradation of the extracted molecule (structural damage) due to prolonged exposure to ultrasonication (Khandare et al. 2021; Kumar et al. 2021).

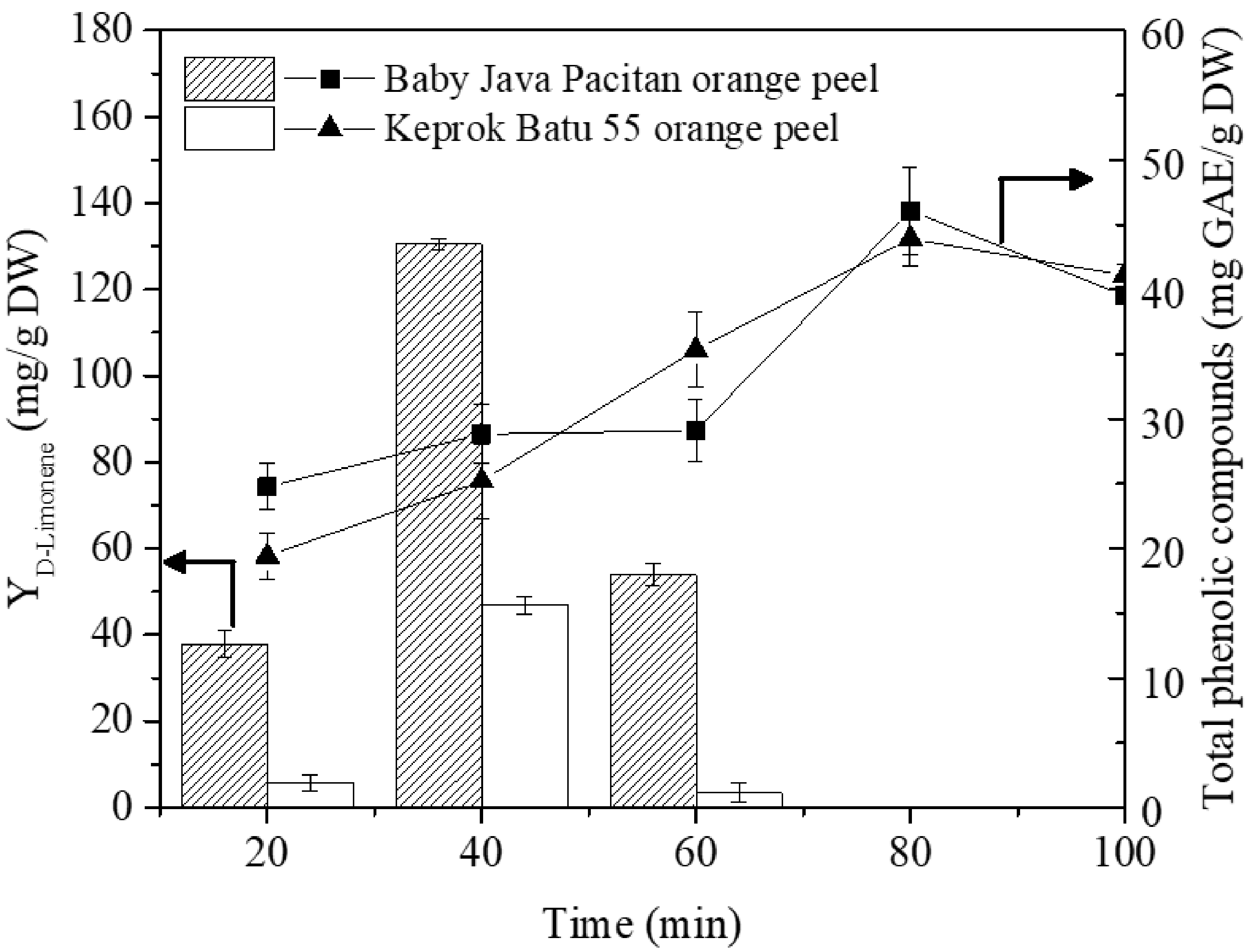

FIGURE 4. Effect of extraction time on the yield of D-limonene and total phenolic compounds of orange peel of Baby Java Pacitan and Keprok Batu 55.

3.4 Effect of solid-solvent ratio (SSR)

The effect of solid-solvent ratio on the extraction of D-limonene and TPC was studied in the range of 1:5 to 1:15. By doing extraction for 40 min with agitation at 70 rpm using EtOH 96%, the yield of D-limonene and TPC was observed. As summarized in Table 1, the ratio of 1:10 w/v resulted in the maximum yield of D-limonene and phenolic content for both orange peel varieties. A lower or higher solid-solvent ratio shows a significant decrease in D-limonene yield and TPC ($p < 0.01$). The sufficient solid-solvent ratio provides better mass transfer of solutes from the particle's surface to the solvent due to a higher driving force in terms of the concentration gradient between the matrix of the solid and the solvent (Khandare et al. 2021). A low amount of solvent (low SSR) caused a decrease in the extraction yield since the viscosity of the solution is high which inhibits the cavitation effect, thereby decreasing fragmentation and pores formation on the plant tissue matrix. A high amount of solvent in the system (high SSR) causes an increased cavitation effect, resulting in the degradation of the target compounds (Kumar et al. 2021).

3.5 Effect of solvent concentration

The concentration of ethanol varied from 70% to 96% to observe its effect on the extraction efficiency of D-limonene and phenolic compounds. The extraction was conducted using SSR 1:10 with agitation at 70 rpm for 40 min. The results are summarized in Table 2. The use of aqueous solvents caused a significant decrease in D-limonene yield ($p < 0.01$). The highest D-limonene and phenolic content was achieved using 96% ethanol for both orange-peel varieties. D-limonene was not detected when using ethanol at concen-

TABLE 1. Effect of solid-solvent ratio on the yield of D-limonene and total phenolic compounds of orange peel of Baby Java Pacitan and Keprok Batu 55.

Solid-solvent ratio	Baby Java Pacitan orange peel		Keprok Batu 55 orange peel	
	Yield D-limonene (mg/g DW)	Total Phenolic Compounds (mgGAE/g DW)	Yield D-limonene (mg/g DW)	Total Phenolic Compounds (mgGAE/g DW)
1:5	1.3±0.3	23.5±3.9	0.2±0.3	21.4±3.1
1:10	130.5±1.3	28.8±2.3	35.0±16.8	25.2±2.9
1:15	0	21.2±1.1	2.1±0.6	15.7±2.3

The obtained data are presented as the mean ± standard deviation (SD) (n = 2). *Significantly different from SSR of 1:10 (*p-values < 0.01)

TABLE 2. Effect of solid-solvent ratio on the yield of D-limonene and total phenolic compounds of orange peel of Baby Java Pacitan and Keprok Batu 55.

Ethanol concentration (%)	Baby Java Pacitan orange peel		Keprok Batu 55 orange peel	
	Yield D-limonene (mg/g DW)	Total Phenolic Compounds (mgGAE/g DW)	Yield D-limonene (mg/g DW)	Total Phenolic Compounds (mgGAE/g DW)
70	0*	22.5±3.0*	0*	10.4±1.2*
80	0*	28.8±3.2	0*	18.9±3.3*
96	130.5±1.3	28.8±2.3	46.9±2.0	25.2±2.9

The obtained data are presented as the mean ± standard deviation (SD) (n = 2). *Significantly different from ethanol concentration of 96% (*p-values < 0.01)

trations of 70% and 80%. This phenomenon may be caused by the presence of water in the solvent. When water is used as the solvent, under the effect of ultrasound, some radicals (reactive species) can be generated. These reactive species can recombine to form other radicals and molecules (Lavilla and Bendicho 2017).

Observation on the relationship between ethanol concentration and phenolic components extracted from orange peel showed a contrary trend to the work of Li et al. (2006). They found that TPC increased with an increase in the ethanol concentration until it reached 85%; after which, the recovery reduced with the increase of concentration. In this work, the highest TPC was reached at the ethanol concentration of 96%. This difference may be caused by differences in the types of phenolic compounds contained in the biomass, which influence the interaction between solute and solvent.

The presence of valuable compounds in orange peel waste opens opportunities for further research on the application of waste or its extracts for food and non-food such as pharmaceuticals and cosmetics. In addition, it is necessary to explore effective ways to prevent the degradation of target compounds due to exposure to heat and/or ultrasonic waves. Study of simultaneous extraction and encapsulation of extracted components is interesting to develop. Feasibility studies on the application of UAE for the extraction of D-limonene or phenolic compounds from orange peel on an industrial scale also need to be conducted. The utilization of ultrasound in the date syrup industry for improving the quantity and quality of syrup extraction (Vilkhu et al. 2008) provides insight into the potential application of UAE on an industrial scale.

4. CONCLUSIONS

In this work, ultrasound-assisted extraction for releasing D-limonene and phenolic compounds (TPC) from Indonesian local orange peel (Baby Java Pacitan and Keprok Batu 55) using ethanol was demonstrated and optimized. The drying method and mechanical agitation had a significant impact on the extraction yield. The use of a vacuum oven and mechanical agitation was more favorable to obtaining a high yield of D-limonene and TPC. The extraction time needed to extract D-limonene was shorter than phenolic compounds. A moderate solid-solvent ratio and high ethanol concentration were more favorable for obtaining high target components. At the optimum extraction conditions, Baby Java Pacitan orange peel contained a higher D-limonene than Keprok Batu 55 but contained almost the same phenolic compounds. In view of the importance and health benefits of target compounds, these findings show the potential of local orange peel waste to derive valuable products that offer an approach towards waste valorization and sustainable waste management encouraging the zero-waste theory.

5. ACKNOWLEDGMENTS

The authors would like to thank the LPPM University of Brawijaya for financially supporting this research through Hibah Penelitian Pemula (HPP) under contract no. 611.39/UN10.C200/2023.

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