

Effect of Extraction Time on the Yield and Color Characteristics of White Pepper (*Piper Nigrum* L.) Oleoresin

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Received: 2025-08-07

Accepted: 2025-08-15

Publication: 2025-08-16

Abstract

This study investigates the effect of extraction time on the yield and color characteristics of white pepper (*Piper nigrum* L.) oleoresin using the microwave-assisted extraction (MAE) method compared to conventional maceration. White pepper powder was extracted with 96% ethanol at four MAE durations (30, 60, 90, and 120 minutes) under a maximum controlled temperature of 50 °C, with maceration for 420 minutes as control. Results showed that MAE at 120 minutes produced the highest yield (10.370%), followed by 90 minutes (9.457%), 30 minutes (8.760%), and 60 minutes (8.323%). Conventional maceration yielded 8.610%, which was lower than MAE at 30 and 90 minutes, indicating higher efficiency of MAE even at shorter durations. Color analysis using CIELAB and RGB systems revealed that MAE at 90–120 minutes yielded brighter oleoresin ($L > 56$) compared to maceration ($L = 53.33$), while chromatic values (a and b) remained stable. Linear regression analysis showed a strong positive correlation between extraction time and yield ($y = 0.1058x$; $R^2 = 0.8819$), indicating that each additional minute increased yield by $\sim 0.1058\%$. The findings suggest that MAE is a time-efficient method that enhances both yield and color quality of white pepper oleoresin, with optimal results obtained at 90–120 minutes.

Keywords: Color Characteristics; Extraction Time; Microwave-Assisted Extraction; Oleoresin

Introduction

The largest pepper production is in Indonesia, with production in 2024 of 62,625 tons and in 2025 of 63,461 tons. Indonesia's pepper production comes from various regions throughout Indonesia. Indonesia is known as a producer of black pepper (Lampung black pepper) produced in Lampung and white pepper (Muntok white pepper) produced in Bangka Belitung Islands Province (Direktorat Jendral Perkebunan, 2024). Indonesian pepper is marketed in the form of various forms and types of products, including pepper seeds, ground pepper, essential oils and oleoresins.

In the world of food industry, one of the methods to separate the main compounds in food ingredients by relying on the difference in solubility of two mutually soluble liquids until an equilibrium phase is reached between the concentration and compounds in the solvent in the material is called the extraction method. Generally, the extraction of chemical compounds in foodstuffs is carried out conventionally which takes quite a long time. Maceration (soaking with solvents) is a conventional method commonly used for the extraction of pepper oleoresin, producing competitive yields and quality according to ISO standards (dark brown color, pepper aroma, thick paste shape) (Budiastra et al., 2020).

Extraction methods play an important role in determining the yield and quality of oleoresins. One of the influential parameters is the length of extraction time, which can affect the release efficiency of active compounds as well as the color characteristics of the final product. The microwave-assisted extraction method is a promising alternative to conventional maceration

because it shortens time, increases mass transfer efficiency, and minimizes thermal damage. Therefore, this study was conducted to examine the effect of varying extraction time using the MAE method on the yield and color characteristics of white pepper oleoresin.

Literature Review

Longer extraction time in maceration (e.g. 4.68-7 h) tends to increase oleoresin yield, but time efficiency is better in ultrasonic method (75 min) with similar yield results (Budiastira et al., 2020). The microwave-assisted extraction method produced higher yields (8.76-10.37%) than conventional maceration (8.61%), although statistically not significantly different. The recommended optimal time is 30 minutes, as it produces high yields with maximum time efficiency. The refractive index and color of oleoresin extracted by microwave were not significantly different from the conventional method, but the chemical composition was significantly different. Microwave extraction extracted 51 chemical components, including 23.03% piperine, higher than conventional maceration (14 components, 6.62% piperine) (Damanik et al., 2022).

The longer the extraction time, the oleoresin yield tends to increase until it reaches an optimum point, after which additional time no longer increases the yield significantly (Ameh et al., 2020). In the extraction of nutmeg and white pepper using ultrasonic method, the longer extraction time (up to 75 minutes) increases the yield, but the time efficiency is better than the conventional method (maceration) which takes up to 7 hours (Budiastira et al., 2021). In the extraction of ginger oleoresin using ethanol and ethyl acetate solvents, the yield increased as the extraction time increased until a certain time (about 60-70 minutes), after which the yield tended to stagnate even though the time was extended (M.S et al., 2020).

In white pepper extraction, the smaller the particle size and the more optimal the extraction time, the higher the yield (Zhang et al., 2022). White pepper oleoresin is widely used in the food and pharmaceutical industries as a flavoring ingredient, natural colorant, antioxidant, and antimicrobial. The main ingredients such as piperine play a role in pungent flavor and biological activity (Aziz et al., 2021a).

The color of the oleoresin (reddish yellow/dark brown) did not differ significantly between maceration and ultrasonic methods, indicating similar quality. A standard color indicates optimal extraction and does not significantly affect yield, but is an indicator of the quality of the final product (Budiastira et al., 2020).

Ethanol is one of the main solvents used in the extraction of pepper (*Piper nigrum*) oleoresin, both for black and white pepper. The use of ethanol is driven by its ability to extract bioactive compounds such as piperine, antioxidants, and volatile components, as well as its status as a food-safe "green solvent" (Aziz et al., 2021b). Green solvents (ethanol, acetone, and their mixtures) effectively extract oleoresins from spices, with acetone being the most effective for colorants, volatile substances, and antioxidants in turmeric, ginger, and coriander, and ethanol for chili and black pepper (Madhusankha et al., 2023). Extraction methods such as maceration, soxhlet, reflux, ultrasound-assisted extraction (UAE), and supercritical fluid extraction (SFE) with ethanol as solvent or co-solvent can affect oleoresin yield and quality. UAE and SFE with ethanol increase efficiency, speed up extraction time, and yield higher piperine than conventional methods (Chaminda et al., 2022).

Piperine has a basic structure of an amide consisting of three main parts: an aromatic ring with a methylenedioxy bridge, a conjugated dienone chain, and a piperidine ring that forms an amide bond (Ezawa et al., 2020). The chemical formula of piperine is C₁₇H₁₉NO₃, with the systematic name (2E,4E)-1-[5-(1,3-benzodioxol-5-yl)-1-oxo-2,4-pentadienyl]piperidine (Mitra et al., 2022).

The methylenedioxy moiety on the aromatic ring and the conjugated chain system play an important role in the biological activity of piperine (Han et al., 2023).

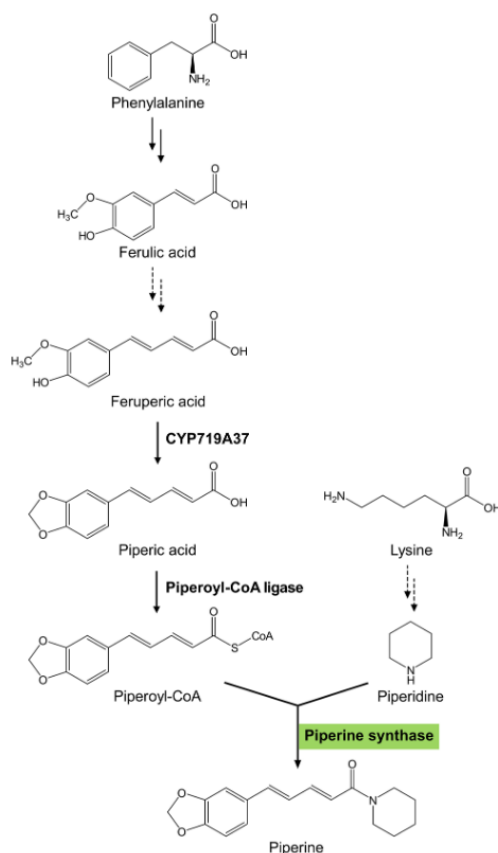


Figure 1. Partly hypothetical pathway of piperine biosynthesis in black pepper fruits (Schnabel et al., 2021)

Research Method

The material used in this study was Lampung Daun Kecil pepper seeds obtained from farmers' gardens in Mendo Barat District, Bangka Belitung. Pepper seeds obtained from farmers are first sorted to remove impurities. The sorted pepper is then measured for moisture content (max. 12%). Pepper seeds that meet the moisture content requirement are then ground into pepper powder using a disk mill, then sieved using a 60-mesh sieve.

White pepper powder is weighed at 200 grams using a digital scale and placed into a 100 mL beaker, then filled with 96% ethanol with a volume of 800 mL (material to solvent ratio 1:4) and subjected to microwave maceration extraction with 4 treatment times (30, 60, 90, and 120 minutes), each treatment repeated 3 times, resulting in 15 experimental units. Extraction was performed at a maximum controlled temperature of 50°C. Temperature control was carried out by measuring the temperature every 5 minutes during the extraction process using a thermocouple, and if the solution temperature exceeded 50°C, the beaker glass was immersed in water at room temperature (29°C) to lower the temperature while manually stirring for approximately 1 minute. The extraction results were then filtered using filter paper, followed by solvent evaporation using a rotary vacuum evaporator until the oleoresin product was obtained. Conventional maceration extraction was performed as a control, by dissolving 200 grams of pepper powder with a 60 mesh size in 800 mL of 96% ethanol in a 1-liter plastic sample bottle. The extraction was conducted for 7 hours at room temperature, with manual stirring every 10 minutes. The extraction results were filtered using filter paper and then evaporated using a rotary vacuum evaporator to obtain the

oleoresin product.

Analysis was performed on oleoresin obtained from microwave maceration and conventional maceration. The analysis included refractive index analysis (BSN 1987), yield, piperine content, color, and oleoresin components. The quality parameters of the oleoresin observed were yield, moisture content, oleoresin constituent content, color, piperine content, and refractive index.

Results and Discussion

Oleoresin Yield of White Pepper (*Piper nigrum* L.)

The oleoresin yield results from extracting white pepper with different times and methods are shown below (Figure 2).

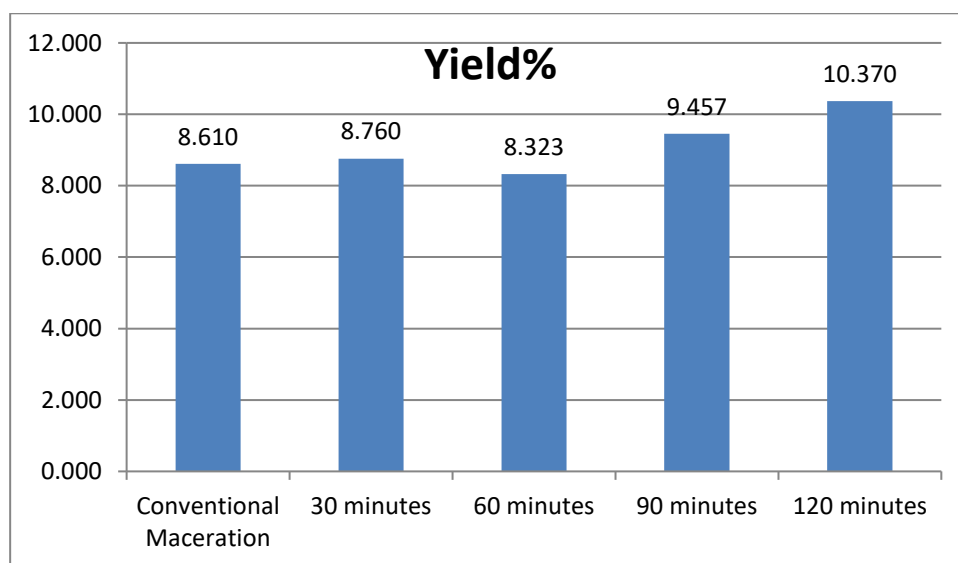


Figure 2. Chart Oleoresin Yield of White Pepper (*Piper nigrum* L.) at Different Extraction Times

It is evident that the microwave extraction method at 120 minutes yielded the highest oleoresin output, reaching 10.370%, followed by extraction at 90 minutes (9.457%), 30 minutes (8.760%), and 60 minutes (8.323%). Meanwhile, the conventional maceration method, despite being conducted over a longer extraction period, produced a yield of 8.610%, which is still lower than the yields obtained through microwave extraction at both 30 and 90 minutes. This indicates that the microwave method is significantly more efficient than maceration, even when applied for a shorter duration.

Scientifically, the increase in oleoresin yield with prolonged microwave extraction time can be explained by the mechanisms of volumetric heating and more effective cellular disruption. Microwaves induce rapid internal heating within plant cells, accelerating the release of oleoresin compounds into the solvent. As extraction time increases, the intensity of cell wall disruption also intensifies, enabling more efficient recovery of oleoresin trapped within the tissue matrix.

However, the observed drop in yield at 60 minutes to 8.323% suggests that fluctuations in extraction efficiency may occur at certain time points. This could be attributed to uneven heat distribution, changes in solvent viscosity, or an imbalance between solvent penetration and active compound diffusion. The overall trend clearly indicates that longer microwave extraction times generally result in higher oleoresin yields, with the optimal yield observed at 120 minutes.

Color Appearance of White Pepper Oleoresin on Paper

The image presented illustrates the color outcomes of white pepper (*Piper nigrum* L.) oleoresin extracted using two different methods: microwave-assisted extraction (at extraction durations of 30, 60, 90, and 120 minutes) and maceration (at 420 minutes of extraction) (Figure 3).

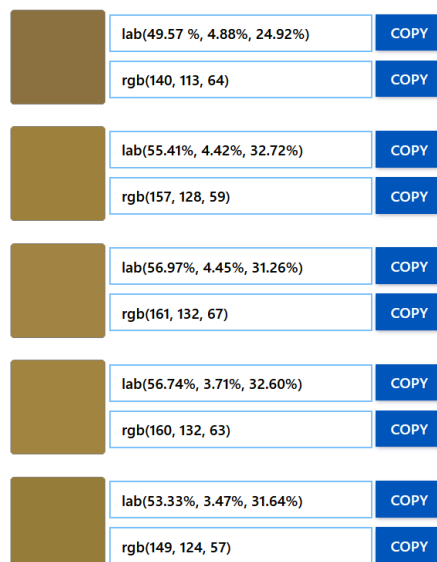


Figure 3. Color Appearance of White Pepper Oleoresin on Paper at Different Extraction Times

Color representations are provided in two formats: the CIELAB (LAB) color space and the RGB (Red-Green-Blue) system, each describing the chromatic characteristics of the extract obtained at each extraction duration.

In the LAB color system, the L parameter represents lightness, a corresponds to the green–red axis, and b corresponds to the blue–yellow axis. In the RGB system, color composition is expressed on a scale from 0 to 255 for each of the red, green, and blue components.

Microwave Extraction (30–120 minutes): An increase in the L value (lightness) was observed from 49.57% (30 minutes) to 56.97% (90 minutes), indicating that the oleoresin color became brighter with prolonged extraction time. After 90 minutes, the L value slightly decreased to 56.74% (120 minutes), suggesting a tendency toward stabilization or even a decline in brightness beyond the optimal extraction time.

The a (green–red) and b (blue–yellow) components exhibited minor fluctuations but remained within ranges that supported the visualization of the characteristic yellowish-brown hue of pepper oleoresin. RGB values confirmed similar color trends, where both red and green components increased over time, indicating a brighter and more homogeneous color profile.

Maceration Extraction (420 minutes): The L value decreased to 53.33%, which was lower than the values recorded at 90 and 120 minutes of microwave extraction, indicating that the oleoresin color resulting from maceration was relatively darker.

The a and b values were 3.47% and 31.64%, respectively, remaining relatively stable but lower compared to the microwave extraction results.

RGB values of (149, 124, 57) indicate a deeper yellow or dark brown coloration compared to microwave extraction at 90 and 120 minutes.

These findings demonstrate that extraction duration significantly affects the color properties of white pepper oleoresin. Microwave-assisted extraction for 90 to 120 minutes produced oleoresin with brighter color (higher L values) and more consistent chromatic properties (a and b stability), compared to the maceration method.

Maceration over 420 minutes resulted in a darker oleoresin color, likely due to the thermal degradation of color compounds during prolonged heating and extended contact time. This color shift reflects a complex interaction between natural pigment compounds, thermal degradation processes, and the efficiency of active compound release during extraction. Prolonged microwave extraction can enhance brightness up to a certain point, after which color lightness tends to plateau or slightly decrease.

In contrast, maceration requires a longer duration but yields a darker oleoresin product. Therefore, the selection of an optimal extraction method and duration is crucial to achieve desirable oleoresin color quality, particularly for applications in the food and cosmetic industries, where final product appearance is critical.

Relationship between extraction time (minutes) and oleoresin yield (%)

Linear regression analysis assessing the relationship between extraction time and oleoresin yield showed a positive linear graph (Figure 4).

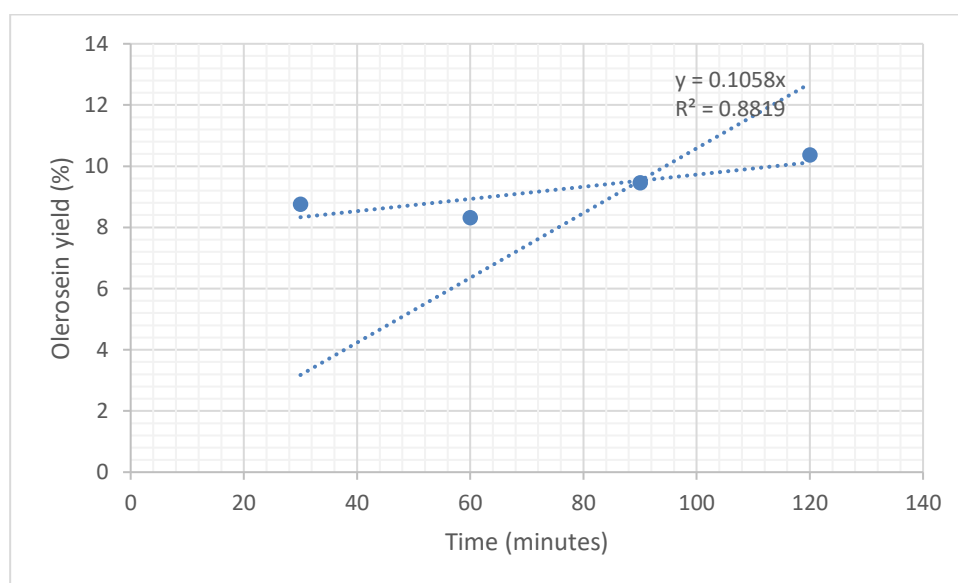


Figure 4. Linearity Curve of White Pepper Oleoresin

The figure illustrates the relationship between extraction time (minutes) and oleoresin yield (%) obtained from the extraction of white pepper (*Piper nigrum* L.) using the microwave-assisted extraction method. The extraction times evaluated were 30, 60, 90, and 120 minutes, with the corresponding yields presented in a scatter plot and fitted with a linear regression line. The resulting regression equation is:

$$y=0,1058x \text{ dengan } R^2=0,8819$$

From the graph, it is generally observed that the oleoresin yield increases with longer extraction times, particularly between 60 and 120 minutes. The highest yield was recorded at 120 minutes, while the lowest occurred at 60 minutes. Although there is a slight dip at the 60-minute point, the overall trend suggests a positive correlation between extraction time and yield.

The regression equation $y = 0.1058x$ indicates that each 1-minute increase in extraction time contributes to an approximate 0.1058% increase in oleoresin yield, following a linear pattern. The coefficient of determination $R^2 = 0.8819$ implies that 88.19% of the variation in oleoresin yield can be explained by the extraction time variable. The remaining 11.81% may be attributed to other factors not included in the model, such as extraction temperature, particle size of the raw material, or the energy distribution of the microwave radiation used during the extraction process.

From a scientific standpoint, increasing extraction time contributes to improved efficiency in the release of active compounds, including oleoresin components. This is due to: Enhanced diffusion of active compounds from plant cells into the solvent over time; Increased energy absorption by the material matrix, which accelerates the release of oleoresin components, especially under microwave systems that induce internal cell heating. However, excessively long extraction times may result in thermal degradation or the release of undesired (non-target) compounds, making time optimization a critical factor.

Interestingly, at the 60-minute mark, the yield slightly decreased compared to the 30-minute extraction. This anomaly may be attributed to: Experimental variability, such as uneven microwave heating distribution; Solvent interactions or possible reverse reactions occurring during transitional extraction phases. Nevertheless, the overall trend remains upward, as confirmed by the positive regression line.

The regression equation $y = 0.1058x$ with $R^2 = 0.8819$ indicates a strong and linear relationship between extraction time and oleoresin yield. While longer extraction durations tend to increase the yield, it is important to note that maximum extraction efficiency is achieved before reaching a saturation point or thermal degradation threshold. Therefore, determining the optimal extraction time is essential for maximizing yield while maintaining oleoresin quality.

Conclusion

Microwave-assisted extraction (MAE) produced higher white pepper (*Piper nigrum* L.) oleoresin yield than conventional maceration, even at shorter extraction times. The highest yield (10.370%) was achieved at 120 minutes, with optimal results at 90–120 minutes. Oleoresin extracted using MAE at 90–120 minutes had brighter and more consistent color characteristics compared to maceration.

Linear regression analysis showed a strong positive correlation ($R^2 = 0.8819$) between extraction time and yield, with an increase of ~0.1058% yield per additional minute. MAE is a time-efficient and quality-enhancing extraction method suitable for industrial applications requiring high yield and appealing product appearance.

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