Effect of particle size of white pepper to yield and quality of oleoresin produced by using an ultrasonic-assisted extraction method

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Abstract. The purpose of this study was to examine the effect of particle size of white pepper to yield and quality of oleoresin produced using Ultrasonic Assisted Extraction (UAE) method. White pepper from Bangka Belitung and South Sulawesi were ground using a milling machine and sifted to produce white pepper powder with a particle size of 30, 60, and 100 mesh. The samples (200 g each) were dissolved in ethanol (1:4) and subjected to UAE method to obtain oleoresin. The UAE was carried out using an ultrasound frequency of 20 kHz and an amplitude of 90% for 75 minutes. For comparison, the maceration extraction method was applied for 7 hours. The product of oleoresin was then subjected to vacuum evaporation to reduce ethanol solvent. The yield of oleoresin was calculated and the quality of oleoresin such color and piperine content was measured using colorimeter and GCMS, respectively. The results show that the particle size significantly affects the yield of oleoresin. The smallest particle size (100 mesh) gives the highest yield of oleoresin. The yield of oleoresin produced by UAE was not significantly different from maceration. The UAE method produced higher piperine content (7.51-11.91%) than the maceration extraction method (3.08-8.02%). The color of oleoresin produced by UAE method was similar to maceration.

1. Introduction

Indonesia is one of the biggest pepper producing countries (No.2) after Vietnam [1]. Pepper (black or white pepper) is commonly used as a spice and spicy aroma. Pepper is generally exported as a raw material such as seeds/grains at low prices. To increase added value, pepper can be processed into oleoresin. Oleoresin is easier to store and transport because of the smaller volume. Oleoresin has a longer shelf life because it is more resistant to oxidation and loss of aroma.

Oleoresin is a thick oil and consists of a mixture of essential oils, fixed oils, and resins [2]. Oleoresin from herbs is widely used as an aroma, flavor enhancer, and natural preservative with so many benefits. Generally, oleoresin is produced using maceration extraction. The disadvantage of this method is that it consumes a lot of organic solvents and time. One of the potential extraction methods to produce oleoresin more efficiently is Ultrasonic-Assisted Extraction (UAE).

Some recent studies on ultrasonic extraction methods have been carried out, such as to extract waste palm oil production [3]. They found that the yield of oleoresin using the UAE was higher than maceration and cut the extraction time to 90 minutes compared to maceration (7 hours). UAE was applied to produce black pepper oleoresin [4], nutmeg oleoresin [5, 6]. The UAE also produced more yield of oleoresin than a maceration. The UAE also produced more chemical constituents than a maceration. Despite the fact that many studies of UAE have been done, investigation on the effect of
particle size to yield and quality of pepper has not been carried out. So the aim of this research is to study the effect of particle size of white pepper to yield and quality of oleoresin produced using the Ultrasonic Assisted Extraction Method.

2. Material and method
2.1. Material and Apparatus
The material used in this study is white pepper from Bangka Belitung and South Sulawesi. Other materials used were 96% technical ethanol solvent and other chemical materials for analysis. White peppers were ground using a milling machine and sifted to produce white pepper powder with a particle size of 30, 60, and 100 mesh.

The tools used are UAE system based on the frequency of 20 kHz (Figure 1), rotary vacuum evaporators, filter paper, aluminum foil, stirrers, mouthpieces, hammer mills, 60 mesh sieves, scales, and glassware. While the equipment used for analysis is an oven, refractometer, distillation apparatus, color reader, and Gas Chromatography-Mass Spectrometry (GCMS).

![UAE system for white pepper oleoresin extraction](image)

1 = Ultrasound Generator  7 = Immersed Pump
2 = Ultrasonic Transducer  8 = Heat Exchanger
3 = Beaker glass  9 = Ice box
4 = Water bath  10 = Coldwater
5 = Temperature sensor  11 = Microcontroller
6 = Ultrasonic Probe  12 = Computer/Laptop (record)

Figure 1. UAE system for white pepper oleoresin extraction

2.2. Method
About 200 grams of white pepper powder (30, 60, 100 mesh) was dissolved into a beaker glass containing 800 mL 96% ethanol, then extracted using UAE system to obtain oleoresin (Figure 1) using the amplitude of 90% for 75 minutes. The UAE system is equipped with a water cooling system to maintain extraction temperature below 50 °C and for maintaining oleoresin quality. For comparison, the maceration extraction method was applied for 7 hours. The extraction product was filtered, and the solvent is evaporated with a rotary vacuum evaporator. The yield and quality are then measured (piperine, oleoresin, and color constituent components). Piperine content was determined by GCMS [7] and color by chromameter.
3. Results and Discussion

3.1. The yield of white pepper oleoresin by UAE and maceration

The yield of white pepper oleoresin produced by UAE is not significantly different from maceration, except for particle size of 60 mesh (Table 1). It may be caused by high variation between samples. The yield of oleoresin from South Sulawesi is higher than Bangka Belitung due to different agro-climate and variety of white pepper. Despite there is no significant difference between UAE and maceration, UAE method can reduce extraction time (75 minutes) than maceration (7 hours).

| Material Source | Extraction Method | Yield (%) * |
|-----------------|-------------------|-------------|
|                 |                   | Mesh  | Mesh  | Mesh  |
| Bangka Belitung | UAE               | 9.9   | 11.0  | 13.7  |
|                 | Maceration        | 10.4  | 8.0   | 13.0  |
| South Sulawesi  | UAE               | 13.1  | 13.9  | 16.6  |
|                 | Maceration        | 10.1  | 9.7   | 15.8  |
| Mean            | UAE               | 11.58b| 12.53b| 15.16c|
|                 | Maceration        | 10.25b| 8.85a | 14.40c|

3.2. Effect of particle size to yield of white pepper oleoresin

Table 2 shows the result of the effect of particle size to yield of white pepper oleoresin of Bangka Belitung and South Sulawesi. The particle size significantly affects to the yield of oleoresin. The smaller the particle size, the higher the yield of oleoresin. The smaller particle size will increase the surface area of contact, improve the extraction process, and produce more yield. The particle size of 100 mesh gave the highest yield of oleoresin.

| Mesh | Yield (%) |
|------|-----------|
| 30   | 11.58a    |
| 60   | 12.53ab   |
| 100  | 15.16b    |

3.3. Piperine content of oleoresin

The UAE method produced the higher piperine content of oleoresin (7.51-11.91%) than the maceration extraction method (3.08-8.02%) (Table 3). The oleoresin was extracted from 100 mesh particle size of white pepper. It indicates that UAE can extract more piperine content than a maceration. Piperine content of white pepper from Bangka Belitung is higher than South Sulawesi, suggesting that white pepper from Bangka Belitung is spicier than South Sulawesi.
Table 3. Piperine content of oleoresin extracted from 100 mesh particle size of white pepper

| Material Source | Piperine (%) |
|----------------|-------------|
|                | UAE     | Maceration |
| Bangka Belitung| 11.91   | 8.02       |
| South Sulawesi | 7.51    | 3.08       |
| Mean           | 9.71\textsuperscript{a} | 5.55\textsuperscript{b} |

3.4. Color of white pepper oleoresin
The color of white pepper oleoresin produced by UAE is similar to maceration, which is a yellowish red color. All particle sizes in the ultrasonic method produce the same color as maceration. This result indicates that UAE can produce similar quality oleoresin in terms of color. This result agrees with previous research \cite{4}\cite{5} that the optimal treatment is an amplitude of 90\%, and an extraction time of 75 minutes resulted in a color that is not significantly different from the control (maceration).

Table 4. Color of white pepper oleoresin

| Extraction Method | Particle Size (Mesh) | Color       |            |            |
|-------------------|----------------------|-------------|------------|------------|
|                   |                      | Bangka Belitung | South Sulawesi |
| UAE               | 30                   | Yellow Red (YR) | Yellow Red (YR) |
|                   | 60                   | Yellow Red (YR) | Yellow Red (YR) |
|                   | 100                  | Yellow Red (YR) | Yellow Red (YR) |
|                   | 30                   | Yellow Red (YR) | Yellow Red (YR) |
| Maceration        | 60                   | Yellow Red (YR) | Yellow Red (YR) |
|                   | 100                  | Yellow Red (YR) | Yellow Red (YR) |

3.5. Chemical constituents of white pepper oleoresin
UAE produced different chemical constituents of white pepper oleoresin than a maceration. For Bangka Belitung pepper, some components are present in the UAE, such as 2,3-Dihydro-3,5-dihydroxy-6-methyl-4-h-pyran-4-one, 1,3-cyclohexadien-1-yl methyl ethyl, N-Isobutyldecaadienamid, Isobornyl Methyl Ether, 2,4,4-Trimethyl-1-hexene and none in maceration. Vice versa, the components such as gamma-Pyrene, 1,2-Cyclohexanediol, 3-Octen-2-ol, Dipropenyl, Cyclohexanol, and Isoborneol are present in maceration and none in UAE. Similar results are also shown by South Sulawesi pepper. The components such as cyclopentanone and coumarin are present in UAE but none in maceration. Vice versa, glycerine, cyclohexanone, 1-pentyl-2,2-d2 acetate, D-Arabinitol, L-Arabinitol and Diazone are present in maceration, but none in UAE.

The most chemical components in white pepper oleoresin from Bangka Belitung and South Sulawesi are glycerine (24.7-32.7\%), gamma-Pyrene (4.96-40.1\%), 5-HYDROXYMETHYLURFURAL (9.19-21.2\%), and piperine (3.08-11.91\%). The amount of these four components dominates from monoterpen e hydrocarbons in the nutmeg oleoresin content both in UAE and maceration with 58.63-70.68\%.
Table 5. Chemical constituents of white pepper oleoresin

| Component                          | Concentration (%) |
|-----------------------------------|-------------------|
|                                   | Bangka Belitung   | South Sulawesi |
|                                   | UAE   | Maceration | UAE   | Maceration |
| Glycerine                         | 32.7  | 24.7       | -     | 27.38      |
| N-Formylpiperidine                | 3.06  | 3.45       | -     | -          |
| Dimethylsulfoxonium Dicyanomethylide | 8.12  | 12         | -     | 2.57       |
| Pipericide                        | 1.42  | 2.18       | -     | 1.3        |
| Pipelanine                        | 6.91  | 7.16       | 4.33  | 1.97       |
| Piperine                          | 11.91 | 8.02       | 7.51  | 3.08       |
| gamma-Pyrone                      | -     | 4.96       | 40.1  | 21.58      |
| 5-HYDROXYMETHYLFURFURAL           | -     | -          | 21.2  | 9.19       |
| 1,2-Cyclohexadiol                 | -     | 2.35       | 5.68  | 3.29       |
| 2,3-Dihydro-3,5-dihydroxy-6-methyl-4H-pyran-4-one | 4.92 | -          | -     | -          |
| 1,3-CYCLOHEXADIEN-1-YL METHYL ETHER | 2.65  | -          | -     | -          |
| Spiro[2.6]nona-4,6-diene          | 4.12  | 4.98       | -     | -          |
| N-Isobutyldodecanamid             | 2.35  | -          | -     | -          |
| Isobornyl Methyl Ether            | 6.84  | -          | -     | -          |
| 2,4,4-Trimethyl-1-hexene          | 5.24  | -          | -     | -          |
| 3-Octen-2-ol                      | -     | 7.7        | -     | -          |
| Dipropenyl                        | -     | 2.86       | -     | -          |
| Cyclohexanol                      | -     | 3.55       | -     | -          |
| Isoborneol                        | -     | 4.48       | -     | -          |
| Cyclopentanone                    | -     | -          | 2.51  | -          |
| coumarin                          | -     | -          | 1.49  | -          |
| Cyclohexanone                     | -     | -          | -     | 1.31       |
| 1-pentyl-2,2-diacetate            | -     | -          | -     | 2.31       |
| D-Arabinol                        | -     | -          | -     | 7.18       |
| L-arabinol                        | -     | -          | -     | 0.81       |
| Diazene                           | -     | -          | -     | 1.07       |

4. Conclusion

Particle size significantly affects to the yield of white pepper oleoresin. The particle size of 100 mesh gave the highest yield of white pepper oleoresin. The yield of white pepper oleoresin produced by UAE is not significantly different from maceration, but the extraction time of UAE (75 minutes) is less than maceration (7 hours). UAE produced higher piperine content than maceration at 100 mesh particle size. The UAE produces similar quality in terms of the color of white pepper oleoresin as maceration. UAE produced different chemical constituents than a maceration.

5. References

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