Comparison of *Curcuma domestica* and *Curcuma xanthorrhiza* oleoresins extracted using maceration, Soxhlet, and ultrasound-assisted extraction (UAE)

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**Abstract.** This research aimed to use conventional and modern extraction methods for extraction of *C. domestica* and *C. xanthorrhiza* oleoresins. For this purpose, maceration, Soxhlet, and Ultrasound-Assisted Extraction (UAE) were implemented to extract oleoresins. All the extraction method used acetone as a solvent with the ratio of ground spices to solvent was 1:5 (w/w). The results showed that the Soxhlet extraction method gave the highest oleoresin yield of *C. domestica* (18.89 ± 0.68%) and *C. xanthorrhiza* (14.70 ± 0.62%). Maceration method gave the highest curcumin yield of 49.52 ± 0.63% and 15.82 ± 0.27% for *C. domestica* and *C. xanthorrhiza* oleoresins, respectively. Meanwhile, the oleoresin obtained from the UAE method gave the lowest residual solvent on both *C. domestica* (16.00 ± 4.67%) and *C. xanthorrhiza* (15.66 ± 3.33%) oleoresins. *C. xanthorrhiza* oleoresin obtained from maceration tends to show the best colour, while the best colour of *C. domestica* oleoresin obtained from UAE. Propylene glycol had the best solubility for *C. domestica* oleoresin, while *C. xanthorrhiza* oleoresin was soluble in all solvents.

**Keywords:** *C. domestica* oleoresin, *C. xanthorrhiza* oleoresin, Maceration, Soxhlet, Ultrasound-assisted extraction

1. **Introduction**

Spices are one of the most important parts of the human diet, which have been used for thousands of years in traditional medicine; also used to enhance the flavor, color, and aroma of foods [1]. *C. domestica* and *C. xanthorrhiza* are spices that widely produce in Indonesia. Spices are generally produced and used in fresh, dry or powder form, but it is considered less efficient, especially in industrial scale that produces food and beverages in large quantities. Oleoresin can be an alternative because it is considered more profitable for the industry. The oleoresin is, therefore, designated as the “true essence” of the spice and can replace spice powders in food products without altering the color or flavor profile [2].

*C. domestica* and *C. xanthorrhiza* oleoresins can be obtained through the extraction process, either conventional or modern extraction methods. Soxhlet extraction, maceration, and hydro-distillation are conventional extraction methods based on the extracting power of different solvents in use and the application of heat and/or mixing [3]. The efficiency of other conventional and modern extraction methods...
methods can be evaluated using the Soxhlet method which is considered as a basic extraction technique [4]. Another traditional method is maceration which is a popular and inexpensive method to get bioactive compound and usually performed in a closed vessel with ground plant material [3]. Conventional methods which have been used for many decades have some drawbacks such as very time consuming, evaporation of the huge amount of solvent, low extraction selectivity, and require relatively large quantities of solvents [5]. Based on that, new extraction methods which are referred as modern extraction methods are introduced to overcome the shortcomings of conventional extraction methods.

Ultrasound-assisted extraction (UAE) is recognized as an efficient extraction method that reduces working times, increasing yields, and also the quality of the extract [6]. Ultrasound is a type of sound wave beyond human hearing, usually between 20 kHz to 100 MHz [3]. Cavitational effect of ultrasound wave enhances the mass transport by disrupting the plant cell walls and facilitates the release of extractable compounds [7]. Several effects such as erosion, particle breakdown and surface peeling were generated by the implosion of cavitation bubbles on a product’s surface causing yields enhancement [8]. Additionally, ultrasound can cause an enlargement of the pores of the cell wall by facilitating swelling and hydration processes, thus improve the diffusion process [9].

This research aimed to use conventional and modern extraction methods for extraction of C. domestica and C. xanthorrhiza oleoresins, and compare their characteristics. For this purpose, maceration, Soxhlet, and Ultrasound-Assisted Extraction (UAE) were implemented to extract oleoresins.

2. Material and methods

2.1. Material

Fresh rhizomes of C. domestica and C. xanthorrhiza were purchased from Balittro (Research Board on Herbs and Spices), Bogor, Indonesia. Standard curcumin (C_{21}H_{20}O_{6}, ≥ 75.0% purity) was acquired from Merck (Germany). The solvent used for the extraction was Acetone (C_{3}H_{6}O, ≥ 99.8% purity) of analytical grade and solvents for the chromatographic purpose that are methanol (CH_{3}O, ≥ 99.8% purity), acetonitrile (C_{2}H_{3}N, ≥ 99.9% purity) and distilled water were of HPLC grade. All solvents were purchased from Merck, Germany.

2.2. Plant material preparation

The fresh C. domestica and C. xanthorrhiza rhizomes collected were first thoroughly washed upon arrival at the laboratory. The rhizomes were sliced, blanched then dried using drying oven at temperature of 50 °C to obtain maximum 10–12% moisture content and then ground into powder. The ground powder was screened through a sieve with mesh 60 to obtained uniform particle size.

2.3. Maceration

200 g of ground sample was weighed and put in a glass jar, then dissolved with 1000 ml acetone (1:5). The mixture was stirred occasionally, then the entire glass jar was covered with aluminum foil to provide a dark environment and allowed to stand for 24 hours at room temperature.

2.4. Soxhlet extraction

200 g of ground sample was weighed and embedded in a thimble then put in the Soxhlet apparatus which was gradually filled with 1000 ml (1:5) acetone as the extraction solvent. The extraction experiment was carried out at 60 °C within 8 h until the extractant was colorless.

2.5. Ultrasound-assisted extraction (uae)

200 g of ground sample was weighed and put in an Erlenmeyer flask, then dissolved with 1000 ml acetone (1:5). The Erlenmeyer flask was immersed in Ultrasonic Cleaner Bath Bransonic 1510E-DTH (Mexico) with power 70 W and frequency 42 kHz, and it was covered with cling wrap and aluminum
foil to prevent solvent loss during extraction. The extraction process was carried out at a temperature of 35 °C for 30 minutes.

2.6. Solvent evaporation
Upon completion of the extraction, the acetone in all extracts was separated from the extract using rotary evaporator (Buchi Rotavapor® R-300, Switzerland) under vacuum at 40 °C.

2.7. Determination of residual solvent
Vacuum oven was used to evaporate the residual solvent in oleoresin. 0.5 gram of oleoresin was placed in a vial then put into the vacuum oven. The oven is operated at a temperature of 40 °C with an absolute pressure of 5 inHg for 3 hours. After evaporation was completed, oleoresin was put into the desiccator for 15 minutes to decrease the temperature into ambient temperature. The final weight was recorded and used to calculate the residual solvent.

\[
\text{Residual solvent (\%)} = \frac{\text{Final weight of oleoresin (g)}}{\text{Initial weight of oleoresin (g)}} \times 100\%
\]  

(1)

2.8. Determination of oleoresin yield
Oleoresin yield was reported in grams per ground turmeric mass (dry mass basis). The total oleoresin yield is expressed in percent units [10].

\[
\text{Oleoresin yield (\%)} = \frac{\text{Oleoresin extracted (g)}}{\text{Ground turmeric used (g)}} \times 100\%
\]  

(2)

2.9. Determination of curcumin yield
High-Performance Liquid Chromatography (HPLC) was used for calculation of curcumin concentration. The HPLC analysis was performed using a Waters Liquid Chromatograph (Milford, MA), which included a binary HPLC pump (Waters 1525), an autosampler (Waters 2707), and an ultraviolet (UV)-Vis detector (Waters 2489). Empower 2 software was used for controlling the analytical system and for data processing. The column was Symmetry® (Ireland) C18 with a dimension of 150 × 4.6 mm; the mobile phase was composed of acetonitrile and water at a ratio of 90/10; the flow rate was 1.0 ml/min at room temperature. The detection wavelength was 420 nm. The calibration curve was constructed by the dilution of curcumin standard with methanol to provide the desired concentration (1, 2.5, 5, 8, 10 ppm) followed by injection into the HPLC system. To determine the curcumin content of unknown samples, a certain concentration of sample (5 ppm for \textit{C. domestica} and 10 ppm for \textit{C. xanthorrhiza}) was made. A certain amount of oleoresin (5 mg for \textit{C. domestica} and 10 mg for \textit{C. xanthorrhiza}) quantitatively weighed, was transferred into a 10 ml volumetric flask. It was dissolved in methanol and diluted to volume with methanol. 0.1 ml portion of this solution then pipetted into a second 10 ml volumetric flask and diluted to volume with methanol. Prior to injection in the HPLC system, both standard solutions and sample were filtered using Waters Acrodisc PTFE 0.45 μm, 13 mm. Standard calibration curves were obtained by plotting the concentration of standard curcumin versus peak area. Quantification of extracted curcumin was calculated using the equation below [4]:

\[
\text{Curcumin yield (\%)} = \frac{\text{Curcumin extracted (mg)}}{\text{Turmeric used (mg)}} \times 100\%
\]  

(3)

2.10. Surface colour analysis
Spectrophotometer (Konica Minolta CM-5Sensing Singapore Pte Ltd) was used to examine the colour characteristics. The results were expressed as L*, a*, and b*. L* value determines lightness or darkness, a* determine redness or greenness, while b* determines yellowness or blueness [11].
2.11. Oleoresin solubility

1 mL of the oleoresins was added by small portions of ethanol, propylene glycol, and palm oil. After each addition, the solution was shaken thoroughly. The additions of ethanol, propylene glycol, and palm oil made until 10 mL of solvent added. The oleoresin solubility was then observed for its appearance [12].

3. Results and discussions

3.1. Residual solvent

In the case of herbs and spices, the stripping of the solvent from the desired extractives is of immense importance to the quality of the end-product. To avoid loss of or damage to the flavour profile, the solvent should be evaporated from other low-boiling constituents as rapidly as possible at the lowest practicable temperature [13]. Although the residual solvents may not be entirely removed by practical manufacturing techniques, they should be removed to meet ingredient and product specification because residual solvents do not provide therapeutic benefit [14].

In this study, oleoresins were obtained by evaporating the extract by rotary vacuum evaporator. It is expected that this process can remove solvent as rapidly as possible due to residual solvents can affect the quality of oleoresins. The remaining solvents contained in oleoresin must comply with FDA requirements. The method of determining residual solvent in oleoresin was carried out using a vacuum oven for 3 hours at a temperature of 40 °C.

Figure 1 shows the residual solvent of *C. domestica* and *C. xanthorrhiza* oleoresins. Oleoresins obtained using the UAE method gave the lowest residual solvent in comparison with the other extraction method, that are 16.00 ± 4.67% for *C. domestica* oleoresin and 15.66 ± 3.33% for *C. xanthorrhiza* oleoresin. However, residual solvent in oleoresins obtained are still very high and far above the maximum limits set by the U. S. FDA, which is 30 ppm or 0.003% for acetone as the solvent [15]. This high residual solvent is because of the evaporation process with a rotary vacuum evaporator could not remove all solvents in oleoresin causing the amount of residual solvent in the oleoresin to be large. It was found that in the traditional method to remove solvent using a vacuum, the difference of solvent contents between the surface and the system drives the diffusion, but the diffusion is very slow and costs much time [16]. If the residual solvent exceeds the limit, the evaporation process needs to be optimized as it will influence its application in food and pharmacy [17]. Sweeping solvent by nitrogen could be an effective additional method to evaporate the remaining solvents. While conducting the sweeping method using nitrogen, the solvent vapor pressure decreased which makes more solvent molecules in liquid vaporized into the upper vapor phase through the liquid surface, then the moving inert gas immediately removing the solvent in the vapor phase [16], so the solvent does not have a chance to return to the liquid and it will increase the evaporation rate.
3.2. Oleoresin yield

The average of oleoresin yields of the different extraction method is presented in figure 2. The result showed that the oleoresin yield of both sample *C. domestica* (18.89 ± 0.68%) and *C. xanthorrhiza* (14.70 ± 0.62%) obtained from Soxhlet extraction method was higher as compared with that of the other extraction method. Soxhlet extraction can extract more sample mass than most of the latest alternatives (microwave-assisted extraction, ultrasound-assisted extraction, etc.), it is possible because during extraction process the sample is repeatedly brought into contact with fresh portions of extractant which facilitates displacement of the transfer equilibrium [18]. Moreover, extraction temperature can also affect the amount of oleoresin yield obtained. The higher temperature causes intermolecular interactions within the solvent to decrease, giving rise to higher molecular motion and causing the solubility to increase [19], thus accelerating the whole extraction.

Although Ultrasound-Assisted Extraction (UAE) method used in this study resulted in lower yield but seem to be more promising from both economic (much less time and energy consumption compared to Soxhlet) and environmental (consumption of much less solvent) views [4]. Result showed that the
oleoresin yield of *C. domestica* and *C. xanthorrhiza* obtained from UAE was higher than maceration method. Ultrasound-Assisted Extraction (UAE) is recognized as an efficient extraction method that reduces working times, increasing yields, and also the quality of the extract [6]. The physical and chemical properties of plant material were altered after its interaction with ultrasound waves and cavitation effects that occur during ultrasound radiation facilitates the release of extractable compounds [7]. In several solid-liquid extraction processes, ultrasound can be effectively used to increase the yield and rate of mass transfer [20].

3.3. Curcumin yield

Table 1 shows the curcumin yields of *C. domestica* and *C. xanthorrhiza* oleoresins obtained from different extraction methods. Curcumin yields calculated with the residual solvents are lower when compared to curcumin yields without residual solvents in each method. This shows that the presence of solvents in oleoresin can reduce the yield of curcumin because it will affect the actual amount of curcumin in oleoresin so the percentage of curcumin is smaller. Result showed that curcumin yield of *C. domestica* was higher when compared with *C. xanthorrhiza* oleoresin. It is in agreement with the previous study stated that *C. domestica* has higher levels of total curcuminoids compared to *C. xanthorrhiza* [21].

| Material        | Method    | Curcumin Yield (%) | Curcumin Yield Without Residual Solvent (%) |
|-----------------|-----------|--------------------|--------------------------------------------|
| *C. domestica*  | Maceration| 33.56 ± 0.43       | 49.52 ± 0.63                               |
|                 | Soxhlet   | 32.41 ± 0.17       | 48.38 ± 0.25                               |
|                 | UAE       | 36.22 ± 0.10       | 43.12 ± 0.12                               |
| *C. xanthorrhiza*| Maceration| 8.13 ± 0.14        | 15.82 ± 0.27                               |
|                 | Soxhlet   | 2.86 ± 0.12        | 5.34 ± 0.22                                |
|                 | UAE       | 3.90 ± 0.03        | 5.30 ± 0.05                                |

It was found that the maceration method gives the highest curcumin yield on *C. domestica* and *C. xanthorrhiza* oleoresins, followed by Soxhlet and UAE. This is possible since curcumin is very sensitive to light [22]. Unlike the maceration method, the ground powder extracted by Soxhlet and UAE method exposed to continuous light. Under the influence of light, curcumin undergoes a self-sensitized photo-decomposition where singlet-oxygen is involved [23].

In the Soxhlet method, the use of high temperatures during the extraction process causes an increase in oleoresin yield (figure 2), but can also lead to higher chances of curcumin degradation than the maceration method. It is in agreement with the previous study stated that curcumin loss from heat processing of turmeric was 27–53% [24]. This is because several modifications can occur during heat treatment of curcumin, such as conjugated double bond shifting, polymerization, and degradation to lower molecular weight compounds, it shows the vulnerability of the 'diketone bridge' in curcumin molecule to heat [25].

Result showed that the curcumin yield of both sample *C. domestica* (43.12 ± 0.12%) and *C. xanthorrhiza* (5.30 ± 0.05%) obtained from the UAE method was the lowest than other methods. It is known that the ultrasonic energy can provide huge heat (pyrolysis) or produce reactive free radicals which influence the chemical reactions [26], so it might be caused by the degradation or oxidation of curcumin structure at prolonged exposure to ultrasound wave [4].

3.4. Surface colour

Colour determines the commercial value of oleoresins and is generally associated with the quality of the sample: the greater the colouring capacity, the higher the quality. The colour parameters of oleoresin are shown in figure 3.
Figure 3 shows that the L* value of both samples, *C. domestica* and *C. xanthorrhiza* oleoresins, obtained from three different methods shows a tendency for dark or less bright colour. The a* value has a positive value, this means that oleoresin has a red colour. The b* value also shows a positive value which means that it tends to yellow colour.

*C. xanthorrhiza* oleoresin with the maceration method showed a higher value of L*, a*, and b* than other methods indicate the oleoresin extracted by the maceration method tends to show a brighter colour and tends to show more red and yellow colours than the oleoresin extracted by other methods. This is related to the results of curcumin content of *C. xanthorrhiza* oleoresin. This is allegedly caused by maceration methods that do not use high temperatures, in contrast to the Soxhlet and UAE that use high extraction temperatures. Pigments may be degraded by isomerization, decarboxylation or cleavage during heat processing, resulting in a gradual reduction of colour and eventually the appearance of a brown colour [27]. Some study also states that curcumin decomposition increased with increasing temperature from 20 to 90 °C [28].

In contrast to *C. xanthorrhiza* oleoresin, it was found that curcumin content was not directly proportional to the surface colour of *C. domestica* oleoresin. Compared with maceration and Soxhlet methods, *C. domestica* oleoresin obtained from the UAE method has a better brightness and yellow colour, but lower curcumin content. One possible reason for the increased brightness and yellow colour of oleoresin might be due to low residual solvent. The surface colour was not always related to the extractable colour. The amount of oil present can also affect the amount of surface colour, the higher amount of oil gave the darker surface colour [29]. It might be due to the tendency of oils in oleoresin to dilute the colour strength [2].

Acetone, the solvent that used in this study, is a semi-polar solvent that capable of dissolving both polar and nonpolar substances [30]. Based on this fact, it is suspected that there is a correlation between the residual solvents and the amount of oil dissolved. Figure 1 shows that the highest residual solvent was obtained from the Soxhlet method while the lowest was obtained from the UAE method. These results are found to be proportional to the brightness of the *C. domestica* oleoresin colour. The higher amount of residual solvent, the more oil is dissolved in acetone which causes the oleoresin surface colour to darken.

### 3.5. Solubility

The solubility of *C. domestica* and *C. xanthorrhiza* oleoresins in a different solvent is presented in table 2. Propylene glycol was found to have the best solubility for *C. domestica* oleoresin in comparison with
two other solvents. Turmeric oleoresin is usually extended with nonvolatile edible diluents or emulsifiers such as a propylene glycol for convenience in application [2]. As emulsifying agent, propylene glycol provides good solubility of *C. domestica* and *C. xanthorrhiza* oleoresin may be due to the nature of curcumin, which contains both a lipophilic and hydrophilic group [31]. The previous study reports that addition of propylene glycol can enhance the solubility of curcumin, this is because of the formation of hydrogen bonds with the hydroxyl groups (–OH) and hydrogen (–H) atoms in curcumin by polyhydric alcohols contained in propylene glycol [32].

**Table 2.** *C. domestica* and *C. xanthorrhiza* oleoresins solubility in ethanol, propylene glycol, and palm oil.

| Material          | Solvent Ethanol 96%                                      | Solvent Propylene Glycol                                    | Palm Oil                                     |
|-------------------|----------------------------------------------------------|------------------------------------------------------------|---------------------------------------------|
| *C. domestica*    | Slightly soluble, yellow fine particles precipitate was formed, the solution becomes transparent-red in colour | Soluble, the solution becomes opaque-orange in colour       | Very slightly soluble, semi-solid lumps precipitate was formed, the solution was translucent-yellow in colour |
| *C. xanthorrhiza* | Soluble, the solution was clear and dark brown in colour | Soluble, the solution becomes opaque-light brown in colour   | Soluble, the solution becomes opaque-brownish yellow in colour |

The solubility in alcohol is strongly influenced by components contained in oleoresin. The polymerized compound will reduce the solubility of oleoresin in alcohol. The polymerization process is easy to occur mainly in essential oils of oleoresins which contain large amounts of terpenes caused by heat [12]. Curcumin is readily soluble in solutions such as dimethyl sulfoxide, ethanol, and acetone but is sparingly soluble in aqueous solutions [33]. The solubility of *C. domestica* oleoresin in palm oil is very slight (table 2). Study reported that the solubility of curcumin was much higher in MCT (Medium Chain Triglyceride) oil compared to other oils (coconut oil, olive oil, and corn oil), it is also known that coconut oil contains a mixture of MCT and LCT (Long Chain Triglyceride), whereas olive oil and corn oil are mainly composed of LCT [34]. A study reports that palm oil does not contain MCT but contains LCT [35]. This fact might explain the poor solubility of *C. domestica* oleoresin in palm oil (table 2).

### 4. Conclusion

The *C. domestica* and *C. xanthorrhiza* oleoresins obtained from Soxhlet extraction method gave the highest oleoresin yield of *C. domestica* (18.89 ± 0.68%) and *C. xanthorrhiza* (14.70 ± 0.62%) oleoresins, but maceration method gave the highest curcumin yield of 49.52 ± 0.63% and 15.82 ± 0.27% for *C. domestica* and *C. xanthorrhiza* oleoresins, respectively. Meanwhile, the oleoresin obtained from the UAE method gave the lowest residual solvent. However, residual solvent in oleoresins obtained are still very high and far above the maximum limits set by the U.S. FDA. *C. xanthorrhiza* oleoresin with the maceration method showed the highest value of L*, a*, and b*, while *C. domestica* oleoresin obtained from the UAE method showed the highest value of L* and b*. Propylene glycol was found to have the best solubility for *C. domestica* oleoresin in comparison with two other solvents, while *C. xanthorrhiza* was soluble in all solvents. Results show that different extraction method produced oleoresin with different characteristics.
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