Isolation of capsaicin compounds from cayenne as a food additives material

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Abstract. This study aims to isolate the capsaicin compound from cayenne by reflux method using methanol solvent. Yield oleoresin obtained as much as 51.71% of 30 grams of dried cayenne at a ratio of 1:16 with extraction time for 7 hours. Oleoresin has a blackish brown color, thick and has a spicy smell. The obtained oleoresin was analyzed by Spectrophotometer FTIR show that chain –OH, -NH, CH$_2$, -CH$_3$, C=C, C=O and O-CH$_3$.

1. Introduction
Cayenne is the one of the basic spices materials that are widely used as flavouring in various types of food. The price of cayenne often fluctuates due to economic turnover. To resolve the fluctuations of the price of cayenne that occur at any time, extraction of capsaicin in cayenne is an alternative way that can be used to resolve these problems. One of the biggest places in cayenne's producing areas is in Takengon, located in Central Aceh, Aceh's Province. The availability of cayenne is very abundant during the harvest season. Data recorded by the Central Statistics Agency (CSA) shows that cayenne production in Aceh in 2017 reached 53,800 tons and its production is expected increase in the following years (BPS, 2017).

Capsaicin is an active substance that has a hot and spicy effect on cayenne. In addition to being used as food or as flavor, capsaicin can also be used in the pharmaceutical sector in making various medicines. The use of capsaicin oleoresins can reduce transportation costs due to the volume per unit weight will be reduced and storage is easier. The capsaicin industry is a solution for farmers not to suffer losses, because it doesn't allow hoarding of cayenne to linger at harvest time. On the others hand to culinary entrepreneurs is the most efficient way to give a spicy taste to food without being affected by fluctuations in the price of chili on the market. The advantages of capsaicin crystals are long-lasting, can be carried everywhere, the spiciness of food can be easily arranged (according to the dosage) and the storage is easier.

The focus of this research is the isolation of capsaicin compounds from cayenne originating from the Takengon for the purposes of food materials. There are several studies on the isolation of capsaicin with different methods including (Amaliah, 2018). The results showed that the highest capsaicin levels were found in red katokkon chilli powder of 0.304% (± 0.016) while green katokkon chilli powder was 0.266% (± 0.015) . According to Thaib's research results, et al. (2015) obtained 39.4 grams (39.4%) of capsaicin oleoresin extract and 0.5 g (0.5%) capsaicin crystals of 100 g dried cayenne pepper products. The capsaicin compound which was successfully isolated was then characterized by FTIR spectra and showed the presence of –OH, -NH, –CH$_2$, -CH$_3$, C=C, C=O and O-CH$_3$ groups.

One of the most important factor for the successfully in extraction process is the amount of solvent. The solvent used must be in equal with the material will be extracted. Amaliah (2018) in her journal said that the extract of capsaicin compounds is not soluble in water but fat, methanol, ethyl acetate and ethyl alkali instead. Testing of solvent types using five types of organic solvents namely hexane, methanol, ethanol, chloroform, and ethyl acetate, a good
solvent in dissolving capsaicin is methanol. Researchers were interested in conducting research on the manufacture of capsaicin oleoresins. Thaib (2015) has produced 39.4 grams of oleoresin (39.4%) using the reflux method with chloroform solvent. However, in the Amaliah study, a type of solvent was tested by maceration method using five types of organic solvents, namely hexan, methanol, ethanol, chloroform, and ethyl acetate.

The researcher was interested in using the reflux method because the highest percentage of capsaicin was obtained using this method and methanol as a solvent because based on the results of testing the type of solvent, methanol was best dissolved in capsaicin.

2. Material and Method

The equipment used in this study include a set of reflux, oven, blender, analytic balance, 80 mesh sieve, desiccator, rotary evaporator, melting point apparatus, digital scales. While the material used in this study include methanol 96% (pa).

2.1. Sample Preparation

Fresh cayenne is washed clean. Then amount of water is measure by moisture content of the raw material. After that cayenne is dried using an oven at a temperature of 50⁰C for 48 hours then the final water content is measure. The sample then size reduction using crusher, then sifted with an 80/100 mesh sieve.

2.2. Isolation Of Capsaicin

30 grams of sample is put into around bottom flask. Methanol solvent is added (according to the independent variable) then refluxed at a temperature of 65⁰C for the time specified. The extract is filtered with a funnel. Then washed the residue with methanol and then filtered it back. The extract was evaporated which was obtained to separate the methanol solvent so that the extract of cayenne was obtained.
3. Results and Discussion

From the results of the measurement of cayenne moisture content, it was obtained the initial water content of fresh cayenne 89.1% and obtained the final moisture content of dried cayenne 63.7%. Extraction was carried out at a temperature of 65 °C according to the boiling point of methanol, if extraction carried out at higher temperature it is feared that it can damage the capsaicin compound in cayenne and the solvent will be evaporated. The research start by reacted 30 grams of cayenne powder in reflux method using methanol as solvent and
obtained the most oleoresin with a treatment of 7 hours of extraction with a total solvent of 480 ml. The oleoresin has a blackish brown color, thick and has a spicy smell. The results obtained in the study can be seen in Figure 1. In the study using the ratio of variations in the number of solvents 1:12, 1:14 and 1:16 (w/v) with extraction time for 5, 6 and 7 hours.

![Figure 6. Effect of increased solvent ratio to yield of capsaicin](image)

After extract capsaicin was obtained, evaporation was carried out to obtain pure capsaicin, evaporation temperature between 65-70 °C, temperature used was in accordance with the temperature of evaporation of methanol. Yield of capsaicin is determined with mass oleoresin of capsaicin divided by mass capsaicin in sample. Example:

\[
\% \text{ yield capsaicin} = \frac{69}{100} \times 30 \text{ gram} = 20.7 \text{ gram}
\]

\[
\% \text{ Yield capsaicin} = \frac{10.744 \text{ gram}}{20.7 \text{ gram}} \times 100\% = 51.71\%
\]

From the Figure 6, it can be seen that at a ratio of 1:16 the yield produced increases, it can be seen that the highest oleoresin was obtained 51.71%. The result obtained were more compared to Thaib (2015) who only got oleoresin as much as 34.5%. This is due to the greater number of solvents used compared to previous, so that the saturation level of the solvent in contact with cayenne powder is lower, because the distribution of particles in the solvent is increasingly diffusing, thus expanding the contact surface (Maslukhah, 2016). Thaib uses chloroform solvent with extraction time for 8 hours, while this study was treatment of 7 hours. It means that the solubility of methanol is higher than chloroform. In terms of sample size, Thaib uses a 65 mesh sieve while in this study using 80/100 mesh sieves, so that the contact area between a cayenne pepper and solvent is wider and can bind more capsaicin compounds.

From the graph also shows that the longer the extraction time, the yield of capsaicin yield increases. This is because time is one of the factors that can influence the results of extraction, because the longer the extraction time, the longer the contact time for cayenne powder with solvents will increase the yield of extraction.
Figure 7. Capsaicin Extract

Capsaicin extract then analyzed by Spectrophotometer FTIR to determination the chain form of capsaicin. From the analyzed show that –OH, -NH, CH$_2$, -CH$_3$, C=C, C=O and O-CH$_3$.

Figure 8. The result of FTIR Spectrophotometer analysis

| Chain | Reference (Thaib, 2015) | Alya Rahmatiz Zahra | Standard (Silverstein and ) |
|-------|-------------------------|---------------------|-----------------------------|
| -OH   | 3409,9                  | 3310,56             | 3800-2700                   |
| -NH   | 920                     | 610,82              | 909-606                     |
| CH$_2$| 2854,5                  | 2835                | 2926-2853                   |
| CH$_3$| 2927,7                  | 2931,11             | 2962-2872                   |
| C=C   | 1643,2                  | 1599,62             | 1900-1500                   |
| C=O   | 1747,4                  | 1519,45             | 1900-1500                   |
| O-CH$_3$| 1164,9                | 1072,53             | 1250-1050                   |

4. Conclusions
Based on the research carried out 51.71% of oleoresin was obtained with the treatment of 7 hours extraction with a total solvent of 480 ml. Oleoresin obtained has a blackish brown
color, thick and spicy smelling. The result of FTIR Spectrophotometer to determination the chain form of capsaicin show that –OH, -NH, CH2, -CH3, C=C, C=O and O-CH3.

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