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Isolation of Lycopene Component from Tamarillo (*Solanum betaceum*)

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Abstract

Lycopene is a red pigment found in tamarillo with its function as an antioxidant that protects body cells from the negative effects of free radicals so that they do not trigger diseases, especially cancer and premature aging. This study was intended to improve the quality of tamarillo by isolating lycopene from tamarillo through an extraction process with chloroform as a solvent by maceration for 3 days at room temperature. The lycopene extract obtained was dissolved by means of a rotary evaporator at 40 ^{o}C under vacuum pressure and oven temperature at 60 ^{o}C. The lycopene obtained was analyzed by Gas Chromatography Mass Spectrometry and the crystal structure of lycopene was characterized by Fourier Transform Infra-Red Spectroscopy. GCMS results showed that 0.21 g of lycopene was successfully isolated from 100 g of dried red tamarillo fruit powder. Functional group analysis using FT-IR at a wavelength of 978.23 cm^{-1} showed the R-CH=CH-R group; the -CH_3- group of 1371.43 cm^{-1}; the 1460.16 cm^{-1} indicates the bending vibration of -CH_2-; the C=C chain of 1656.91 and 1745.64 cm^{-1}; and the C-H of 2856.67 and 2926.11 cm^{-1} of the lycopene chain.

Keywords: Chloroform, extraction, FTIR, GCMS, lycopene, maceration, tamarillo

INTRODUCTION

The use of lycopene has recently become widely known in the industrial world driven by its wide use in both the food industry and the pharmaceutical industry. In the food industry, lycopene is used as a natural dye, according to its functions as a natural red color for fruits and vegetables, while in the pharmaceutical industry, lycopene plays a role in the body to capture free radicals.

Free radicals are a by-product of metabolic processes in the human body (Haroon, 2014). Free radical compounds come from environmental pollutants and also from unhealthy human lifestyles with lower immunity (Arnanda & Nuwarda, 2019) (Fadiyah, Lestari, & Mahardika, 2020) (Surtina et al., 2020). Free radicals are atoms or molecules that are unstable and highly reactive because they contain one or more unpaired electrons in their outermost orbital. To achieve atomic or molecular stability, free radicals will react with surrounding molecules to gain electron pairs. This reaction will continue in the body and if not stopped will cause various diseases such as cancer, heart disease, cataracts, premature aging, cardiovascular, diabetes, osteoporosis, infertility, and other degenerative diseases.

Therefore, the body needs an important substance to protect cells from damage caused by free radicals, namely antioxidant in the form of lycopene which is able to capture these free radicals so that they do not trigger the onset of a disease. The antioxidant potential of lycopene as a singlet oxygen catcher is twice of β-carotene and ten times of α-tocopherol (Arifulloh, Oktavianawati, & Winata, 2016). In addition, the ability of lycopene to control free radicals is 100 times more efficient than vitamin E or 12,500 times than glutathione. Lycopene is a natural pigment that protects the body by neutralizing the negative effects of antioxidants(Hattu, Latupeirissa, Fransina, Seumahu, & Latupeirissa, 2014) (Lilwani & Nair, 2015).

Lycopene is found in many red fruits and vegetables such as tomatoes, papaya, watermelon and tamarillo. Tamarillo (*Cyphomandra betacea*) is a horticultural plant that grows in Indonesia, especially in Tana Toraja, South Sulawesi. Based on data from Central Bureau of Statistics of Indonesia, the production of tamarillo in Indonesia in 2012 was 518,448 tons. Tamarillo contains a variety of chemical compounds that vary greatly, depending on several factors including fruit maturity, genetics, as well as geographical and environmental conditions in which the plant grows (Wang & Zhu, 2020).

So far, tamarillo as shown in Figure 1 is only known as a fruit that can only be consumed directly in

the form of fresh fruit and processed into juice like the use of other fruits in general.

Figure 1. Tamarillo *(Solanum betaceum)*

The impact of a lack of knowledge on this tamarillo fruit, so that there are no derivative products from this fruit. This is further made worse by perishable nature of this fruit. Ripe fruit that is picked and stored at room temperature is only able to last for 5 to 6 days, after which the peel will turn black with rot so that this fruit cannot be consumed anymore and then it is thrown away (Pakiding, Muhidong, & Hutabarat, 2015). Lycopene is an unstable compound, so to get a pure compound, a difficult process is needed (Kong et al., 2010). This has an impact on the price of pure lycopene compounds in the market which is quite expensive (Desmiaty, Alatas, & Sugianti, 2008).

Previous investigation of Atiqah, Maisarah, & Asmah, (2014) have established the comparison of antioxidant content in tamarillo, cherry tomatoes, and tomatoes. Tamarillo showed the highest antioxidant content among the three fruits with levels of 2.92-3.85%. The lycopene content in the peel of the red tamarillo is 2.22 mg/100 g (Siddick & Ganesh, 2005).

The lycopene compounds obtained in the separation process by extraction tend to be perfect if the solvent used is a non-polar solven (Dewi, 2018). (Christianty, Gavra, & Masyithah, 2015) also obtained pure lycopene crystals in tomatoes from the extraction process with a mixed solvent of hexane: ethyl acetate (1:1). To obtain pure lycopene compounds, an antisolvent crystallization process was carried out by adding methanol. In addition, (Indriani, Ruslan, Prismawiryanti, & Satrimafitrah, 2018) succeeded in producing lycopene from tomato fruit extract by soaking it in VCO (Virgin Coconut Oil). Optimization of the extraction process of lycopene from tomatoes with a mixed solvent, namely acetone:ethyl acetate with a solvent ratio of 1:1. The extraction process was carried out at 40 ^{o}C for 5 hours. The total lycopene that was successfully extracted was 61.105 mg/100 g (Hiren, Akbari, Pandya, & Josdi, 2017).

Maulida & Naufal, (2010) succeeded in isolating lycopene from tomatoes through an extraction process at a temperature of 65 ^{O}C using chloroform solvent, then lycopene crystals were obtained by the antisolvent crystallization method, namely methanol under supersaturation conditions. The total lycopene crystals that were isolated were 4.87 mg/100 g, the crystal structure was analyzed by FTIR (Fourier Transform Infra Red).

Temperature and particle size are one of the main factors in the separation process by extraction. The solubility of the extracted material increases with increasing temperature and the smaller the particle size, the easier the extraction process because of the wider contact surface area between the solid and the solvent (Damayanti, Sukasri, & Nurdin, 2020).

Therefore, the aim of this investigation has been to find out a simple and effective method of extracting lycopene from tamarillo through the extraction process using the maceration method using chloroform solvent and the method of separating lycopene from chloroform solvent using a rotavapor. The concentrated lycopene obtained was then analyzed by GCMS (Gas Chromatography Mass Spectrometry) and Spectrophotometer FTIR (Fourier Transform Infra Red).

METHODOLOGY

Materials and Instrumentals

The research materials used were tamarillo fruit (*Solanum betaceum*) from Tana Toraja, South Sulawesi Province, Indonesia. Other ingredients, namely: distilled water, chloroform (Merck), and filter paper (no brand). The equipments used in this study were glassware (Pyrex), digital balance (Precisa), blender, knife, vacuum pump, oven (Ecocell), rotavapor (Buchi), FT-IR (Shimadzu 8400S), and GC-MS (Shimadzu QP2010 Ultra).

Methods

Raw Material Preparation

Tamarilo fruit that has been ripe was first cleaned by washing with clean water to remove impurities that stick to the skin of the fruit. The tamarillo fruit was then cut into pieces and dried in an oven at 60 ^{o}C for 4 days. Tamarillo fruit that has been dried, then was mashed using a blender to get dry tamarillo powder.

Lycopene Isolation

Samples (tamarillo powder) as much as 100 g were weighed and put into an erlenmeyer. 300 ml of chloroform solvent was added to an erlenmeyer containing a sample of tamarillo powder. The

extraction process using the maceration method was carried out by soaking the tamarillo powder with chloroform solvent for 2 days at room temperature, stirring occasionally. After 2 days, the samples were filtered through filter paper. The filtrate and residue are separated. The filtrate (lycopene extract) was stored in another erlenmeyer while the residue was added again with 200 ml of chloroform. The sample was then macerated again for 1 day at room temperature with occasional stirring. After maceration for 1 day in the next stage, the sample was filtered again. The solution (filtrate) obtained was combined with the filtrate from the initial stage of maceration. The filtrate (lycopene extract) in erlenmeyer was poured into the evaporator flask. The sample was concentrated by means of an evaporator at a temperature of 40 ^{o}C to a volume of 50 ml. The lycopene extract was poured into a beaker and placed in an oven at 60 ^{o}C until all the chloroform solvent was evaporated.

Data Analysis

Gas Chromatography Mass Spectrometry (GC-MS Analysis)

Lycopene extract was analyzed using GCMS (Gas Chromatography Mass Spectrometry), brand Shimadzu with type QP2010 Ultra. A sample of 1 L was injected into the GCMS which was operated with a glass column of 25 m long, 0.25 mm in diameter and 0.25 m in thickness with a stationary phase of CP-Sil 5CB with an oven temperature programmed between 70-270 ^{o}C with a temperature rise rate of 10 ^{o}C/min, Helium carrier gas has a pressure of 12 kPa, a total rate of 30 ml/min and a split ratio of 1:50. The weight of extracted lycopene was calculated by Equation 1.

$$Lycopene\ weight = \frac{percentage\ of\ extracted}{amount\ of\ extracted\ tamarillo} \quad (1)$$

Fourier Transform Infrared (FT-IR) Spectroscopy

FT-IR was used to identify functional groups and to characterize lycopene extract from red tamarillo fruit. The FT-IR spectroscopic instrument used was the Shimadzu brand. A total of 1 drop of concentrated lycopene was scanned 40 times from 4000 to 500 cm^{-1}.

RESULTS AND DISCUSSION

Isolation of Lycopene

The separation of lycopene from tamarillo was carried out in a simple and effective way through the extraction process by maceration method at room temperature using chloroform as solvent. Figure 2 displays the isolation of lycopene by maceration method that produced lycopene extract to obtain lycopene extract that has been free from chloroform solvent, using a rotary evaporator (rotavapor) and oven. Carotenoids are pigments that give yellow-red color to fruits and vegetables. The red color concentration of the lycopene extract is caused by the conjugated double bonds of carotenoid compounds. There are more conjugated double bonds in the structure of lycopene than other carotenoid compounds, as many as 11 conjugated double bonds cause lycopene to have an orange color (Arifulloh et al., 2016)

Figure 2. Lycopene isolation: (a) maceration, (b) lycopene extract, and (c) concentrated lycopene extract.

Lycopene is a nonpolar compound that will dissolve in nonpolar solvents (Hamsina, Hasani, & Irfan, 2019). The solvent chosen will determine the ability to take up lycopene which is expected to be isolated. The solvent chloroform is a nonpolar solvent, which is why this solvent was chosen in this study. The solvent power of chloroform in the extraction process is the strength of the dispersion of chloroform molecules to interact with lycopene components. Lycopene compounds are basically completely dissolved in chloroform as a non-polar solvent because the intermolecular forces between similar compounds tend to have the same strength (Dewi, 2018).

Lycopene Analysis with GCMS

Analysis with GCMS (Gas Chromatoraphy Mass Spectroscopy) was used to measure qualitatively and quantitatively the compounds in the lycopene extract samples. The GCMS analysis was intended to determine the components contained in the lycopene extract from the tamarillo fruit. Figure 3 provides the spectrum of the analysis results from GCMS is the spectrum of the lycopene extract of the red tamarillo fruit and the reference spectrum of the Wiley data base, namely retinal. The retention time was 27,575 minutes

and the peak area was 35,062, indicating the interpretation of retinal compounds with the molecular formula $C_{20}H_{28}O$.

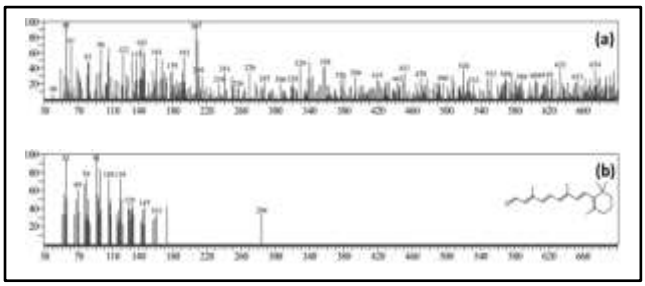

Figure 3. GCMS spectrum of (a) lycopene extract from red tamarillo and (b) retinal according to the Wiley Data Base reference spectrum.

Figure 4. Extracted lycopene from tamarillo

The results of the GCMS analysis showed that the chemical component referring to the constituents of the lycopene extract (Figure 4) was retinal as much as 0.21% which was equivalent to 0.21 g of lycopene in 100 g of red tamarillo powder. Retinal pigments, which are a group of carotenoids converted by plants, are the source of the pigments that give fruits and vegetables their red color. Meanwhile, Dewi, (2018) succeeded in isolating lycopene from dried tomatoes powder through an extraction process at a room temperature with hexane solvent, then lycopene crystals were obtained by antisolvent crystallization method, namely methanol under supersaturation conditions. The total isolated lycopene crystals were 2.25 mg/100 g of dried tomatoes powder.

Lycopene Chracterization by FTIR

Lycopene was successfully isolated from red tamarillo through an extraction process by maceration method for 3 days as shown in the FTIR spectrum in Figure 5. The FTIR spectrum of lycopene extracted from red tamarillo showed a spectrum that was in accordance with the FTIR spectrum of the lycopene standard.

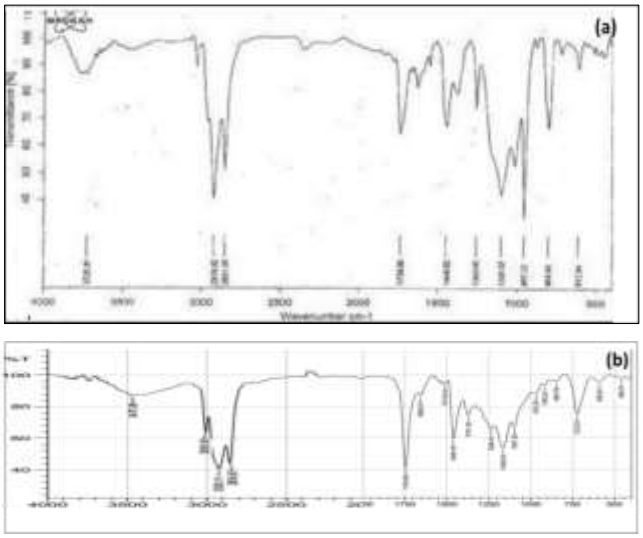

Figure 5. FTIR spectra of (a) standardized lycopene (Priam et al., 2017) and (b) lycopene extract from red tamarillo fruit.

The bottom half of the table shows the wavelength of the characterization of standard lycopene compounds and lycopene extracts using FTIR from various references. The wavelengths that are read each indicate the functional group in the chemical structure of the lycopene compound. As shown in Table 1, functional group analysis using Fourier Transform Infrared (FT-IR) spectroscopy at a wave number of 978.23 cm^{-1} shows the R-CH=CH-R group in the lycopene chain.

Table 1. Interpretation of FTIR spectra on lycopene characteristics from various references

Functional groups	Wavelength (cm^{-1})		
	Lycopene standard (Priam, Marcelin, Marcus, & Jô, 2017)	Lycopene from tomatoes (Tarigan, Sinaga, & Masyithah, 2016)	Lycopene from red tamarillo
R-CH=CH-R	957.23	956.72	978.23
$-CH_3-$		1373.36	1371.43
$-CH_2-$	1448	1456.30	1460.16
C=C	1744	1647.26 and 1743.71	1656.91 and 1745.64
C-H	2851 and 2918	2879.82 and 2910.68	2856.67 and 2926.11

The -CH_3- group was read at a wavelength of 1371.43 cm^{-1}. The wave number of 1460.16 cm^{-1} indicates the bending vibration of -CH_2- of the lycopene chain. While the C=C chain is shown at wavelengths of 1656.91 and 1745.64 cm^{-1} and the C-H chain is shown at wavelengths of 2856.67 and 2926.11 cm^{-1}. The IR spectra of the lycopene extracted from tamarillo through maceration at the room temperature matched well with the standard lycopene from reference.

CONCLUSION

In summary, the results of this study indicate that 0.21 g of lycopene was isolated from 100 g of dried red tamarillo fruit powder through an extraction process by maceration method using chloroform as a solvent, as shown from the results of the analysis by GCMS and the characterization of the structure of lycopene by FTIR.

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