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Utilization of Fatty Acids in Moringa Seed Oil as a Basic Material for Making Azelaic Acid

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Abstract: Moringa (*Moringa oleifera L.*) is a plant belonging to the moringaceae family. Moringa seeds contain oil which can be used as a raw material for making azelaic acid. Azelaic acid is used as an anti-acne in the pharmaceutical and cosmetic industries. Azelaic acid can be obtained from the oxidation process of fatty acids in moringa seed oil using potassium permanganate as an oxidizing agent. The resulting azelaic acid is in the form of white crystals with a conversion percentage of 72.17%. The results of the FT-IR characterization showed the formation of azelaic acid characterized by widening of the hydroxyl group at 3448.46 cm^{-1} followed by a shift of the ester group at absorption of 1633.46 cm^{-1}. The ester bond is strengthened at a wavelength of 1115.95 cm^{-1}.

Keywords: Moringa seed oil; Fatty acids; Azelaic acid

Introduction

One of the natural resources that is often found in various regions in Indonesia is the moringa plant (*Moringa oleifera L.*). Indonesia is currently one of the exporting countries for moringa seeds in the world (Sudaryanto et al., 2016). Plants belonging to the *moringaceae* family can grow in tropical and subtropical regions. (Manzoor et al., 2007) stated that the moringa plant is an important food commodity because all parts of the plant are edible and highly nutritious.

The leaves of the Moringa plant are often used by Indonesian people as vegetables (Laksmiani et al., 2020). Moringa leaves contain secondary metabolites including alkaloids, saponins, phenolics, tannins, flavonoids and steroids (Fachriyah et al., 2020). Meanwhile, Moringa seeds have the potential to be a source of vegetable oil because they contain very high fat. Moringa seeds also contain carbohydrates, proteins, fats, minerals and vitamins. Moringa seeds are good antioxidants because they can reduce oxidative damage accompanied by aging and cancer (Singh et al., 2009).

In general, vegetable oils contain mostly triglycerides up to 90% more and some mono and diglycerides (Wahyudi et al., 2018). According to (Febliza et al., 2020) oil is fat that is liquid at room temperature which is triglyceride from glycerol and fatty acids. Moringa seeds contain 30% to 42% oil or triglycerides which are rich in oleic acid. According to (Gunstone et al., 2007), the oleic acid content in Moringa seed oil reaches more than 72%. The fatty acid profile in vegetable oil will be affected by the age of the plant and weather conditions during the cooking process (Nguyen et al., 2020).

Fatty acids can be separated from their original vegetable oil through a hydrolysis process and produce fatty acids and glycerol (Brenna et al., 2020). Hydrolysis reactions can be carried out by reacting oil with water at high temperatures with the help of acid or base catalysts (Setiaji et al., 2013). The presence of acids or bases in the hydrolysis process of vegetable oil can significantly increase the rate of the decomposition reaction. According to (Sihotang, 2007), unsaturated fatty acids have double bonds which can be oxidized using potassium permanganate and ozone (O_3) or other peroxide compounds to produce azelaic acid. Meanwhile, according to (Godard et al., 2013) oleic acid can be oxidized to azelaic acid with pelargonic acid as a side product.

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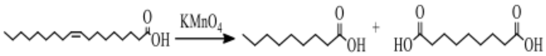


Azelaic acid or 1,9-nonanadinoic acid is a dicarboxylic acid which is one of the oleochemical derivatives. This acid can be produced from the process of oxidative breakdown of double bonds. Azelaic acid has many applications in the polymer sector, namely the polyester fiber industry, bio-lubricants and corrosion inhibitors (Benessere et al., 2015). Several studies have also examined the effectiveness of azelaic acid as anti-acne (anti-acne) and skin lightening in cosmetics because it has antibacterial, anti-inflammatory and anti-inflammatory activity (Tomić et al., 2019). According to (Worret et al., 2006), azelaic acid is natural and has no systemic side effects, so it is safe for the treatment of acne during pregnancy and breastfeeding. Azelaic acid is also bactericidal against a number of Gram-negative and Gram-positive microorganisms (Sieber et al., 2013). Currently azelaic acid has been used in the form of 20% cream in anti-acne products (Avilés et al., 2018).

The process of oxidation of fatty acids in Moringa seed oil is the process of breaking the alkene double bond in oleic acid, which is the highest component in Moringa seed oil. Oleic acid, which is one of the unsaturated fatty acids such as linoleic acid and linolenic acid, is very susceptible to oxidation (Brühl et al., 2022). (Kadhum et al., 2012) oxidized the double bond of oleic acid through an ozonolysis process at 150 °C. However, the resulting yield is still too low, namely 20%. In this research, the oxidation of fatty acids resulting from the hydrolysis of moringa seed oil will be carried out using $KMnO_4$ as an oxidizer. The permanganate ion (MnO_4^-) can oxidize the double bond (C=C) under acidic or basic conditions and produces a diol compound that has two hydroxyl groups. The double bonds of triacylglycerols can be epoxidized or converted to hydroxyl groups. This research consisted of several stages, namely extraction of moringa seed oil, hydrolysis of moringa seed oil, oxidation of fatty acids resulting from hydrolysis and product characterization. Characterization of compounds resulting from oxidation will be carried out using FTIR. Comparison of the synthesized FTIR spectra with reference data showing absorption bands in almost the same area, especially in the characteristic absorption band region can be an indicator of the success of the synthesis process. The study on the development of the conversion of moringa seed oil into azelaic acid is expected to increase the added value of moringa plants, which so far have not been optimally utilized.

Method

Extraction of Moringa Seed Oil

Moringa seed oil extraction was carried out using a soxhletation process with n-hexane for 6 hours. The extracted pure Moringa seed oil is evaporated in the evaporator and then tested for acid number and iodine number.

Hydrolysis

Moringa seed oil was hydrolyzed by incorporating 25 g of Moringa seed oil into a three-neck flask, then 50 mL of 50 mL of methanol along with 12% KOH was added. The mixture was refluxed at 60℃ for 90 minutes and 125 mL of distilled water and 31.25 mL of n-hexane were added, then extracted until 2 layers were formed (aqueous phase and organic phase). In the aqueous phase, 1 M sulfuric acid was added to pH 1 and 20 mL n-hexane was added. after that it was extracted again to form 2 layers. Free fatty acids are separated by a layer of water to be evaporated.

Oxidation

Oxidation of fatty acids resulting from hydrolysis was carried out by dissolving 6 g of fatty acids resulting from hydrolysis of Moringa seed oil into 50 mL of KOH solution (1.75 g) and stirring until completely mixed. 15.8 g of $KMnO_4$ was then dissolved in 200 mL of distilled water and stirred at 35 °C until completely dissolved. The salt solution of the mixed fatty acids was then added slowly at 75 °C for 30 minutes until the permanganate color disappeared. The mixture was then added with 23% $H2SO_4$ solution, and heated again at 90 °C until MnO_2 precipitate was formed and then filtered under hot conditions. The filtered filtrate was then boiled and added 50 mL of distilled water and filtered again in a hot state. The next step is that the mixture is concentrated to a volume of about 50 mL, then cooled with ice until a white solid is formed. The white solid was filtered with a Buchner filter and dried over silica gel in a desiccator and then analyzed by FT-IR.

Result and Discussion

Characterization of Moringa Seed Oil

Moringa seed oil produced from the soxhletation process is 36.63%. The results of the characterization of Moringa seed oil obtained data as in Table 1.

Table 1: Characterization Results of Moringa Seed Oil

Parameter	Before purification	After purification
Seed weight	0.23 g	-
Seed water content	6.73%	-
Oil yield	36.63%	-
Acid number	2.00 mgKOH/g	1.04 mgKOH/g
Saponification Number	97.41 mgKOH/g	140.50 mgKOH/g
Iodine Number	61.05 mg iod/100g	56.21 mg iod/100g

Figure 1. Moringa seed oil before and after refining

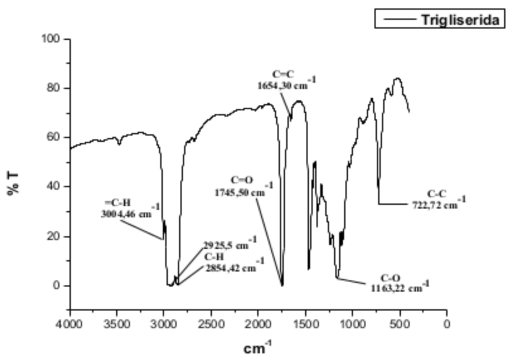


Figure 2. Infrared spectrum of moringa seed oil triglycerides

The results of the FT-IR analysis of Moringa seed oil show that there is an absorption in the C-H region at wave 3004.46 cm^{-1} indicating the presence of unsaturated groups, this is reinforced by the absorption area at 1654.30 cm^{-1} which is caused by the C=C group. Strong absorption in the 2925.50 and 2854.42 cm^{-1} areas indicates the presence of alkyl groups. In the FT-IR analysis of Moringa seed oil there is an ester group at the wavelength of 1745.50 cm^{-1} and is strengthened at 1163.22 cm^{-1}. The spectrum shown at wave 722.72 cm^{-1} is the strain of saturated carbon-carbon bonds.

Hydrolysis

The reaction of triglycerides with KOH will produce potassium salts and glycerol. Separation of potassium salt and glycerol can be done by shaking using a separatory funnel. The potassium salt formed is in the aqueous phase. Non-polar compounds or impurities are in the organic phase. The addition of H_2SO_4 is carried out until the pH of the salt becomes one, the aim is to convert the salt into fatty acids.

The fatty acids obtained were separated from other compounds by adding n-hexane and shaking with a separatory funnel. The top layer is a fatty acid dissolved in n-hexane, while the bottom layer is a polar compound such as methanol, K_2SO_4 and glycerol. The fatty acids dissolved in n-hexane are separated by evaporation.

Characterization of Oxidation Products

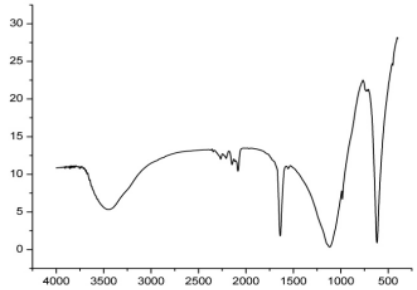
The decrease in iodine number is an indicator that shows the success of the oxidation solution which is carried out by reducing the number of double bonds present in the oxidation products. The iodine number is a number used to reflect unsaturation in fatty acids. The higher the iodine number, the more double bonds there are in the sample (Handayani et al., 2015). The iodine number test on the sample before and after oxidation is used to determine changes in unsaturation in the sample.

The fatty acid iodine value of Moringa seed oil before oxidation was 51.09 mg iodine/100g and after oxidation it was 14.79 mg iodine/100g. The decrease in iodine number after the oxidation process indicates the release of the double bonds that occur during the process. According to (Chebet et al., 2016) unsaturated fatty acids are able to bind iodine and form saturated compounds which result in a decrease in iodine number. The concentration of components and aromas in the sample will change during the oxidation process (Zhang et al., 2021).

Figure 3. Azelaic acid crystals

Separation of azelaic acid and pelargonic acid formed from the oxidation process is carried out by filtering the sample. Azelaic acid and pelargonic acid can be produced from oxidative cleavage of the double bond of oleic acid using an oxidizing agent and catalyst (Masyithah et al., 2018).

The azelaic acid obtained from this study is in the form of white crystals. Azelaic acid crystals from the oxidation of fatty acids in Moringa seed oil can be seen in Figure 3. The FT-IR spectrum of the synthesized azelaic acid can be seen in Figure 4.



Wave Number (cm-1)

Figure 4. FT-IR spectrum of the synthesized azelaic acid

Comparison of the FT-IR spectrum between the synthesized azelaic acid and the fatty acids of moringa seed oil can be seen in Table 2.

Table 2: The Binding Sites on Fatty Acids and Oxidation Result

Bond Type	Wave Number (cm^{-1})	
	Fatty Acid	Azelaic Acid
-OH	3465.12	3448.46
=C-H	3004.68	-
-C-H	2926.00	-
	2854.48	-
C=O	1747.29	1633.46
C=C	1638.00	-
C-O	1164.05	1115.95
C-C	722.90	618

Based on Figure 4, it can be seen that the absorption for the double bond at the wavelength of 3004.68 and 1638 cm^{-1} is reduced. This indicates that most of the double bonds in fatty acids have undergone a double bond breaking process. (Maisaroh et al., 2016) stated that the absence of absorption of unsaturated carbon groups indicates that there has been a reaction to the double bonds present in the fatty acids, which is indicated by a low iodine number. The absorption of the hydroxyl group is widened in the azelaic acid spectrum at a wavelength of 3448.46 cm^{-1}. The widening of the hydroxyl group was followed by a shift in the ester bond which appeared at the absorption area of 1633.46 cm^{-1} and was strengthened at the absorption area of 1115.95 cm^{-1}.

Conclusion

Azelaic acid can be synthesized from moringa seed oil using $KMnO_4$ oxidizer. Formation of azelaic acid, indicated by the formation of white crystals. The successful conversion of azelaic acid is also proven by the increasing hydroxyl number and decreasing iodine number after being oxidized. Functional groups in the results of FT-IR analysis of oxidation products showed that azelaic acid has been formed. The formation of azelaic acid is characterized by a decrease in the double bond and widening of the hydroxyl group in the azelaic acid spectrum which is seen in the region of 3448.46 cm^{-1} and is followed by a shift in the ester group at absorption of 1633.46 cm^{-1}. The ester bond is strengthened at a wavelength of 1115.95 cm^{-1}.

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References

Avilés, F., Caracundo, V., & Feijoo, D. (2018). *Synthesis of azelaic acid from castor oil* (Issue October). https://doi.org/10.13140/RG.2.2.21279.61602

Benessere, V., Cucciolito, M. E., De Santis, A., Di Serio, M., Esposito, R., Ruffo, F., & Turco, R. (2015). Sustainable Process for Production of Azelaic Acid Through Oxidative Cleavage of Oleic Acid. *JAOCS, Journal of the American Oil Chemists' Society*, *92*(11–12), 1701–1707. https://doi.org/10.1007/s11746-015-2727-z

Brenna, E., Colombo, D., Di Lecce, G., Gatti, F. G., Ghezzi, M. C., Tentori, F., Tessaro, D., & Viola, M. (2020). Conversion of oleic acid into azelaic and pelargonic acid by a chemo-enzymatic route. *Molecules*, *25*(8), 1882. https://doi.org/10.3390/molecules25081882

Brühl, L., Bonte, A., N'Diaye, K., & Matthäus, B. (2022). Oxidation of Cyclo-Lino Peptides in Linseed Oils during Storage. *European Journal of Lipid Science and Technology*, 2200137. https://doi.org/10.1002/ejlt.202200137

Chebet, J., Kinyanjui, T., & Cheplogoi, P. K. (2016). Impact of frying on iodine value of vegetable oils before and after deep frying in different types of food in Kenya. *Journal of Scientific and Innovative Research*, *5*(5), 193–196. https://doi.org/10.31254/jsir.2016.5508

Fachriyah, E., Kusrini, D., Haryanto, I. B., Wulandari, S. M. B., Lestari, W. I., & Sumariyah, S. (2020). Phytochemical Test, Determination of Total Phenol, Total Flavonoids and Antioxidant Activity of Ethanol Extract of Moringa Leaves (Moringa oleifera Lam. *Jurnal Kimia Sains Dan Aplikasi*, *23*(8), 290–294. https://doi.org/10.14710/jksa.23.8.290-294

Febliza, A., Okatariani, O., & Putri, A. M. (2020). Kualitas Minyak Blend Kelapa Kopra dan Minyak Kelapa Sawit ditinjau dari Kadar Air, Kadar Asam Lemak

Bebas dan Bilangan Peroksida. *Jurnal Riset Kimia*, *11*(1), 1–8. https://doi.org/10.25077/jrk.v11i1.347

Godard, A., De Caro, P., Thiebaud-Roux, S., Vedrenne, E., & Mouloungui, Z. (2013). New environmentally friendly oxidative scission of oleic acid into azelaic acid and pelargonic acid. *JAOCS, Journal of the American Oil Chemists' Society*, *90*(1), 133–140. https://doi.org/10.1007/s11746-012-2134-7

Gunstone, F. D., & Harwood, J. L. (2007). Lipid Handbook. In *Nature* (3rd ed., Vol. 231, Issue 5304). Chapman and Hall. https://doi.org/10.1038/231541b0

Handayani, S. S., Gunawan, E. R., Kurniawati, L., Murniati, M., & Budiarto, L. H. (2015). Analisis Asam Lemak Omega-3 dari Minyak Kepala Ikan Sunglir (Elagatis bipinnulata) melalui Esterifikasi Enzimatik. *Jurnal Natur Indonesia*, *15*(2), 75. https://doi.org/10.31258/jnat.15.2.75-83

Kadhum, A. A. H., Wasmi, B. A., Mohamad, A. B., Al-Amiery, A. A., & Takriff, M. S. (2012). Preparation, characterization, and theoretical studies of azelaic acid derived from oleic acid by use of a novel ozonolysis method. *Research on Chemical Intermediates*, *38*(2), 659–668. https://doi.org/10.1007/s11164-011-0406-8

Laksmiani, N. P. L., Widiantara, I. W. A., Adnyani, K. D., & Pawarrangan, A. B. S. (2020). OPTIMASI METODE EKSTRAKSI KUERSETIN DARI DAUN KELOR (Moringa oleifera L.). *Jurnal Kimia*, 19. https://doi.org/10.24843/jchem.2020.v14.i01.p04

Maisaroh, M., Susetyo, I. B., & Rusmandana, B. (2016). Sintesis Asam 9,10-Dihidroksi Stearat (DHSA) Melalui Hidrolisa Epoksida dari Oksidasi Asam Oleat dengan Asam Performat. *REAKTOR*, *16*(2), 57. https://doi.org/10.14710/reaktor.16.2.57-64

Manzoor, M., Anwar, F., Iqbal, T., & Bhanger, M. I. (2007). Physico-chemical characterization of moringa concanensis seeds and seed oil. *JAOCS, Journal of the American Oil Chemists' Society*, *84*(5), 413–419. https://doi.org/10.1007/s11746-007-1055-3

Masyithah, Z., & Ginting, A. (2018). Optimization of a two-step method to synthesize azelaic acid from oleic acid. *Oriental Journal of Chemistry*, *34*(3), 1249–1256. https://doi.org/10.13005/ojc/340307

Nguyen, Q. H., Talou, T., Evon, P., Cerny, M., & Merah, O. (2020). Fatty acid composition and oil content during coriander fruit development. *Food Chemistry*, *326*. https://doi.org/10.1016/j.foodchem.2020.127034

Setiaji, B., & Syoufian, A. (2013). Fractionation Of Medium Chain Fatty Acid Ethyl Esters From Virgin Coconut Oil Via Transesterification and Distillation Under Reduced Pressure. *International Journal of Basic & Applied Sciences*, *13*(01), 60–64.

Sieber, M. A., & Hegel, J. K. E. (2013). Azelaic acid: Properties and mode of action. *Skin Pharmacology and Physiology*, *27*(SUPPL.1), 9–17. https://doi.org/10.1159/000354888

Sihotang, H. (2007). Pembuatan Ester 1,9-Nonanil Dilaktat Melalui Reaksi Esterifikasi Antara Asam Laktat Dengan 1,9-Nonanadiol. *Jurnal Sains Kimia*, *1*(11), 1–4.

Singh, B. N., Singh, B. R., Singh, R. L., Prakash, D., Dhakarey, R., Upadhyay, G., & Singh, H. B. (2009). Oxidative DNA damage protective activity, antioxidant and anti-quorum sensing potentials of Moringa oleifera. *Food and Chemical Toxicology*, *47*(6), 1109–1116. https://doi.org/10.1016/j.fct.2009.01.034.

Sudaryanto, Herwanto, T., & Putri, S. H. (2016). Antioxidant Activity in Oil Seeds Moringa (Moringa oleifera l.) Soxhletation Method with using Solvents N-hexane, Methanol, and Ethanol. *Jurnal Teknotan*, *10*(2), 16–21.

Tomić, I., Juretić, M., Jug, M., Pepić, I., Cetina Čižmek, B., & Filipović-Grčić, J. (2019). Preparation of in situ hydrogels loaded with azelaic acid nanocrystals and their dermal application performance study. *International Journal of Pharmaceutics*, *563*, 249–258. https://doi.org/10.1016/j.ijpharm.2019.04.016

Wahyudi, W., I.N.G., W., A., W., & W. (2018). Improving Vegetable Oil Properties by Transforming Fatty Acid Chain Length in Jatropha Oil and Coconut Oil Blends. *Energies*, *11*, 394. https://doi.org/10.3390/en11020394.

Worret, W.-I., & Fluhr, J. W. (2006). Acne therapy with topical benzoyl peroxide, antibiotics and azelaic acid. *Jddg*, *4*(4), 293–300. https://doi.org/10.1111/j.1610-0387.2006.05931.x

Zhang, Y., Luo, J., & Zeng, F. (2021). Volatile composition analysis of tree peony (Paeonia section Moutan DC.) seed oil and the effect of oxidation during storage. *Journal of Food Science*, *86*(8), 3467–3479. https://doi.org/10.1111/1750-3841.15790

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