PENENTUAN KOMPONEN DAN KOMPOSISI SENYAWA SERTA KARAKTERISTIK AROMA MINYAK ROSEMARY (*Rosmarinus officinalis* L.) HASIL OKSIDASI MENGGUNAKAN ALIRAN GAS OKSIGEN

SKRIPSI

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Sarjana Sains Program Studi Kimia

Diusulkan oleh :

Alumina Zenith Female Nuryana

1405283

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Perubahan Komponen dan Komposisi Senyawa serta Karakteristik Aroma Minyak Rosemary (*Rosmarinus officinalis* L.) Hasil Oksidasi Menggunakan Aliran Gas Oksigen

Oleh
Alumina Zenith Female Nuryana

Sebuah skripsi yang diajukan untuk memenuhi salah satu syarat memperoleh gelar Sarjana Sains pada Fakultas Pendidikan Matematika dan Ilmu Pengetahuan Alam

Universitas Pendidikan Indonesia
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ALUMINA ZENITH FEMALE NURYANA

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disetujui dan disahkan oleh:

Pembimbing I,

Prof. Dr. H. R. Asep Kadarohman, M.Si
NIP. 196305091987031002

Pembimbing II,

Dr. Siti Aisyah, M.Si
NIP. 197509302001122001

Mengetahui,
Ketua Departemen Pendidikan Kimia

Dr. rer. nat. H. Ahmad Mudzakir, M.Si
NIP. 196611211991031002

ABSTRAK

Karakteristik aroma minyak atsiri dipengaruhi oleh kandungan senyawa yang mengandung oksigen. Minyak atsiri dengan kandungan senyawa yang mengikat oksigen lebih tinggi memiliki kekuatan *fragrance* yang lebih kuat dan memiliki potensi kegunaan yang lebih luas. Peningkatan kadar senyawa yang mengikat oksigen dalam minyak atsiri dapat dilakukan dengan oksidasi menggunakan sistem *batch*. Penelitian ini bertujuan untuk menganalisa perubahan yang terjadi dalam minyak atsiri yang dioksidasi dan pengaruhnya terhadap karakteristik aromanya. Dalam penelitian ini telah dilakukan oksidasi menggunakan aliran O_2 terhadap minyak rosemary menggunakan sistem *batch* pada suhu 115℃. Analisis komposisi senyawa minyak rosemary dilakukan dengan menggunakan *Gas Chromatography-Mass Spectrometry* (GC-MS), sedangkan analisa karakteristik aroma dilakukan dengan mnggunakan *software Perfumer's Workbook.* Hasil analisis GC-MS menunjukkan bahwa kandungan senyawa minyak rosemary terdiri atas α-pinen, 1,8-sineol, linalool, camphor, linalil asetat, isobornil asetat, dan β-terpinil asetat dengan kadar masing-masing senyawa di atas 1%. Oksidasi terhadap minyak rosemary menghasilkan perubahan komposisi dan komponen senyawa utamanya. Jumlah kandungan senyawa yang mengikat oksigen pada minyak rosemary hasil oksidasi mengalami peningkatan, termasuk didalamnya yaitu kandungan senyawa camphor, *cis*-linalool oksida, *trans*-linalool oksida, dan isobornil asetat. Selain itu ditemukan juga keberadaan puncak senyawa baru yang mengandung oksigen dengan struktur yang diduga sebagai fenchyl asetat, α-terpinil asetat dan epoksilinalool dalam minyak rosemary hasil oksidasi. Peningkatan kandungan camphor diduga berasal dari oksidasi α-pinen, sementara peningkatan kandungan linalool oksida diduga berasal dari oksidasi senyawa linalool. Selain itu peningkatan isobornil asetat dan pembentukan senyawa fenchyl asetat dan α-terpinil asetat diduga berasal dari reaksi isomerisasi linalil asetat. Analisis karakteristik aroma menunjukkan adanya perubahan karakter minyak rosemary hasil oksidasi. Karakteristik aroma, minyak rosemary sebelum oksidasi didominasi oleh 5 karakter aroma yaitu aroma konifer, *iceberg*, *herb*, *light chemical floral* dan *citrus*. Sedangkan karakteristik aroma minyak rosemary hasil oksidasi menunjukkan peningkatan karakter aroma konifer, *iceberg* dan *herb* dan penurunan karakter aroma *light chemical floral, woody, citrus, zolvent*, dan *fruity*.

Kata kunci: *minyak rosemary, Rosemarinus officinalis L., oksidasi, minyak atsiri, sistem batch, refluks, oksigen*.

ABSTRACT

The fragrance of an essential oil is influenced by the amount of its oxygen-containing compounds. Essential oil with higher oxygen-containing compounds usually has strong fragrance with plenty of application possibilities. Oxidation using batch system reaction can be used to increase the amount of oxygen-containing compounds in essential oils. This research aim is to analyse components and composition in oxidized rosemary oil and the effects to its odour characteristics. In this research, rosemary crude oil was oxidized by pure O_2 under thermal condition, 115℃. Rosemary oil components and composition was analysed by Gas Chromatography-Mass Spectrometry (GC-MS) and its odour characteristics was determined using Perfumer's Workbook software. Based on GC-MS analysis, main compounds of rosemary crude oil are α-pinene, 1,8-cineole, linalool, camphor, linalyl acetate, isobornyl acetate, and β-terpinyl acetate. Rosemary oil oxidation resulting changes in its components and composition. The amount of oxygen-containing compounds such as camphor, cis-linalool oxide, trans-linalool oxide, and isobornyl acetate were increased. The changes of composition was proven by the presence of new peak in oxidized rosemary's chromatogram, those suspected new compounds are fenchyl acetate, α-terpinyl acetate and epoxylinalool. Some compounds were suspected to oxidized and form another compound, such as α-pinene oxidized into camphor, while cis-linalool oxide, trans-linalool oxide and epoxylinalool formed from linalool oxidation, and linalyl acetate was suspected to isomerized resulting isobornyl acetate to increased, linalyl acetat was also suspected to formed new compounds such as fenchyl acetate and α-terpinyl acetate. Oxidized rosemary showed changes in several main odour characteristics. Rosemary crude oil consists of 5 main odour characteristics such as konifer, iceberg, herb, light chemical floral and citrus. In oxidized rosemary oil, several odour characteristics such as konifer, iceberg, and herb percentage were increased, while light chemical floral, woody, citrus, solvent, and seemed to be decreased.

Keywords: *rosemary oil, Rosemarinus officinalis L., oxidation, essential oils, batch system, reflux, oxygen*.

DAFTAR ISI

DAFTAR GAMBAR

DAFTAR TABEL

DAFTAR LAMPIRAN

DAFTAR PUSTAKA

Althea Press. (2013). *ESSENTIAL OILS FOR-BEGINNERS the guide to get started with essential oils and aromatherapy*. New York: Callisto Media Inc.

Bogert, M. T. (1903). *Organic Chemistry. Journal of the American Chemical Society* (8th ed., Vol. 25). Boston: Cengage Learning.

Bråred Christensson, J., Andersen, K. E., Bruze, M., Johansen, J. D., Garcia-Bravo, B., Gimenez Arnau, A., … White, I. R. (2012). Air-oxidized linalool-a frequent cause of fragrance contact allergy. *Contact Dermatitis*, *67*(5), 247–259. doi: https://doi.org/10.1111/j.1600-0536.2012.02134.x

Browne, A. W. (2019). *Industrial Chemistry*. *Journal of Education* (Vol. 91). India: Krishna Prakashan Media.

Carberry, J. J. (1976). *Chemical and catalytic reaction engineering*. *McGraw-Hill*. New York.

Clark, B. C., Chamblee, T. S., & Iacobucci, G. A. (1989). Micellar-Induced Selectivity and Rate Enhancement in the Acid-Catalyzed Cyclization and Rearrangement of Monoterpenes. The Solvolysis of Linalyl and Geranyl Acetates. *Journal of Organic Chemistry*, *54*(5), 1032–1036. doi: https://doi.org/10.1021/jo00266a007

Demyttenaere, J. C. R. (2001). Biotransformation of terpenoids by microorganisms. *Studies in Natural Products Chemistry*, *25*(PART F), 125–178. doi: https://doi.org/10.1016/S1572-5995(01)80007-7

Dowthwaite, S. V. (2012). *The Art and Technology of Perfumery*. Bangkok, Thailand.

Elliott, J. (1976). NASA's advanced control law program for the F-8 digital fly-by-wire aircraft. *1976 IEEE Conference on Decision and Control Including the 15th Symposium on Adaptive Processes*, 1–5. doi: https://doi.org/10.1109/CDC.1976.267694

Ėmanuėl′, N. M. (Nikolaĭ M., Zaikov, G. E. (Gennadiĭ E., & Maĭzus, Z. K. (Zinaida K. (1984). *Oxidation of organic compounds : medium effects in radical reactions*. Amsterdam: Elsevier.

Ferhat, M. A., Meklati, B. Y., Smadja, J., & Chemat, F. (2006). An improved microwave Clevenger apparatus for distillation of essential oils from orange

peel. *Journal of Chromatography A*, *1112*(1–2), 121–126. doi: https://doi.org/10.1016/j.chroma.2005.12.030

Fessenden, F. R. J. dan J. S. (2010). *Dasar-Dasar Kimia Organik*. Boston, United States: W. Grant Press.

H, W. (2007). *Antioksidan Alami dan Radikal Bebas*. *Cetakan Kelima*. Bandung: Kanisius.

Hagvall, L., Berglund, V., & Christensson, J. B. (2015). Air-oxidized linalyl acetate - An emerging fragrance allergen? *Contact Dermatitis*, *72*(4), 216–223. doi: https://doi.org/10.1111/cod.12350

Herbert, C. G., & Johnstone, R. A. W. (2003). *Mass Spectrometry Basics - Chapter 10*. *Mass Spectrometry Basics*. United States: CRC Press.

Hickman, D. A. (2011). *Chemical Reactor Design, Optimization, and Scaleup By E. Bruce Nauman*. *AIChE Journal* (Vol. 57). United States: McGraw Hill Professional.

Hyldgaard, M., Mygind, T., & Meyer, R. L. (2012). Essential oils in food preservation: Mode of action, synergies, and interactions with food matrix components. *Frontiers in Microbiology*, *3*(JAN). doi: https://doi.org/10.3389/fmicb.2012.00012

Jahnke, J. A. (2013). *Continuous Emission Monitoring Systems Worldwide*. United Kingdom.

Jiang, Y., Wu, N., Fu, Y. J., Wang, W., Luo, M., Zhao, C. J., … Liu, X. L. (2011). Chemical composition and antimicrobial activity of the essential oil of Rosemary. *Environmental Toxicology and Pharmacology*, *32*(1), 63–68. doi: https://doi.org/10.1016/j.etap.2011.03.011

Julianto, T. S. (2016). *Minyak Atsiri Bunga Indonesia*. Daerah Istimewa Yogyakarta: Deepublish.

Kitson, F. G., Larsen, B. S., & McEwen, C. N. (1996). *Gas chromatography and mass spectrometry : a practical guide*. Cambridge: Academic Press.

Koensoemardiyah. (2010). *A to Z Minyak Atsiri*. Bandung: Penerbit Andi.

Lehman, J. W. (1999). *Operational Organic Chemistry: A Problem-Solving Approach to the Laboratory Course*. United States: Prentice Hall.

Meyer, M. R. (2016). *Hans-Joachim Hübschmann: Handbook of GC-MS:*

fundamentals and applications, 3rd ed. Analytical and Bioanalytical Chemistry (Vol. 408). New Jersey: John Wiley & Sons.

Mirata, M. A., Wüst, M., Mosandl, A., & Schrader, J. (2010). Linalool biotransformation with fungi. *Expression of Multidisciplinary Flavour Science*, *10*(4), 349–353.

Mitchell, H. K. (1945). *Organic chemistry*. *Journal of Heredity* (Vol. 36). New Jersey: John Wiley & Sons.

Moreira, M. R., Alvarez, M. V., & Ponce, A. G. (2016). *Essential oils*. *Postharvest Management Approaches for Maintaining Quality of Fresh Produce*. Amsterdam: Elsevier Inc.

Moss, M., Cook, J., Wesnes, K., & Duckett, P. (2003). Aromas of rosemary and lavender essential oils differentially affect cognition and mood in healthy adults. *International Journal of Neuroscience*, *113*(1), 15–38. doi: https://doi.org/10.1080/00207450390161903

Muff, B. J. (1947). *General Chemistry*. *Nature* (Vol. 159). doi: https://doi.org/10.1038/159590b0

Nakatsu, T., Andrew T. Lupo, J., John W. Chinn, J., & Raphael K.L. Kang. (2000). Biological Activity of Essential Oils and Their Constituents. *Studies in Natural Products Chemistry*, *21*, 826.

Olmedo, R. H., Asensio, C. M., & Grosso, N. R. (2015). Thermal stability and antioxidant activity of essential oils from aromatic plants farmed in Argentina. *Industrial Crops and Products*, *69*, 21–28. doi: https://doi.org/10.1016/j.indcrop.2015.02.005

Özcan, M. M., & Chalchat, J. C. (2008). Chemical composition and antifungal activity of rosemary (Rosmarinus officinalis L.) oil from Turkey. *International Journal of Food Sciences and Nutrition*, *59*(7–8), 691–698. doi: https://doi.org/10.1080/09637480701777944

Pateiro, M., Barba, F. J., Domínguez, R., Sant'Ana, A. S., Mousavi Khaneghah, A., Gavahian, M., … Lorenzo, J. M. (2018). Essential oils as natural additives to prevent oxidation reactions in meat and meat products: A review. *Food Research International*, *113*, 156–166. doi: https://doi.org/10.1016/j.foodres.2018.07.014

Raharjo, S. (2006). *Kerusakan Oksidatif Pada Makanan*. Daerah Istimewa Yogyakarta: UGM PRESS.

Ramishvili, T. M., Yushchenko, V. V., & Charkviani, M. K. (2007). Catalytic conversions of linalool and linalyl acetate over large-pore zeolites and mesoporous MCM-41. *Moscow University Chemistry Bulletin*, *62*(4), 180–186. doi: https://doi.org/10.3103/s0027131407040025

Riley, P. A. (2004). Oxygen free radicals in tissue damage. *Journal of Chemical Neuroanatomy*, *8*(2), 148–149. doi: https://doi.org/10.1016/0891-0618(95)90019-5

Robertson, G. R. (2009). Technique of organic chemistry. volume VII: organic solvents. *Journal of Chemical Education*, *32*(11), 602. doi: https://doi.org/10.1021/ed032p602.2

Rusli, M. S. (2010). *Sukses Memproduksi Minyak Atsiri*. *Aegypti Fact Sheet*. Bandung: AgroMedia Pustaka.

Sayorwan, W., Ruangrungsi, N., Piriyapunyporn, T., Hongratanaworakit, T., Kotchabhakdi, N., & Siripornpanich, V. (2013). Effects of inhaled rosemary oil on subjective feelings and activities of the nervous system. *Scientia Pharmaceutica*, *81*(2), 531–542. doi: https://doi.org/10.3797/scipharm.1209-05

Schmal, M. (2010). *Chemical reaction engineering. Essentials, Exercises and Examples*. *Chemical Engineering Science*. United States: CRC Press. doi: https://doi.org/10.1016/0009-2509(64)85017-X

Shunying, Z., Yang, Y., Huaidong, Y., Yue, Y., & Guolin, Z. (2005). Chemical composition and antimicrobial activity of the essential oils of Chrysanthemum indicum. *Journal of Ethnopharmacology*, *96*(1–2), 151–158. doi: https://doi.org/10.1016/j.jep.2004.08.031

Sköld, M., Hagvall, L., & Karlberg, A. T. (2008). Autoxidation of linalyl acetate, the main component of lavender oil, creates potent contact allergens. *Contact Dermatitis*, *58*(1), 9–14. doi: https://doi.org/10.1111/j.1600-0536.2007.01262.x

Tancini, F., Wu, Y. L., Schweizer, W. B., Gisselbrecht, J. P., Boudon, C., Jarowski, P. D., … Diederich, F. (2012). 1,1-Dicyano-4-[4-(diethylamino)phenyl]buta-

1,3-dienes: Structure-property relationships. *European Journal of Organic Chemistry*, (14), 2756–2765. doi: https://doi.org/10.1002/ejoc.201200111

Tribuzi, G., & Laurindo, J. B. (2016). Dehydration and Rehydration of Cooked Mussels. *International Journal of Food Engineering*, *12*(2), 2848. doi: https://doi.org/10.1515/ijfe-2015-0275

Xu, R., Ye, Y., & Zhao, W. (2011). *Introduction to Natural Products Chemistry*. *Introduction to Natural Products Chemistry*. United States: CRC Press.

Yamada, S. (2006). Researches on Camphor, Borneol and Their Allied Substances (Continued and Concluded) . *Bulletin of the Chemical Society of Japan*, *16*(9), 336–347. doi: https://doi.org/10.1246/bcsj.16.336

Yuliani, S., & Satuhu, S. (2012). *Panduan Lengkap Minyak Atsiri*. (B. Prasetya W., Ed.). Jakarta: Penebar Swadaya.

Zanella, C. A., Treichel, H., Cansian, R. L., & Roman, S. S. (2012). The effects of acute administration of the hydroalcoholic extract of rosemary (Rosmarinus officinalis L.) (Lamiaceae) in animal models of memory. *Brazilian Journal of Pharmaceutical Sciences*, *48*(3), 389–397. doi: https://doi.org/10.1590/s1984-82502012000300005