ALKIMIA : Jurnal Ilmu Kimia dan Terapan

ARTICLES

View Article Online
View Journal | View Issue

OPEN ACCESS

Synthesis and Characterization of Vertosin Schiff Base with Temperature and Time Optimization

Revina Yuliastuti[a], Nurlela[a]*, Devy Susanty[a], Nina Ariesta[a]

Abstract. Perfume is a product designed to mask or eliminate unpleasant odors and impart a pleasant fragrance to the body, rooms, or various objects. One synthetic ingredient commonly added to fragrances is a schiff base, a compound formed through a condensation reaction between an aldehyde and an amine. Schiff bases enhance the chemical and thermal stability of fragrances due to their distinctive and long-lasting aroma. One such compound, vertosin, is known for its strong green, citrus, and herbal scent and is suitable for personal care products. This research aimed to synthesize vertosin via a condensation reaction between triplal and methyl anthranilate under varying temperatures and durations, and to characterize the resulting product using Gas Chromatography-Mass Spectrometry (GCMS). The study employed a combination research method consisting of synthesis, characterization, and data analysis. The synthesis was conducted at temperatures of 90, 100, and 110°C for 30 and 60 minutes. Characterization included organoleptic tests (color and odor), density and specific gravity measurements, and compound confirmation using GCMS. Data analysis was performed using two-way ANOVA. The results showed that the optimal condition for vertosin synthesis was at 110°C for 60 minutes, yielding the highest concentration. Mass spectrometric fragmentation patterns confirmed the product as methyl 2-[[(E)-2,4-dimethyl-1-cyclohex-3-enylidene)methyl]amino]benzoate with a molecular weight of 271 g/mol. The synthesized vertosin had a green, lily, orange blossom, fruity, floral scent, an orange color, and average density and specific gravity values of 1.092 and 1.094, respectively, which were in accordance with standard specifications

Keywords : Schiff base, vertosin, fragrance synthesis, condensation reaction, gas chromatography-mass spectrometry

[a]*Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Nusa Bangsa. Jl. KH. Sholeh Iskandar Km 4, Bogor 16166, West Java, Indonesia*

Correspondence and requests for materials should be addressed to Nurlela (email: nnurlela16@gmail.com).

Introduction

Fragrances or perfumes are an integral part of daily life, widely used to eliminate unpleasant odors and impart pleasant scents to the body, rooms, and various objects [1]. These products are typically formulated from fragrance bases—complex mixtures of aromatic compounds, both natural and synthetic, that create characteristic scents when blended [2, 3]. One class of compounds commonly added to enhance fragrance performance and longevity is schiff bases.

Schiff bases are compounds formed through the condensation of aldehydes and primary amines, producing water as a by-product. In the fragrance industry, schiff bases derived from methyl anthranilate and aromatic aldehydes such as triplal are of particular interest due to their ability to improve the chemical stability and volatility profiles of perfume formulations [4-6]. One notable schiff base is vertosin, which exhibits a distinctive green, citrus, and herbal aroma—traits that are valuable in body care and home fragrance products [7, 8].

Vertosin is synthesized via a condensation reaction between methyl anthranilate and triplal, a process that is influenced significantly by reaction parameters such as temperature and time [9]. Optimization of these parameters is critical, as they directly affect the yield and purity of the product [3, 10]. While previous research has explored schiff base synthesis for related compounds like aurantiol [3, 11], the optimal synthesis conditions for vertosin remain unexplored.

Moreover, schiff bases are not only valued for their olfactory properties but also for their biological activities, such as antimicrobial, antioxidant, and anti-inflammatory effects, which could potentially contribute to fragrance longevity and stability [12, 13]. Characterization of these compounds is commonly carried out using Gas Chromatography–Mass Spectrometry (GC–MS), which enables detailed analysis of their molecular structure and fragmentation patterns [5].

This study aims to synthesize vertosin through a condensation reaction using varying temperatures (90°C, 100°C, and 110°C) and durations (30 and 60 minutes), followed by characterization using GC–MS, organoleptic tests, refractive index, density, and specific gravity measurements. Two-way ANOVA will be employed to determine the statistically significant effects of temperature and time on the yield and quality of the synthesized schiff base.

While the synthesis and characterization of other schiff bases, such as aurantiol and verdantiol, have been documented, comprehensive optimization studies specifically targeting vertosin have not yet been reported. This study is the first to investigate the optimal synthesis conditions for vertosin using a systematic statistical approach and to characterize the compound's GC–MS fragmentation pattern in detail. The findings will provide valuable insights into improving the formulation of stable and long-lasting fragrances in the cosmetic and fragrance industries.

Experimental

Materials and Instruments

The materials used in this study included triplal, methyl anthranilate, distilled water, 95% ethanol, and 99% ethanol. Instruments employed comprised beakers, spatulas, test tubes, droppers, thermometers, magnetic stirrers, analytical balances, hotplates, retort stands with clamps, test papers, pycnometers, vials, and a Gas Chromatography-Mass Spectrometry (GC-MS) Agilent Technologies 5975C.

Research Methodology

This research employed a mixed-method approach, integrating both qualitative and quantitative analysis, as the synthesis of the schiff base compound vertosin has been rarely reported. The study consisted of three main stages: synthesis, characterization, and data analysis. The synthesis process involved weighing the starting materials (methyl anthranilate and triplal) followed by a condensation reaction carried out at varying temperatures (90°C, 100°C, and 110°C) and reaction times (30 minutes and 60 minutes). Characterization of the synthesized compound included organoleptic tests (color and odor), density and specific gravity measurements, and compound confirmation via GC-MS. Data were analyzed using two-way ANOVA.

Synthesis of Schiff Base Vertosin. Triplal (5 g) and methyl anthranilate (6.5 g) were weighed according to the theoretical molar ratio of 100:130 [14]. The components were mixed and stirred until a color change from colorless to yellow was observed, with the reaction time recorded. The mix-

ture was stirred using a magnetic stirrer and heated on a hotplate at various temperatures (90°C, 100°C, and 110°C) for either 30 or 60 minutes, depending on the treatment group.

Organoleptic Tests. Organoleptic properties including odor and color were assessed. Odor test: paper strips were labeled and dipped in either the sample or reference solutions to a depth of 1 cm. After drying, the strips were placed on an acrylic board, and the odors were compared by five trained panelists in a well-ventilated room. Color test: sample and reference solutions were poured into test tubes to ¾ full. Color comparisons were made visually from vertical (top view) and horizontal (side view) angles against a white background.

Density and Specific Gravity Measurements. Measurements were conducted using a 10 mL pycnometer at room temperature (25°C). First, the pycnometer was calibrated and filled with distilled water; the mass was recorded. It was then emptied, cleaned with ethanol, and dried. The schiff base vertosin sample was measured using the same procedure.

Volume determination of pycnometer (Vp):

$$Vp\ (mL) = \frac{b - a)}{\rho air} \qquad (1)$$

Where, a : mass of empty pycnometer (g); b : mass of pycnometer with water (g); ρwater : density of water at 20°C (0.998 g/mL).

Density Calculation:

$$Density = \frac{c-a}{Vp} \qquad (2)$$

Where = c : mass of pycnometer with vertosin (g).

Specific Gravity Calculation:

$$SG = \frac{P\ vertosine}{\rho air} \qquad (3)$$

Results were compared to industry standards and analyzed for trends across temperature and reaction time variations.

Compound Confirmation by GC-MS. Vertosin was diluted to 10% in 99% ethanol before GC-MS analysis. Instrument conditions for GC-MS are provided in Table 1, based on Supriyono et al. [11].

Table 1. GC-MS operating conditions

Parameter	Specification
GC-MS Model	Agilent Technologies 5975C
Column	HP Innowax capillary column, 30 m × 0.2 mm, 0.25 µm
Carrier Gas Pressure	7.05 Psi
Detector	Triple Quadrupole Mass Spectrometer (TQMS)
Injector Temperature	100°C
Injection Volume	0.2 µL
Injection Mode	Split
Split Ratio	80:1
Column Temperature Program	150°C (5 min), ramp 15°C/min to 250°C (hold 5 min)
Total Run Time	20 minutes

Data Analysis. A factorial randomized block design (RBD) with three replications was used. The experimental design involved two factors: synthesis temperature (90°C, 100°C, 110°C) and synthesis time (30 minutes and 60 minutes), resulting in 18 experimental units. Data were analyzed using two-way ANOVA via SPSS version 26 to assess the significance of main and interaction effects. A further Least Significant Difference (LSD) test was conducted to determine specific group differences. Hypotheses:

H0a: Temperature variation does not significantly affect vertosin concentration.

H1a: Temperature variation significantly affects vertosin concentration.

H0b: Reaction time variation does not significantly affect vertosin concentration.

H1b: Reaction time variation significantly affects vertosin concentration.

H0c: The interaction of temperature and time does not significantly affect vertosin concentration.

H1c: The interaction of temperature and time significantly affects vertosin concentration.

Result and Discussion

Synthesis of Schiff Base Vertosin

The formation of vertosin before and after the condensation reaction is illustrated in Figure 1. As shown in Figure 1(a), the compound vertosin had not yet formed. Two cloudy, light-yellow phases were observed, which were attributed to the starting materials. Upon synthesis (Figure 1b), the

mixture changed to a darker yellow-orange, indicating the formation of vertosin as confirmed by the color change and increased homogeneity. This transformation is associated with a condensation reaction between the aldehyde (triplal) and the amine (methyl anthranilate), involving the elimination of a water molecule and the formation of a new schiff base compound, vertosin.

The condensation was carried out at varying temperatures to accelerate the reaction, facilitate the removal of water, and prevent biphasic product separation, in accordance with the findings of Madiabu et al. [12]. The reaction mechanism is illustrated in Figure 2.

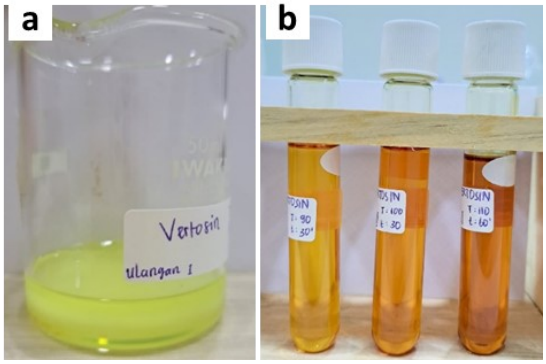

Figure 1. Formation of vertosin: (a) Before condensation reaction; (b) After condensation reaction

As illustrated, water is released as a by-product, with the oxygen atom from the aldehyde (triplal) combining with two hydrogen atoms from the amine (methyl anthranilate), yielding the schiff base methyl 2-[[(E)-(2,4-dimethyl-1-cyclohex-3-enylidene)methyl]amino]benzoate, known as vertosin.

Characteristics of Schiff Base Vertosin

Organoleptic Properties. Organoleptic testing is essential for evaluating the quality of synthesized schiff bases. Odor assessment was conducted by five trained panelists. Results are summarized in Table 2.

The odor characteristics arise from the volatile nature of the constituent compounds. Triplal (aldehyde) contributes to the green scent, while methyl anthranilate (amine) imparts floral and orange blossom notes. The variation in odor profiles corresponds to the volatility of each compound, affecting their position as top, middle, or base notes [11, 15]. Odor testing was conducted after the sample cooled to room temperature (~30 minutes post-synthesis).

At lower synthesis temperatures and shorter durations (e.g., 90°C, 30–60 min), fruity top notes were still detectable. However, higher temperatures and longer synthesis times (e.g., 110°C, 60 min) re-

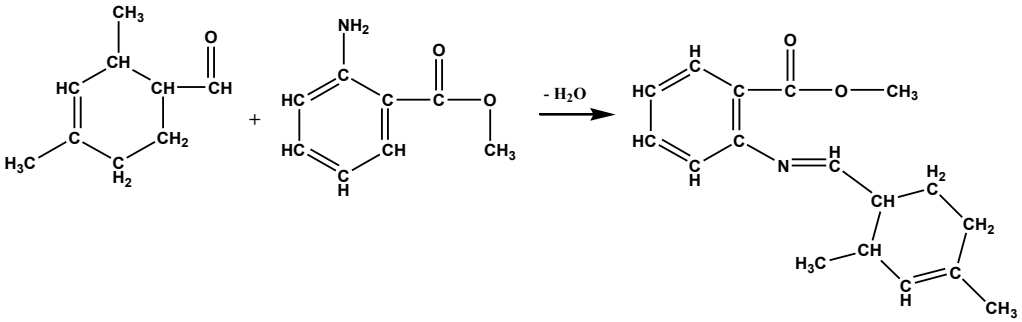

Figure 2. Reaction scheme of vertosin formation [12]

Table 2. Odor evaluation of vertosin

Temperature (°C)	Time (min)	Odor Description	Reference Odor	Conclusion
90	30	Green, Floral, Lily, Fruity	Green, Floral, Orange blossom, Lily, Fruity	Closest to standard: 110°C, 60 min
90	60	Green, Floral, Lily, Fruity		
100	30	Aldehydic, Fruity, Green, Floral		
100	60	Green, Lily, Orange blossom		
110	30	Green, Lily, Floral, Orange blossom, Herbal		
110	60	Green, Lily, Orange blossom, Fruity, Floral		

sulted in diminished top notes, with stronger presence of middle and base notes. This suggests that volatile components like triplal (bp 196°C) evaporate more readily under these conditions, while methyl anthranilate remains and contributes to the persistent scent of vertosin. The visual color characteristics under various synthesis conditions are summarized in Table 3.

All samples fell within the acceptable color range for standard vertosin (yellow to orange). Higher temperatures and longer reaction times resulted in deeper color intensity, indicating increased formation of the target compound. This observation is consistent with schiff base reactions, where darker coloration can occur due to Maillard-type browning reactions between primary amines and aldehydes, forming melanoidin compounds [4, 16].

Density and Specific Gravity. Average values for density and specific gravity measured by pycnometry are presented in Table 4. The density and specific gravity values of all samples were within the acceptable specification range. These parameters increased with rising temperature and extended reaction time, indicating a higher concentration of synthesized vertosin. This trend aligns with the principle that the greater the mass per unit volume, the higher the density and specific gravity [17].

Identification of Schiff Base Vertosin Compound. The identification of the synthesized schiff base compound, vertosin, was conducted using Gas Chromatography–Mass Spectrometry (GC-MS). The mass spectrum provided information on molecular fragmentation patterns, which were analyzed based on similarity percentage, base peak intensity, and comparison with a standard reference spectrum of vertosin from Symrise. The average values of retention time (RT), similarity index, and area percentage concentration data are summarized in Table 5.

Table 3. Color observation of vertosin

Temp (°C)	Time (min)	Observed Color	Reference Color	Conclusion
90	30	Yellow	Yellow–Orange	All within standard range
90	60	Yellow		
100	30	Dark Yellow–Yellow-Orange		
100	60	Light Orange–Orange		
110	30	Yellow-Orange–Light Orange		
110	60	Orange		

Table 4. Average density and specific gravity of vertosin

Temp (°C)	Time (min)	Density (g/cm³)	Standard [9]	Specific Gravity	Standard [9]
90	30	1.088	1.077–1.094	1.090	1.079–1.096
90	60	1.089		1.091	
100	30	1.090		1.092	
100	60	1.091		1.093	
110	30	1.091		1.093	
110	60	1.092		1.094	

Table 5. Average retention time, similarity index, and area percentage concentration of synthesized schiff base vertosin

Synthesis Temperature (°C)	Reaction Time (minutes)	Retention Time (RT)	Similarity (%)	Concentration of Vertosin (% Area)
Symrise Standard	–	39.376	99	85.30
90	30	39.356	97	75.53[a]
	60	39.351	98	77.57[b]
100	30	39.354	98	78.06[c]
	60	39.358	99	79.06[d]
110	30	39.359	98	80.26[e]
	60	39.356	99	84.21[f]

Note: Different superscript letters indicate statistically significant differences.

All synthesized vertosin samples demonstrated a similarity index exceeding 90%, confirming structural resemblance to the Wiley Library standard of vertosin. The average retention time ranged from 39.351 to 39.376 minutes, indicating consistency with the standard.

Statistical analysis revealed that variations in synthesis temperature, reaction time, and their interactions significantly affected the concentration of synthesized vertosin (Sig. 0.000 < 0.05). The calculated F-values exceeded the 5% critical F-value, further supporting the significance of these factors. Thus, the alternative hypotheses (H1a, H1b, H1c) were accepted, and post hoc analysis was conducted. The post hoc LSD test confirmed that all treatment combinations significantly differed from one another. The highest vertosin concentration was observed at 110°C for 60 minutes, indicating this condition as the optimal synthesis setting.

Chromatograms of synthesized vertosin (Figure 3) closely resembled that of the symrise standard (Figure 4), suggesting successful synthesis. Similar chromatographic patterns were observed for all treatment repetitions, with the best match achieved at 110°C and 60 minutes. At this optimal condition, vertosin with the molecular formula $C_{17}H_{21}NO_2$ (molecular weight = 271.36 g/mol) was identified at a retention time of 39.327 minutes, exhibiting 99% similarity. The late elution of the vertosin peak was attributed to its high molecular weight and strong interactions with the stationary phase.

As reported by Darmapatni et al. [18], derivatization is often necessary for large, polar molecules that are not readily volatile due to intermolecular hydrogen bonding. Blocking these polar groups increases volatility and improves GC-MS detection.

Further confirmation of compound identity was obtained through mass spectral fragmentation analysis. According to Sakiah [19], molecular structure elucidation via mass spectrometry relies on interpreting characteristic fragmentation patterns. The mass spectrum of synthesized vertosin (Figure 5) aligned with that of the standard (Figure 6), especially for the sample synthesized at 110°C for 60 minutes.

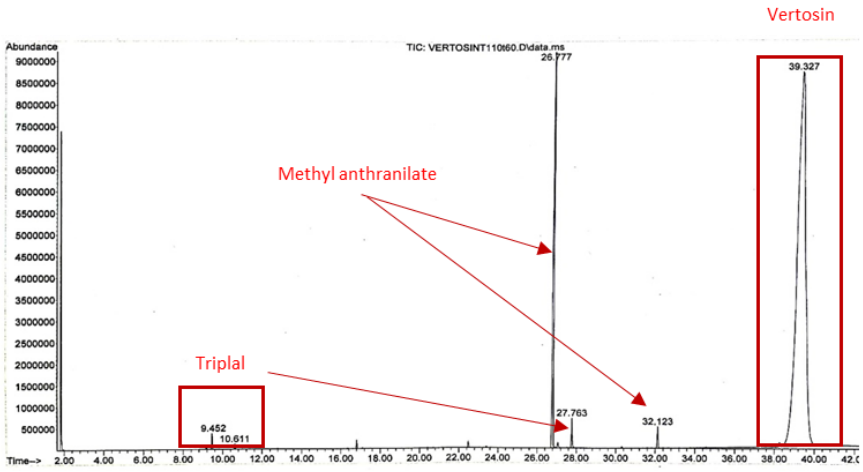

Figure 3. Chromatograms of synthesized vertosin at 110°C for 60 minutes

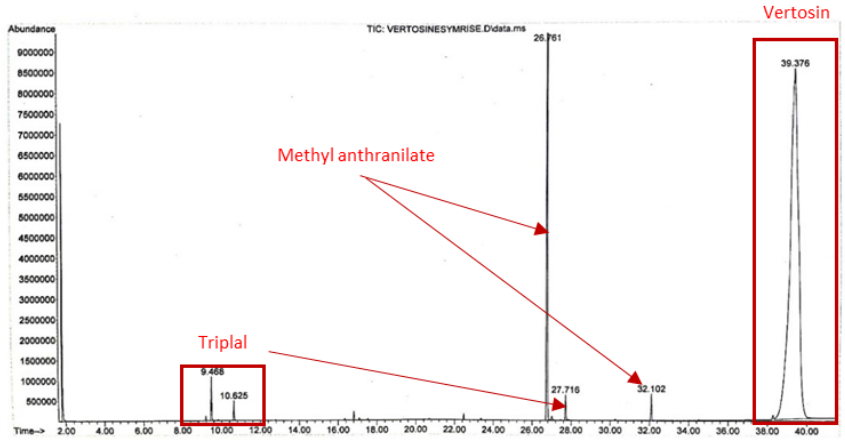

Figure 4. Chromatograms of the symrise standard

The mass spectrum of this sample exhibited a base peak at m/z = 271.1, corresponding to the molecular ion (M^+) of methyl 2-[[(E)-(2,4-dimethyl-1-cyclohex-3-enylidene)methyl]amino] benzoate (vertosin). Fragmentation peaks observed at m/z = 256.1, 244.1, 196.1, 151.1, and 138.1 were consistent with the proposed structure and its degradation pathways, as illustrated in Figure 5. These fragmentation patterns confirmed the identity of the synthesized compound as methyl 2-[[(E)-(2,4-dimethyl-1-cyclohex-3-enylidene)methyl]amino]benzoate (vertosin) with a molecular weight of 271 g/mol.

Conclusion

The optimal synthesis condition for schiff base vertosin was achieved at 110°C for 60 minutes, resulting in the highest concentration and a GC-MS fragmentation pattern consistent with methyl 2-[[(E)-(2,4-dimethyl-1-cyclohex-3-enylidene) methyl]amino]benzoate (vertosin) with a molecular weight of 271 g/mol. The synthesized compound exhibited a green, lily, orange blossom, fruity, and floral scent, orange coloration, and average density and specific gravity values of 1.092 and 1.094, all within the standard specification range for vertosin. Although this condition produced optimal results, it is recommended that vertosin be used at low concentrations in fragrance formulations to minimize potential color changes in the final product. Further studies are necessary to explore the effects and ideal concentrations of schiff base vertosin in various product applications.

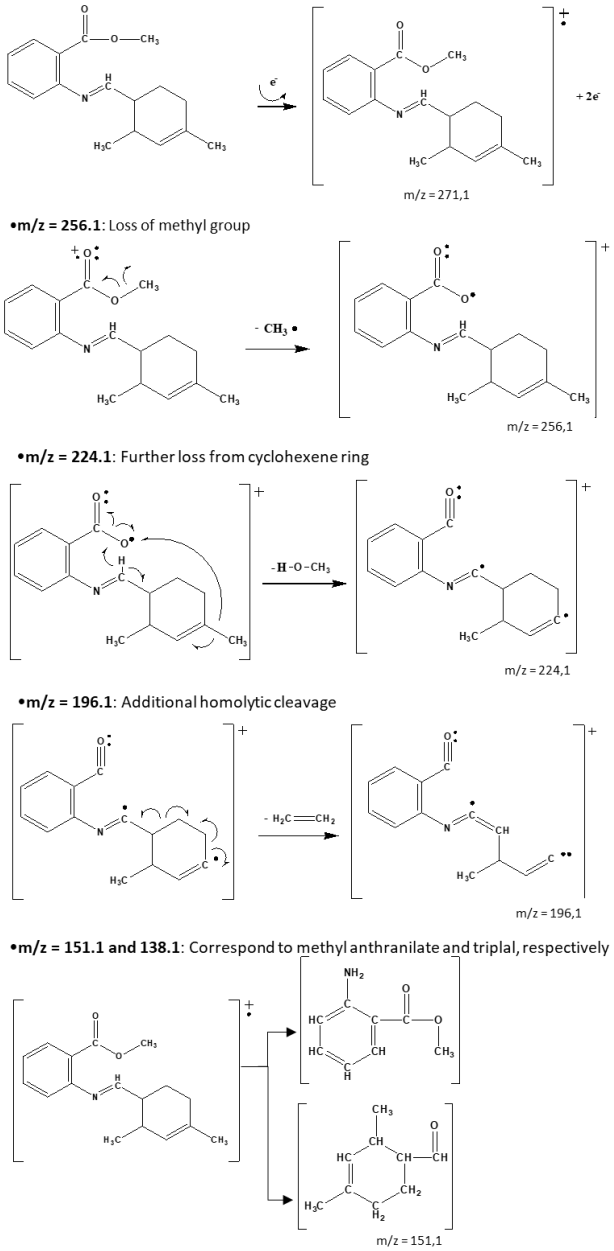

Figure 5. Fragmentation of proposed structure and its degradation pathways

References

[1] Iswara, F. P., Dwiarso, R., and Tatang, S. J., "Analisis Senyawa Berbahaya dalam Parfum dengan Kromatografi Gas-Spektrometri Massa Berdasarkan Material Safety Data Sheet (MSDS)," Indonesian Journal of Chemical Research, vol. 2, no. 1, pp. 18–27, 2014.

[2] Rowe, D. Chemistry and Technology of Flavours and Fragrances, USA: Blackwell Publishing Ltd., 2005.

[3] Wibowo, S., Candra, I., Dian, I., Rika, P. P., and Imalia, D. P., "Aurantiol Schiff base as A Raw Material in Fragrance Industry Synthesized by Simple Condensation Method and Its Characterization Using GC-MS," Orient. J. Chem., vol. 36, no. 3, pp. 577–580, 2020.

[4] Schmidt, C., "Schiff Bases – A Primer," Perfumer & Flavorist, vol. 27, p. 32, 2002.

[5] Yuliana, E., Candra, I., Dian, I., Singgih, W., Rika, P. P., and Imalia, D. P., "Synthesis and Spectral Characterization of Verdantiol Schiff base with GC-MS for Synthetic Ingredients in Fragrances Industry," Orient. J. Chem., vol. 35, no. 6, pp. 1737–1741, 2019.

[6] Utami, A., Candra, I., Dian, I., Rika, P. P., and Singgih, W., "Synthesis Study of Precursor Mixture of Verdantiol, Aurantiol and Lyrame Schiff Base and Its Characterization Using GC-MS," Orient. J. Chem., vol. 35, no. 3, pp. 1244–1247, 2019.

[7] Inamdar, A. "Vertosin," Perfumer & Flavorist, 2009.

[8] International Flavors & Fragrances Inc., Fragrance Ingredients: Triplal, Perfumer's Compendium, p. 183, 2018.

[9] Ventos, Spesification Sheet: Schiff Base, Triplal/Methyl Anthranilate, Ventos Spesification Sheet, no. 2, 2022.

[10] Kuswandi, M., Bayu, A. Y., and Broto, S., "Aktualisasi Kimia dalam Pengelolaan Sumber Daya Alam Berwawasan Lingkungan pada Era Globalisasi," in Seminar Nasional Kimia UNJANI-HKI, 2016, pp. 276–280.

[11] Supriyono, Candra, I., Suci, I., Endang, S. L., and Arinzani, H. "Synthesis and Characterization of Aurantiol Schiff Base, Relationship Between Synthesis Time and Some Physical Properties," Orient. J. Chem., vol. 34, no. 1, pp. 394–400, 2018.

[12] Adesina, A. D., "Synthesis of schiff bases by non-conventional methods," IntechOpen, 2022, DOI: 10.5772/intechopen.108688.

[13] Irawan, C., Swatika, J., Hanafi, H., Henny, R., Miskah, Y. F. and Rika, P. P., "Synthesis and spectral characterization Schiff base cyclantine with GC-MS," Int. J. Chem. Stud., vol. 5, no. 5, pp. 475–479, 2017.

[14] Arctender, S., Perfume And Flavor Chemicals, 2nd ed., Montclair, N.J., U.S.A: Publisher Montclair, 1969.

[15] Kristanti, R., "Pemanfaatan Minyak Atsiri Kapulaga (Amomun cardamom Willd), Kayu Manis (Cinnamon burmanni Ness), dan Jeruk Purut (Citrus hystrix DC) dalam pembuatan parfum," Skripsi, Universitas Kristen Satya Wacana, Salatiga, 2015.

[16] Arsa, M., "Proses Pencoklatan (Browning Process) pada Bahan Pangan," Jurusan Kimia, Fakultas MIPA, Universitas Udayana, Denpasar, 2016.

[17] Saputra, A. T., Muhammad, A. W., and Irsan, "Utilization of Used Oil for Biodisel Manufacturing Using Zeolite Activated Catalyst," Jurnal Chemurgy, vol. 01, no. 2, pp. 1–6, 2017.

[18] Darmapatni, K. A. G., Basori, A., and Suaniti, N. M., "Pengembangan Metode GC-MS untuk Penetapan Kadar Acetaminophen pada Spesimen Rambut Manusia," Boisains Pascasarjana, vol. 18, no. 3, pp. 64–71, 2016.

[19] Sakiah, S., "Modifikasi Proses Penyulingan dengan Variasi Tekanan Uap untuk Memperbaiki Karakteristik Aroma Minyak Pala," Tesis, Sekolah Pascasarjana IPB, Bogor, 2006.