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by Dewa Katja 21

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DAMMARANE-TYPE TRITERPENOIDS FROM THE STEMBARK OF *CHISOCHETON PENTANDRUS* (MELIACEAE)

D. G. Katja[1*], S. Salam[2], Nurlelasari[2], D. Harneti[2], R. Maharani[2,3], U. Supratman[2,3] and Y. Shiono[4]

[1]Department of Chemistry, Faculty of Mathematics and Natural Sciences, Sam Ratulangi University, Kampus Kleak, Manado, 95115, North Sulawesi, Indonesia
[2]Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Padjadjaran, Jatinangor 45363, Indonesia.
[3]Central Laboratory, Universitas Padjadjaran, Jatinangor 45363, Indonesia.
[4]Department of Food, Life, and Environmental Science, Faculty of Agriculture, Yamagata University, Tsuruoka, Yamagata 997-8555, Japan
** E-mail: dewakatja@yahoo.com*

ABSTRAK

Dua senyawa triterpenoid tipe damaran, cabraleadiol (**1**) dan cabraleahidrosilakton (**2**), telah diisolasi dari ekstrak *n*-heksana kulit batang *Chisocheton pentandrus* (Meliaceae). Struktur kimia senyawa **1** dan **2** diidentifikasi berdasarkan data-data spektroskopi terutama, NMR dan massa serta perbandingan data spektra dari laporan sebelumnya. Senyawa **1** dan **2** pertama kali dilaporkan pada tumbuhan *Chisocheton pentandrus.*

Kata kunci: Cabraleadiol, Cabraleahidrosilakton, *Chisocheton pentandrus*, Meliaceae.

ABSTRACT

Two dammarane-type triterpenoids, cabraleadiol (**1**) and cabraleahydroxylactone (**2**), have been isolated from *n*-hexane extract of the stembark of *Chisocheton pentandrus* (Meliaceae). The structure of compounds **1** and **2** were determined by spectroscopic data mainly NMR and mass as well as by comparing with previously reported spectral data. Compounds **1** and **2** were reported for the first time from *Chisocheton pentandrus*.

Keywords: Cabraleadiol, Cabraleahydroxylactone, *Chisocheton pentandrus*, Meliaceae.

INTRODUCTION

Triterpenoids are the most important group of terpenoids because they exhibit a great diversity of biological activities and they are the major constituents of tropical higher plants. Recently, research of new biologically active compounds from plants used in traditional medicine has led to the isolation of numerous triterpenoids with important biological activities (Fu *et al*., 2015; Nguyen *et al*., 2015). Higher plants are major source of triterpenoids with several biological activities and numerous reports have shown that family of Meliaceae, Rhamnaceae, Cucurbitaceae, Ganodermataceae and Apocynaceae produce a wide variety of tetracyclic triterpenoids, whereas, family of Ranunculaceae, Burseraceae, Capparidaceae, Celastraceae and Lamiaceae families are recognized to contain active pentacyclic triterpenoids (Harneti *et al*., 2012; Farabi *et al* 2017; Tian *et al*., 2005; Zhang *et al*., 2005).

Chisocheton genera is belong to the Meliaceae family, which consist of more than 50 plant species distributed mainly in tropical countries (Yang *et al*., 2009; Heyne, 1982). Previous phytochemical studies on *Chisocheton* species have yielded a number of interesting compounds, including limonoids (Supratman *et al*., 2019; Supriatno *et al*., 2018; Katja *et al*., 2016) and triterpenoids (Inada *et al*., 1993; Katja *et al*., 2017).

In our continous search for novel constituents from *Chisocheton pentandrus*, we isolated new limonoids, pentandricin A from the stembark of *C. pentandrus* (*Suprianto et al*., 2018). In the further investigation for novel compounds from non polar fraction of *C. pentandrus*, we found two dammarane-type

triterpenoids from the *n*-hexane extract. In this paper, we report the isolation and structural determination of two dammarane-type triterpenoids, cabraleadiol (**1**) and cabraleahydroxylactone (**2**).

MATERIAL AND METHODS

Plant material

The stem bark of *C. pentandrus* were collected in Bogor Botanical Garden, Bogor, West Java Province, Indonesia in June 2016. The plant was identified by Mr. Ismail, the staff of the Herbarium and a voucher specimen (No. Bo-104) was deposited at the Herbarium.

Extraction and isolation

The dried ground stembark (1.8 kg) of *C. pentandrus* was extracted with methanol (3 x 4 L) at room temperature for 6 days. After removal of the solvent under vacuum, the viscous concentrate of methanol extract (340 g) was first suspended in water and then partitioned with *n*-hexane, ethyl acetate and *n*-butanol, successively. Evaporation resulted in the crude extracts of *n*-hexane (10.9 g), ethyl acetate (25.2 g) and *n*-butanol (228.6 g), respectively. The *n*-hexane extract (10.9 g) was fractionated by column chromatography on silica gel using a gradient *n*-hexane-ethyl acetate-methanol to give eight fractions (I–VIII). Fraction II (5.4 g) was subjected to column chromatography over silica gel using a gradient mixture of *n*-hexane-dichloromethane-ethyl acetate (5% stepwise) as eluting solvents to afford thirteen subfractions (II_1-II_{13}). Subfraction II_9 (912.2 mg) was column chromatographed on silica gel, eluted with *n*-hexane:dichloromethane:ethyl acetate (5:4:1), to give six subfractions (II_{9-1}-II_{9-6}). subfraction II_{9-3} (62.2 mg) was further subjected to column chromatography over silica gel using a gradient mixture of *n*-hexane-dichloromethane-ethyl acetate (7:2:1) to give **1** (12.2 mg) and **2** (8.3 mg) .

General Experimental Procedure

Melting points were determined on an electrothermal melting point apparatus. The IR spectra were recorded on a Perkin Elmer spectrum-100 FT-IR in KBr. Mass spectra were obtained with a Synapt G2 mass spectrometer instrument. NMR data were recorded on a JEOL-ECZ-600 spectrometer at 600 MHz for ^{1}H and 150 MHz for ^{13}C. Chemical shifts are given on a δ (ppm) scale with tetramethylsilane (TMS) as an internal standard. Column chromatography was conducted on silica gel 60 (Merck). TLC plates were precoated with silica gel GF_{254} (Merck, 0.25 mm) and detection was achieved by spraying with 10% H_2SO_4 in ethanol, followed by heating.

RESULTS AND DISCUSSION

Cabraleadiol (1)

Physical properties: colourless needle crystal, m.p. 170-172°C. IR λ_{max} (KBr) cm^{-1}: 3457, 2948, 2861, 1461, 1380. HR-TOFI-MS *m/z*: 461.3879 $[M+H]^+$. ^{1}H-NMR (600 MHz, $CDCl_3$) and ^{13}C-NMR (125 MHz, CDCl3) see Table 1.

Cabraleahydroxylactone (2)

Physical properties: white amorphous powder, m.p. 240-242 °C, IR λ_{max} (KBr) cm^{-1}: 3550, 2900, 2811, 1760, 1457, 1386, 1249, 1196. HR-TOFI-MS *m/z*: 416 $[M+H]^+$; ^{1}H-NMR (600 MHz, $CDCl_3$) and ^{13}C-NMR (125 MHz, CDCl3) see Table 1.

Compound **1** was isolated as a colourless needle crystal. The molecular formula of **1** was determined to be $C_{30}H_{52}O_3$ from its molecular ion peak $[M+H]^+$ at *m/z* 460.3916 (calcd. for $C_{30}H_{52}O_3$, 460.3920) in the LC-TOFMS. The IR spectrum showed absorption peaks due to hydroxyl (3457 cm^{-1}), C-C aliphatic (2948 and 2861 cm^{-1}) and ether functional groups (1380 cm^{-1}).The ^{1}H NMR spectrum (Table 1) displayed eight tertiary methyl singlets [δ_H 0.87, 0.82, 0.92, 1.09, 1.17, 1.13, 0.95 and 0.84 (each 3H, s)], two oxymethine protons [δ_H 3.38 (1H, t, *J*=3.0 Hz, H-3 and 3.62 (1H, dd, *J*=4.8, 10.2 Hz, H-24)], and some aliphatic protons in the up field region. The ^{13}CNMR and DEPT-135 spectra (Table 1), exhibited the presence of eight methyl signals [δ_C 15.6 (Me-30), 22.2 (Me-29), 28.4 (Me-28), 24.1 (Me-27), 27.9 (Me-26), 27.3 (Me-21), 16.6 (Me-18) and 16.2 (Me-19)], ten methylenes [δ_C 33.7 (C-1), 25.4 (C-2), 18.3 (C-6), 34.8 (C-7), 21.7 (C-11), 27.1 (C-12), 31.5 (C-15), 25.9 (C-16), 35.3 (C-22), 26.4 (C-23)], six methines including two oxygenated methines [δ_C 76.4 (C-3), 49.6 (C-5), 50.7 (C-9), 42.8 (C-13), 49.8 (C-17), and

86.3 (C-24)] and eight quartenary carbon signals [δ_C 37.3 (C-4), 40.7 (C-8), 37.7 (C-10) and 50.2 (C-14). The NMR data suggested that **1** had a triterpenoid tetracyclic skeleton similar to the dammarane-type triterpenoid (Harneti et al., 2014). The structure of the tetracyclic system in **1** was determined by analysis of COSY and HMBC spectra (Figure 2). Key HMBC spectra were the ^2J correlations from the eight methyl groups (Me-18, Me-19, Me-21, Me-14, Me-26, Me-27, Me-29 and Me-30) to their attached carbons enabled the assignment of the eight singlet methyls. A secondary alcohol was assigned at C-3 by the HMBC correlations from H-1 (δ_H 1.42), H-2 (δ_H 1.55), and H-5 (δ_H 1.2[illegible]) to C-3 (δ_C 76.4), whereas a tertiary alcohol was located at C-25 by the HMBC correlation from Me-26 (δ_H 1.17), Me-27 (δ_H 1.09), H-24 (δ_H 3.62) to C-25 (δ_C 70.3).

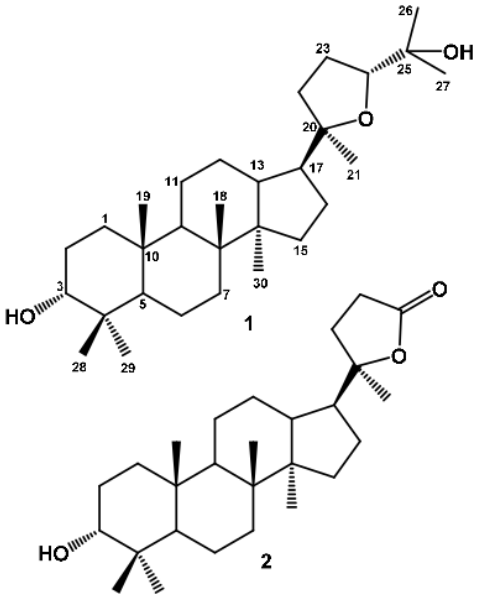

Figure 1. Chemical Structure of **1** and **2**

The relative stereochemistry of **1** was identified based on coupling constants in the ^{1}H-NMR and biogenetic point of view occurrence of dammarane-type triterpenoid in *Chisocheton* genus (Supratman *et al.*, 2019).

In comparison of **1** with literature data of a cabraleadiol (Phongmaykin *et al.*, 2008), showed good agreement, therefore compound **1** was identified as a cabraleadiol, which shown in this plant for the first time.

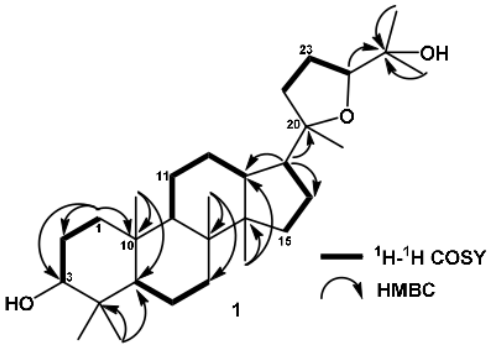

Figure 2. ^{1}H-^{1}H COSY and HMBC for **1**

Compound **2** was obtained as a white amorphous powder. The IR spectra showed absorption peaks at 3550 cm^{-1} (OH), 2960 and 2865 cm^{-1} (aliphatic), 1382 and 1238 cm^{-1} (*gem*-dimethyl groups), and 1040 cm^{-1} (C-O). The NMR spectra of **2** was very similar with **1**. The main differences was the absence of an isopropyl alcohol group at [(δ_H 1.17 (3H, s) and 1.09 (3H, s), δ_C 24.1 (Me-27), 27.9 (Me-26) and 70.3 (H-25)] and the presence of carbonyl lactone signal at δ_C 176.9, suggested that compound **2** was a lactone derivative of **1**. In the HMBC spectrum, methylene signal at δ_H 2.52 was correlated to carbonyl lactone at δ_C 176.9, indicated that a carbonyl lactone was located at C-24 to make a lactone ring as a side chain ring.

The relative stereochemistry of **2** was identified based on coupling constants in the ^{1}H-NMR and biogenetic point of view occurrence of dammarane-type triterpenoid in *Chisocheton* genus (Supratman et al., 2019). In comparison of **1** with literature data of a cabraleahydroxylactone (Phongmaykin et al., 2008), showed good agreement, therefore compound **1** was identified as a cabraleahydroxylactone, which shown in this plant for the first time.

Tabel 1. NMR data for compounds **1** and **2** ($CDCl_3$ 600 MHz for ^{1}H and 150 MHz for ^{13}C)

Position of C	Compound **1**		Compound 2	
	^{13}C NMR δc (mult.)	^{1}H NMR δ_H (Integral, mult., *J*=Hz)	^{13}C NMR δc (mult.)	^{1}H NMR δ_H (Integral, mult., *J*=Hz)
1	33.7 (t)	1.42 (1H, m)	35.2 (t)	1.17 (1H, m)
		1.34 (1H, m)		1.50 (1H, m)
2	25.4 (t)	1.55 (1H, m)	33.7 (t)	1.40 (1H, dd; 2,4; 9,6)
		1.28 (1H, m)		1.46 (1H, m)
3	76.4 (d)	3.38 (1H, t, 3)	76.3 (d)	3.37 (1H, s)
4	37.3 (s)	-	37.3 (s)	
5	49.6 (d)	1.24 (1H, m)	49.4 (d)	1.95 (1H, m)
6	18.3 (t)	1.39 (1H, m)	18.3 (t)	1.37 (1H, m)
		1.30 (1H, m)		1.56 (1H, m)
7	34.8 (t)	1.63 (1H, m)	26.9 (t)	1.71 (1H, m)
		1.76 (1H, m)		1.82 (1H, m)
8	40.7 (s)	-	40.6 (s)	-
9	50.7 (d)	1.44 (1H, m)	50.4 (d)	1.41 (1H, dd, 2.4, 13.2)
10	37.7 (s)	-	37.7 (s)	-
11	21.7 (t)	1.53 (1H, m)	25.4 (t)	1.20 (1H, m)
		1.46 (1H, m)		1.68 (1H, m)
12	27.1 (t)	1.75 (1H, m)	21.3 (t)	1.49 (1H, m)
		1.60 (1H, m)		1.24 (1H, m)
13	42.8 (d)	1.62 (1H, m)	43.2 (d)	1.53 (1H, m)
14	50.2 (s)	-	50.3 (s)	
15	31.5 (t)	1.04 (1H, m)	31.2 (t)	1.90 (1H, m)
		1.80 (1H, m)		1.10 (1H, m)
16	25.9 (t)	1.51 (1H, m)	25.1 (t)	1.52 (1H, m)
		1.72 (1H, m)		1.80 (1H, m)
17	49.8 (d)	1.83 (1H, m)	49.5 (d)	1.[illegible] (1H, m)
18	16.2 (q)	0.95 (3H, s)	15.6 (q)	0.92 (3H, s)
19	16.6 (q)	0.84 (3H, s)	16.1 (q)	0.82 (3H, s)
20	86.7 (s)	-	90.3 (s)	-
21	27.3 (q)	1.13 (3H, s)	25.4 (q)	1.33 (3H, s)
22	35.3 (t)	1.22 (1H, m)	31.3 (t)	1.47 (1H, m)
		1.40 (1H, m)		2.01 (1H, m)
23	26.4 (t)	1.85 (1H, m)	29.3 (t)	2.52 (1H, d, 10)
		1.90 (1H, m)		2.62 (1H, d, 9.9)
24	86.3 (d)	3.62 (1H, dd, 4.8, 10.2)	176.9 (s)	-
25	70.3 (s)	-	-	
26	27.9 (q)	1.17 (3H, s)	-	
27	24.1 (q)	1.09 (3H, s)	-	
28	28.4 (q)	0.92 (3H, s)	28.4 (q)	0.91 (3H, s)
29	22.2 (q)	0.82 (3H, s)	22.2 (q)	0.81 (3H, s)
30	15.6 (q)	0.87 (3H, s)	16.4 (q)	0.87 (3H, s)

CONCLUSIONS

Two dammarane-type triterpenoids have been isolated from the the stembark of *Chisocheton pentandrus* belong to Meliaceae family and identified by spectroscopic data as cabraleadiol (**1**) and cabraleahydroxylactone (**2**). The investigation of these dammarane-type triterpenoids were shown in this species for the first time and strenghten the occurance of this compounds in this genera.

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Katagori Publiasi Jurnal Ilmiah : ☐ Jurnal Ilmiah Internasional/Internasional bereputasi
(beri √ pada katagori yang tepat) ☑ Jurnal Ilmiah Nasional Terakreditasi
☐ Jurnal Ilmiah Nasional/Nasional Terindeks di DOAJ, CABI, COPERNICUS*

Hasil penilaian *Peer review*:

Komponen Yang Dinilai	Nilai Maksimal Jurnal Ilmiah			
	Internasional/Internasional bereputasi**	Nasional Terakreditasi	Nasional***	Nilai Akhir Yang Diperoleh
a. Kelengkapan unsur isi artikel (10%)		97		9,7
b. Ruang lingkup dan kedalaman pembahasan (30%)		97		29,1
c. Kecukupan dan kemutahiran data/informasi dan metodologi (30%)		96		28,8
d. Kelengkapan unsur dan kualitas terbitan/jurnal (30%)		96		28,8
Total = (100%)				
Nilai Pengusul =				96,4

Catatan Penilaian Artikel oleh Reviewer:
Artikel dimuat pada jurnal Nasional, isi jurnal lengkap dan ruang lingkup pembahasan memadai.

Manado, 24 Februari 2020
Reviewer 1

Nama : Prof. Dr. Ishak Isa, M.Si
NIP : 196105261987031005
Unit Kerja : FMIPA Universitas Negeri Gorontalo

* Dinilai oleh dua Reviewer secara terpisah
** Coret yang tidak perlu
*** Nasional/terindeks di DOAJ, CABi, Copernicus

LEMBAR
HASIL PENILAIAN SEJAWAT SEBIDANG ATAU *PEER REVIEW*
KARYA ILMIAH: Jurnal Ilmiah

Judul karya ilmiah (artikel)	: Dammarane-Type Triterpenoids From The Stembark of *Chisocheton Petandrus* (Metiaceae)	
Jumlah Penulis	: 7 orang	
Status Pengusul	: Penulis pertama	
Identitas Jurnal ilmiah	: a. Nama Jurnal	: Jurnal Kimia
	b. Nomor ISSN	: p-ISSN 1907-9850 : e-ISNN2599-2740
	c. Volume, nomor, bulan dan tahun	: Januari 2020
	d. Penerbit	: Jurnal Kimia
	e. DOI artikel (jika ada)	: https://doi.org/10.24843/JCHEM.2020.v14.i01.p15
	f. Alamat WEB Jurnal	: https://www.ojs.unud.ac.id
	g. Terindeks di	:

Katagori Publiasi Jurnal Ilmiah : ☐ Jurnal Ilmiah Internasional/Internasional bereputasi
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Hasil penilaian *Peer review*:

Komponen Yang Dinilai	Nilai Maksimal Jurnal Ilmiah			
	Internasional/Internasional bereputasi**	Nasional Terakreditasi	Nasional***	Nilai Akhir Yang Diperoleh
a. Kelengkapan unsur isi artikel (10%)		98		9,8
b. Ruang lingkup dan kedalaman pembahasan (30%)		98		29,4
c. Kecukupan dan kemutahiran data/informasi dan metodologi (30%)		98		29,4
d. Kelengkapan unsur dan kualitas terbitan/jurnal (30%)		97		29,1
Total = (100%)				
Nilai Pengusul =				97,7

Catatan Penilaian Artikel oleh Reviewer: Artikel ini ditulis dengan baik dan sistematika serta penelusuran yg mendalam dan berreputasi ~~inter~~ nasional serta ?

Manado, 24 Februari 2020
Reviewer 1

Nama : Prof. Dr. Edi Suryanto, M.Si
NIP : 196407241996031001
Unit Kerja : Kimia FMIPA Universitas Sam Ratulangi

* Dinilai oleh dua Reviewer secara terpisah
** Coret yang tidak perlu
*** Nasional/terindeks di DOAJ, CABi, Copernicus

LEMBAR
HASIL PENILAIAN SEJAWAT SEBIDANG ATAU *PEER REVIEW*
KARYA ILMIAH: Jurnal Ilmiah

Judul karya ilmiah (artikel)	: Dammarane-Type Triterpenoids From The Stembark Of *Chisocheton Pentandrus* (Metiaceae)	
Jumlah Penulis	: 7 orang	
Status Pengusul	: Penulis pertama	
Identitas Jurnal ilmiah	: a. Nama Jurnal	: Jurnal Kimia
	b. Nomor ISSN	: p-ISSN 1907-9850 : e-ISSN 2599-2740
	c. Volume, nomor, bulan dan tahun	: Januari 2020
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	f. Alamat WEB Jurnal	: https://www.ojs.unud.ac.id
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Katagori Publiasi Jurnal Ilmiah : ☐ Jurnal Ilmiah Internasional/Internasional bereputasi
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☐ Jurnal Ilmiah Nasional/Nasional Terindeks di DOAJ, CABI, COPERNICUS*

Hasil penilaian *Peer review*:

Komponen Yang Dinilai	Nilai Maksimal Jurnal Ilmiah			
	Internasional/Internasional bereputasi**	Nasional Terakreditasi	Nasional***	Nilai Akhir Yang Diperoleh
a. Kelengkapan unsur isi artikel (10%)		96		9,6
b. Ruang lingkup dan kedalaman pembahasan (30%)		96		28,8
c. Kecukupan dan kemutahiran data/informasi dan metodologi (30%)		96		28,8
d. Kelengkapan unsur dan kualitas terbitan/jurnal (30%)		96		28,8
Total = (100%)				
Nilai Pengusul =				96

Catatan Penilaian Artikel oleh Reviewer:
Artikel ini dengan ruang lingkup yang luas serta pembahasan cukup mendalam sesuai dg bidang penulis.

Manado, 24 Februari 2020
Reviewer 1

Nama : Prof. Dr. Feti Fatimah, MSi
NIP : 196807291994 032002
Unit Kerja : Kimia FMIPA Universitas Sam Ratulangi

* Dinilai oleh dua Reviewer secara terpisah
** Coret yang tidak perlu
*** Nasional/terindeks di DOAJ, CABi, Copernicus

HASIL PENILAIAN SEJAWAT SEBIDANG ATAU *PEER REVIEW*
KARYA ILMIAH : Jurnal Ilmiah

Judul Artikel Ilmiah : Dammarane-Type Triterpenoids From The Stembark of *Chisocheton Pentandrus* (Meliaceae)

Jumlah Penulis : 7 orang

Status Pengusul : Penulis pertama

Nama Pengusul : Dr. Dewa Gede Katja, M Si

Identitas Jurnal ilmiah :
- a. Nama Jurnal : Jurnal Kimia
- b. Nomor ISSN : p-ISSN 1907-9850
  : e-ISSN2599-2740
- c. Volume, nomor, bulan dan tahun : Januari 2020
- d. Penerbit : Jurnal Kimia
- e. Jumlah halaman : 5

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- ☐ Jurnal Ilmiah Internasional bereputasi
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- ☐ Jurnal Ilmiah Nasional Tidak Terakreditasi
- ☐ Jurnal Ilmiah Nasional Terindeks di DOAJ dll.

Hasil penilaian Validasi

No.	ASPEK	KOMENTAR PENILAIAN
1.	Indikasi Plagiasi	tidak terindikasi plagiat
2.	Linearitas	sesuai dengan bidang kajian yg.

Hasil penilaian *Peer Review* :

Komponen yang dinilai	Nilai Maksimal Jurnal ~~25~~ - 20					
	Internasional Bereputasi	Internasional	Nasional Terakreditasi	Nasional Tidak Terakredirasi	Nasional Terindeks DOAJ dll	Nilai Akhir yang diperoleh
Kelengkapan & kesesuaian unsur isi jurnal (10%)			2.0			1.2
Ruang lingkup dan kedalaman pembahasan (30%)			6.0			3.6
Kecukupan dan kemutahiran data/informasi dan metodologi (30%)			6.0			3.6
Kelengkapan unsur dan kualitas penerbit (30%)			6.0			3.6
Total = (100%)			20			12.0
Kontribusi Pengusul (Penulis Pertama / Penulis Anggota)						

Komentar *Peer Review* :

Kelengkapan & kesesuaian unsur isi jurnal	Kelengkapan dan unsur isi jurnal lengkap
Ruang lingkup dan kedalaman pembahasan	Ruang lingkup pembahasan memenuhi.
Kecukupan dan kemutahiran data/informasi dan metodologi	Data dan metodologi mutakhir
Kelengkapan unsur dan kualitas penerbit	Kelengkapan dan kualitas penerbit baik

Jatinangor, 24 Februari 2020
Reviewer 1,

Prof. Dr. Ace Tatang Hidayat, M.Si
NIP 196712291997021001
Unit Kerja : Departemen Kimia FMIPA Unpad