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A review of chemistry and biological activities of the Indonesian Octocorallia

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ABSTRACT

Marine invertebrates are known to produce secondary metabolites that may have potential as new drug candidates. Numerous chemical studies have shown that sponges are a rich source of marine bioactive compounds. However, recent studies demonstrate that octocorals also produce secondary metabolites with promising pharmacological activities. In this paper, we report on the chemical and biological activities of the Indonesian octocorals, particularly soft corals, gorgonians and sea pens. Octocorals from the Indonesian coasts have been demonstrated to contain a wide variety of compounds including steroids, sesquiterpenes and diterpenes. Cembranoid diterpenes have been largely founds in gorgonian and soft corals and they are believed to function as chemical defenses. Anti-inflammatory, antimicrobial, cytotoxic to antitumor have been disclosed for members of the cembranoid class.

INTRODUCTION

Marine organisms are an important source of new bioactive molecules; thus the scientific community worldwide is focusing its efforts on the isolation and characterization of biologically active natural products (Almeida *et al.*, 2014). Since the early days of marine natural products research in the 1960s, sponges have yielded the largest number of new metabolites reported per year compared to any other plant or animal phylum known from the marine environment (Edaba and Proksch, 2012). However, with the development of new methods in analytical technology, spectroscopy, and high-throughput screening, the bioprospecting potential of cnidarians as they have also produced secondary metabolites with promising pharmacological activities (Molinski *et al.*, 2009). A total of 3244 marine natural products was described from this phylum alone between 1990 and 2011, which shows the importance of cnidarians for marine natural product research (Rocha *et al.*, 2015).

The phylum Cnidarian is a large, diverse and ecologically important group of marine invertebrates that are divided into five classes: Anthozoa, Hydrozoa, Cubozoa, Straurozoa and Chyphozoa and it contains over 11000 species, 7500 of them belonging to the class Anthozoa (Rocha *et al.*, 2011; Daly *et al.*, 2007). From the 3244 new compounds yielded by marine Cnidarians since 1990, 99% were discovered within Anthozoa. The remaining 1% is associated with species from Hydrozoa (Rocha *et al.*, 2015). Anthozoa is divided into the subclasses Hexacorallia and Octocorallia, each one further separated into multiple orders. Octocorals comprise soft corals, sea pens and gorgonians, with eight tentacles and eight internal mesenteries that exhibit less variation in polyp morphology than hexacorals (Almeida *et al.*, 2014; Rocha *et al.*, 2011; McFadden *et al.*, 2010). Octocorallia comprise approximately 3200 species of soft corals (Alcyonacea) found in all marine environments and 94% of new compounds from cnidarians were discovered from soft corals or Alcyonacea (Rocha *et al.*, 2015). With all of these compounds, soft corals, or alcyonacea, proved to be the most promising source of compounds for pharmaceutical use.Terpenoid chemistry predominates across the Octocorallia class.

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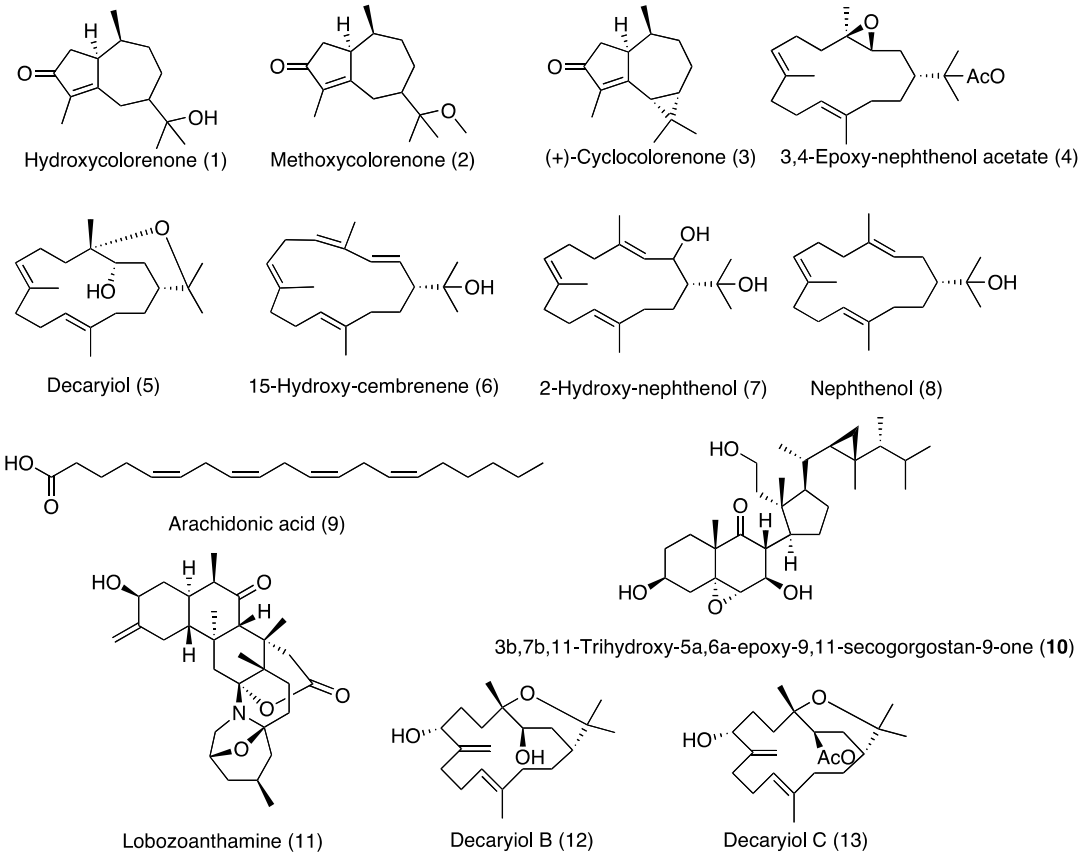
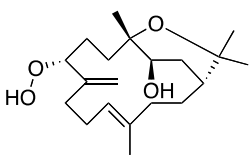
Sesquiterpenes and diterpenes are the most common terpenes isolated from Octocorallia. Numerous ecological studies have shown that to survive in the complicated marine environment, most octocorals produce diterpenes compounds, and these compounds are generally believed to function as chemical defenses. Several bioactivities including anti-inflammatory (Putra *et al.,* 2012; Kapojos *et al.,* 2010), antimicrobial (Wang *et al.,* 2009), cytotoxic (Rodríguez*et al.,* 1998; Fu *et al.,* 1999), antitumor (Chen *et al.,* 2011) have been isolated from members of the terpenoid class of Octocorallia. Since 1997-2014, more than 22 publications have reported on the bioactive compounds from Indonesian octocorallia such as sea pens, soft corals and gorgonians. Soft corals are the dominant reef-dwelling octocorals in the Indo-Pacific regions including Indonesia. Almost 99% of new marine natural products from Indonesian octcorals were collected from soft corals. In this review we report the marine compounds isolated from octocorals from Indonesia, specifically focusing on their structures and biological activities.

Soft corals

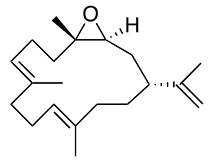
Soft corals are rich sources of secondary metabolites, particularly diterpenoids, sesquiterpenoid and steroids. The bioactive compounds in eight species of Indonesian soft corals such as *Sinularia* sp, *Lobophytum* sp, *Cladiella* sp, *Xenia* sp, *Sarcophyton* sp, *Nephthea* sp and *Minabea* sp, have been reported. Handayani and colleagues isolated two new oxygenated sesquiterpenes, hydroxycolorenone (**1**) and methoxycolorenone(**2**), and the known sesquiterpene, (+)-cyclocolorenone (**3**) from *Nephthea chabrolii* collected by snorkelling off the shores of Sinyary Island of West Sumatera (Handayani *et al.,* 1997). Compound **1** exhibited insecticidal activity towards neonate larvae of the polyphagous pest insect *Spodoptera littoralis*, with an EC_{50} of 8.8 ppm and LC_{50} of 453 ppm, when incorporated in an artificial diet and offered to larvae in a chronic feeding bioassay.

Januar and colleagues also isolated a new 3, 4-epoxy-nephthenol acetate (**4**) along with five known compounds: decaryiol (**5**), 15-hydroxy-cembrenene (**6**),2-hydroxy-nephthenol (**7**), nephthenol (**8**) and arachidonic acid (**9**) from two specimens of the soft coral *Nephthea sp.*, collected from the Seribu Islands (Januar *et al.,* 2010). Compounds **4** – **9** were screened for their whole cell anticancer activity against three human tumor cell lines; SF-268 (CNS), MCF-7 (breast), H460 (lung). Unfortunately, compounds **4** – **9** demonstrated weak (GI50> 100 μM) non-selective activity towards the three cell lines. Morris *et al.* described a new -epoxy-9,11-secogorgostan-9-oneα,6α,11-Trihydroxy-5β,7β3 (**10**), with a gorgosterol side chain and an unusual oxygenation pattern on the A and B rings from *Lobophytum* sp., collected at Mayu Island, Molucca Sea (Morris *et al.,* 1998). Fattorusso *et al.* identified a new member of the family of zoanthamine-type alkaloids named lobozoanthamine (**11**) and three novel cembrane diterpenoids, decaryiols B–D (**12**–**14)**, along with three known cembranoids named 2-hydroxy-nephthenol (**7**), epoxycembrane (**15**), decaryiol (**16**) from the Indonesian soft coral *Lobopphytum* sp., collected along the island of Siladen, in the Bunaken Marine Park near Manado (North Sulawesi, Indonesia) (Fattorusso *et al.,* 2009).

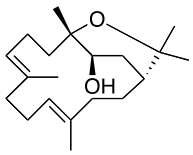




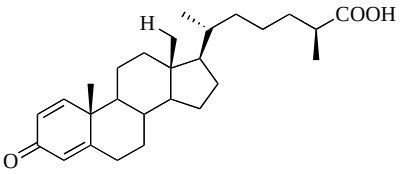
Decaryiol D (14)



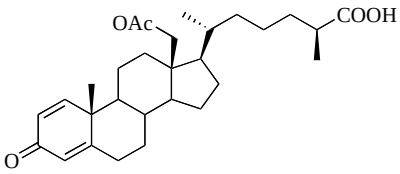
Epoxycembrane (15)



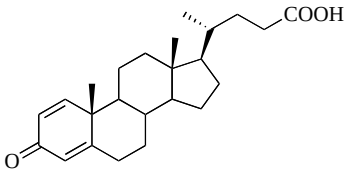
Decaryiol (16)



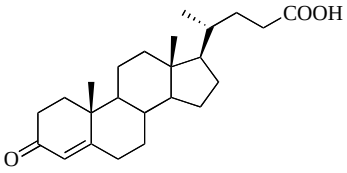
(25S)-3-oxocholesta-1,4-dien-26-oic acid (17)



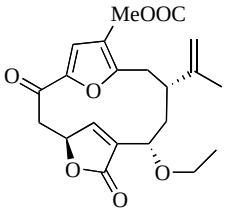
(25S)-18-Acetoxy-3-oxocholesta-1,4-dien-26-oic acid (18)



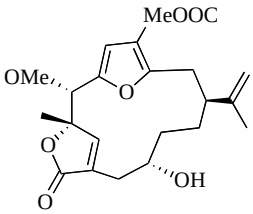
3-Oxochol-1,4-dien-24-oic acid (19)



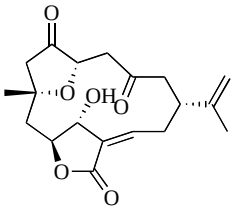
3-Oxochol-4-en-24-oic acid (20)



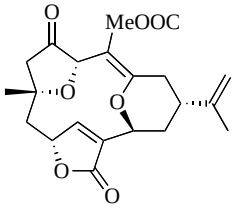
Sarcofuranocembrenolide A (21)



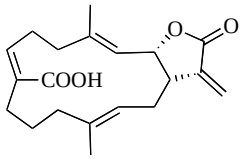
Sarcofuranocembrenolide B (22)



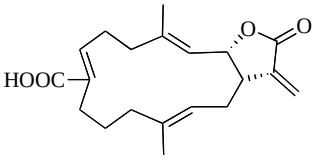
5-epi-sinuleptolide (23)



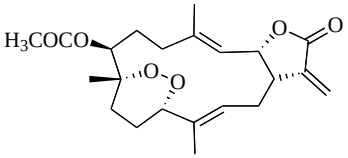
(24)



Lobohedleolide (25)



(7Z)-Lobohedleolide (26)



Denticulatolide (27)

(25S)-3-Oxocholesta-1,4-dien-26-oic acid (**17**) and a new (25S)-18-acetoxy-3-oxocholesta-1,4-dien-26-oic acid (**18**), together with two known cholic-acid-type compounds, 3-oxochol-1,4-dien-24-oic acid (**19**) and 3-oxochol-4-en-24-oic acid (**20**), were isolated by Wang *et al*. from a soft coral *Minabea sp*. (cf. aldersladei) collected in North Sulawesi, Indonesia (Wang *et al.,* 2009).

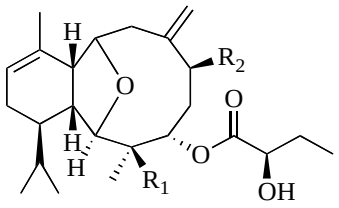
This is the first report of compound **17** as a natural product. Antimicrobial activity against Gram-positive (*S. aureus*) and negative bacteria (*E. coli*), yeast (*S. cerevisiae*), and a Filamentous fungus (*M. hiemalis*) and cytotoxicity against V79 and L1210 cells were examined, and compounds **17**–**20** showed no apparent activityat 100μg/disk (antimicrobial), 10 μM(V79 cells), and 50 μg/mL(L1210).

Kapojos *et al.* reported two unusual cembranoids, sarcofuranocembrenolides A (**21**) and B (**22**), together with five known cembranoids named 5-epi-sinuleptolide (**23**),**24**, lobohedleolide(**25**), (7Z)-lobohedleolide (**26**) and denticulatolide (**27**) (Kapojos *et al.,* 2010). Compounds **25**–**27** inhibited the

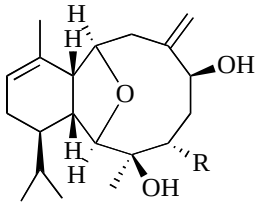
colony formation of V79 cells at ED_{50} values of 4.6, 3.7, and 3.6 mM, respectively, and reduced TNF α production from lipopolysaccharide (LPS)-stimulated mousemacrophage RAW 264.7 cells at 3.0–10.0 mM. Compounds **21** – **27** were isolated from a soft coral *Sarcophyton* sp., collected by scuba diving in Manado, North Sulawesi. Chen *et al.* described nine new diterpenoids named cladielloides A – B (**28** –**29**), cladieunicellins A—F (**30**—**35**) and (–)-solenopodin C (**36**), which were isolated from an Indonesian octocoral identified as *Cladiella* sp (Chen *et al.*, 2011).

Compound **29** exhibited moderate cytotoxicity toward CCRF-CEM tumor cells and this compound displayed significant inhibitory effects on superoxide anion generation and elastase release by human neutrophils. The cytotoxicity of metabolites **30**-**34** toward a limited panel of tumor cell lines, including DLD-1, HL-60, CCRF-CEM (human T-cell acute lymphoblastic leukemia), and P388D1 cells was evaluated. The results showed that cladieunicellins B (**31**) and E (**34**) exhibited significant cytotoxicity against DLD-1 and HL-60 cells. Compound **32** displayed a significant inhibitory effect against superoxide anion generation by human neutrophils. Compound **36** displayed significant inhibitory effects on the generation of superoxide anion and the release of elastase by human neutrophils at a concentration of 10 mg/mL. Similarly, from the same university, Tai *et al.* found two novel eunicellin-based diterpenoids, cladielloides C (**37**) and D (**38**) (Tai *et al.*, 2011).

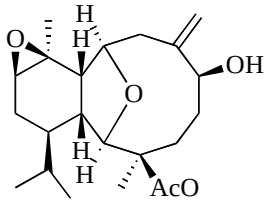
Compound **37** exhibited significant cytotoxicity toward CCRF-CEM tumor cells and metabolites **37** and **38** displayed moderate inhibitory effects on superoxide anion generation by human neutrophils. Anta *et al.* found two new xeniolides, xeniolide-F (**39**) and 9-hydroxyxeniolide-F (**40**), along with isoxeniolide-A (**41**) and 7,8-oxido-isoxeniolide-A (**42**), isolated from *Xenia* sp, collected in the Togian Islands near Sulawesi Island (Anta *et al.*, 2002a). Compounds **39** – **42** showed an IC_{50}>1 g/mL against mouse (P-388) and human (A-549, HT-29, MEL-28) tumorcell lines. Fattorusso and colleagues also isolated four novel xenicane diterpenoids, xenimanadins A-D (**43** – **46**), characterized by the unusual 2, 6-dimethoxytetrahydropyran functionality, have been isolated from the Indonesian soft coral *Xenia* sp., together with three known xeniolides, named blumiolide C (**47**), xeniolide F (**39**), and deoxyoxidoisoxeniolide A (**48**) (Fattorusso *et al.*, 2008). A new secosterol (**49**), along with pachyclavulariaenone B (**50**), was isolated from *Pachyclavularia violacea* by Anta *et al.* Compounds **49** and **50** showed an IC_{50}> 1 µg/mL against mouse (P-388) and human (A-549, HT-29, MEL-28) tumor cell lines (Anta *et al.*, 2002b).



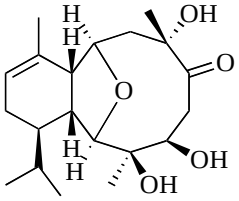
Cladielloide A (28) : R1= OAc, R2 = OH
Cladielloide B (29) : R1 = OH, R2 = OAc



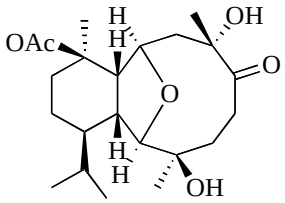
Cladieunicellin A (30) : R = H
Cladieunicellin B (31) : R = OH



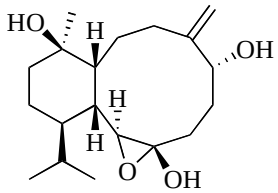
Cladieunicellin C (32)



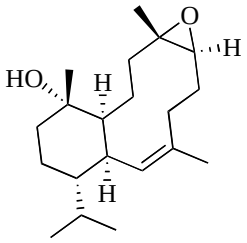
Cladieunicellin D (33)



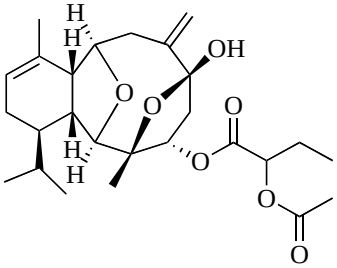
Cladieunicellin E (34)



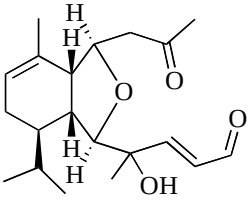
Cladieunicellin F (35)



(-)-Solenopodin C (36)



Cladielloide C (37)



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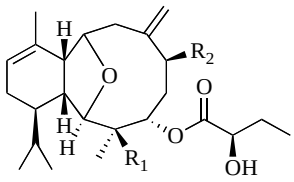
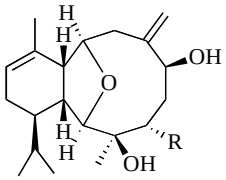
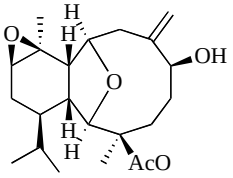


Cladielloide A (28) : R1= OAc, R2 = OH
Cladielloide B (29) : R1 = OH, R2 = OAc



Cladieunicellin A (30) : R = H
Cladieunicellin B (31) : R = OH



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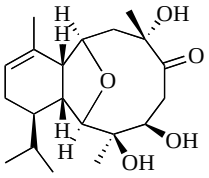

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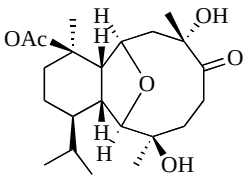
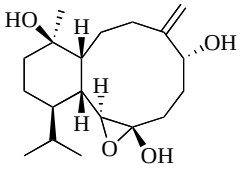


Cladieunicellin E (34)



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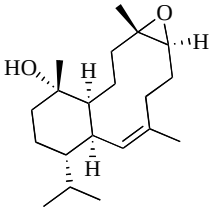
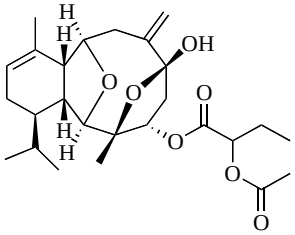


(-)-Solenopodin C (36)



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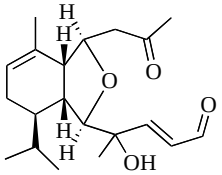

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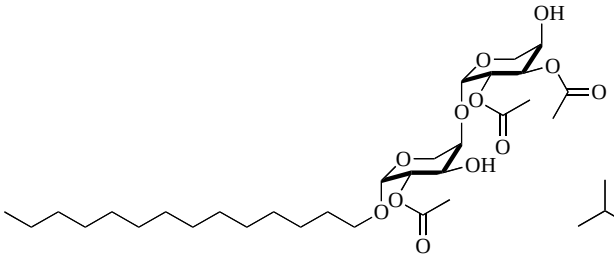
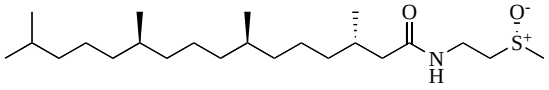
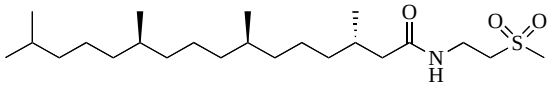


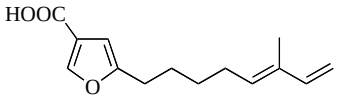
Sinularioside (51)



Sinulasulfoxide (52)

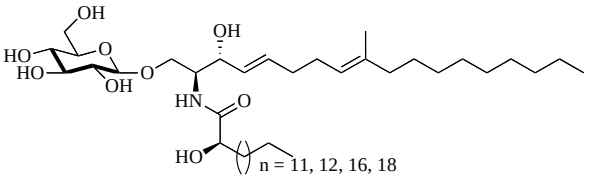


Sinulasulfone (53)

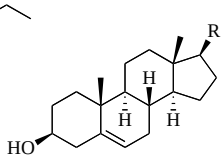


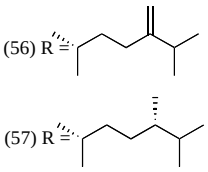
Furanosesquiterpenoid (54)

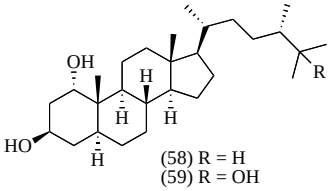
Putra and colleagues isolated three new compounds named sinularioside (**51**), sinulasulfoxide (**52),** sinulasulfone(**53**), along with two known compounds named furanosesquiterpenoid (**54**) and cerebroside (**55**) from Indonesian soft coral *Sinularia* sp collected in Siladen Island, Bunaken Marine National Park (North Sulawesi) (Putra *et al.,* 2012a; Putra *et al.,* 2012b). Compound **51** proved to consistently inhibit the expression of iNOS protein, a potentially anti-inflammatory activity. Putra *et al.* also isolated three new sterols (**56** – **58**) and five known sterols (**59** – **63**) (Putra *et al.*, 2012c) characterized by either 24-methylcholestane or gorgostane skeletons. The isolated steroids have been evaluated for their interaction with the farnesoid X-activated receptor (FXR) and some of them, including the new compound **58** and gorgosterol (**62**), showed a consistent antagonistic activity, potentially useful for the treatment of cholestasis. The FXR antagonistic activity of gorgosterol was also supported by gene expression experiments. Compound **62** represents the first evaluation of soft coral steroids for interaction with nuclear receptors and qualify gorgosterol as a new chemotype of FXR antagonist. Similarly, from the same university, Fattorusso *et al.* isolated two known C-4 norcembranoids,, named leptocladolide B **(64)** and scabrolide D **(65)**, and three new ones, named chloroscabrolides A **(66)** and B **(67)** and prescabrolide **(68)**. All the norcembranoids were evaluated for anti-inflammatory activity, scabrolide D (**65**) at a concentration of 10 μM, a 15% inhibition of NO_2^- production was observed (Fattorusso *et al.,* 2011).



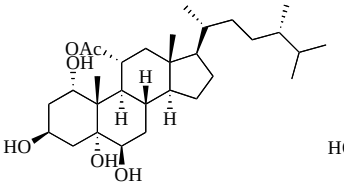
Cerebroside (55)



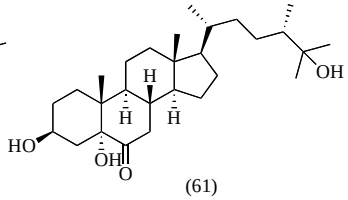




(58) R = H
(59) R = OH



(60)



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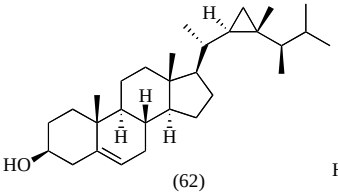
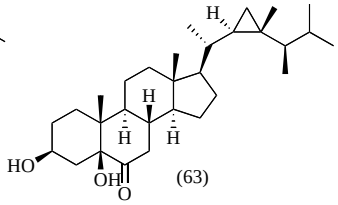
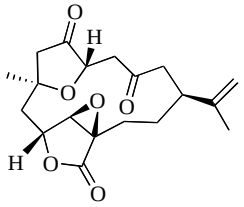


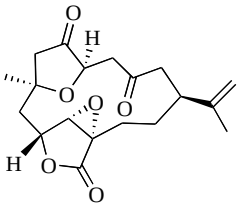
(62)



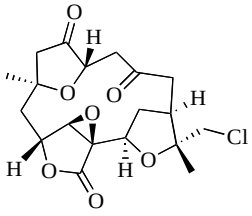
(63)



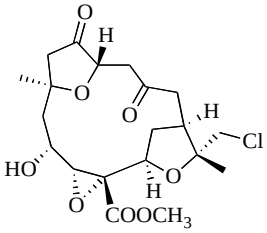
scabrolide D (64)



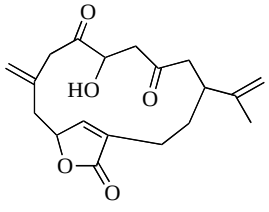
leptocladolide B (65)



chloroscabrolide A (66)



chloroscabrolide B (67)

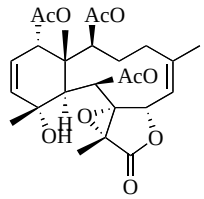


prescabrolide (68)

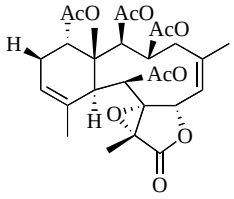
3. Gorgonians

Rodriguez *et al*. reported the Indonesian gorgonian *Briareum* sp. has afforded two new briarane stecholide diterpenes, named 2,9-diacetyl-2-debutyrylstecholide H (**69**) and 13-dehydroxystecholide J (**70**), the semisynthetic 2β -acetoxy-2-(debutyryloxy) stecholide E acetate (**71**), which was isolated as a natural product for the first time, along with the known compounds stecholides I - M (**72-76**), stecholide A acetate (**77**), and stecholide C acetate (**78**) (Rodríguez *et al.*, 1998).

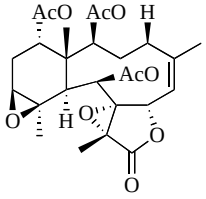
Compound **71** was reported to exhibit significant activity against the growth of P-388 cells (EC_{50} 1.59 μg/mL) and this is the first time that mild cytotoxic activity is found for the known compound 75. The gorgonian *Briareum* sp., was collected along the coast of the Togian Islands. Four new diterpenoids with the briarane skeleton, -epoxy-4- deacetoxyjunceellolide D (**82**)β, 20β-epoxy-4- deacetyljunceellolide D (**81**), and (-)-11β, 20β -epoxyjunceellolide D (**80**), (-)-11α,20α(-)-4-deacetyljunceellolide D (**79**), (+)- 11, (+)-junceellolide A (**83**) [the antipodal derivative of the known (-)-junceellolide A], along with three known briaranes, (-)-junceellolide D (**84**), (-)-junceellin (**85**), and (-)-praelolide (**86**), were isolated from the Indonesian gorgonian *Junceella fragilis* by García *et al*. (1999). Specimens of *Junceella fragilis* were collected at Halmahera Island. Gonzalez *et al*. isolated thirteen new Polyoxygenated steroids (**87-99**) along with the known compounds (**100-103**) from the Indonesian gorgonian *Isis hippuris* (González *et al*., 2011).



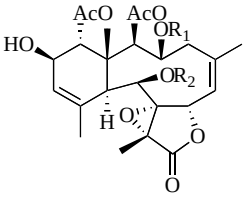
2,9-Diacetyl-2-debutyrylstecholide H (69)



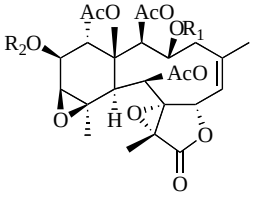
13-Dehydroxystecholide J (70)



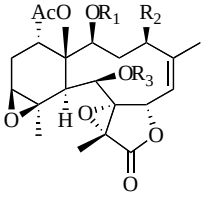
2b -Acetoxy-2-(debutyryloxy)stecholide E acetate (71)



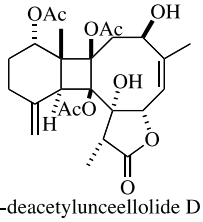
Stecholide I (72), $R_1 = COC_3H_7$, R_2 = Ac
Stecholide J (73), $R_1 = R_2$ = Ac



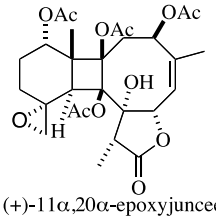
Stecholide K (74), $R_1 = R_2$ = Ac
Stecholide L (75), R_1 = Ac, R_2 = H
Stecholide M (76), $R_1 = C_OC_3H7$, R_2 = H



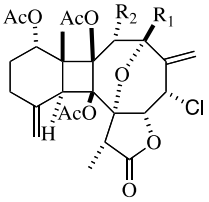
Stecholide A acetate (77), $R_1 = COC_3H_7$, R_2 = OAc, R_3 = Ac
Stecholide C acetate (78), R_1 = R3 = Ac, R_2 = OAc



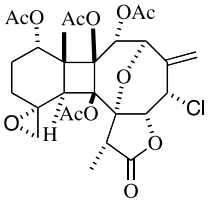
(-)-4-deacetylunceellolide D (79)



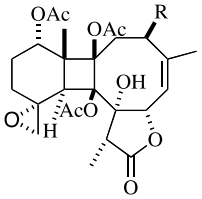
(+)-11α,20α-epoxyjunceellolide D (80)



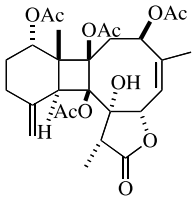
R_1=OH, R_2=H, (+)-junceellolide A (83)
R_1=H, R_2=OAc, (-)-junceellin A (85)



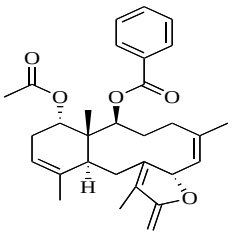
(-)-4-praeolide (86)



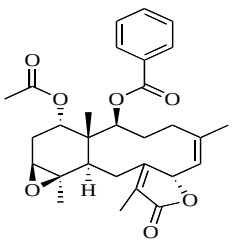
R=OH, (-)-11α,20α-epoxy-4-deacetyljunceellolide D (81)
R=H, (-)-11α,20α-epoxy-4-deacetoxyjunceellolide D (82)



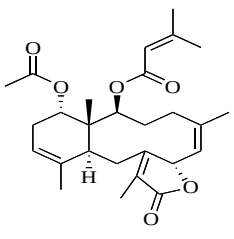
(-)-Junceellolide D (84)



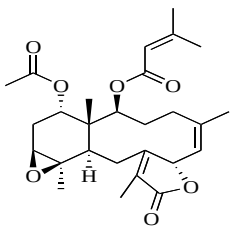
Malayenolides A (104)



Malayenolides B (105)



Malayenolides C (106)



Malayenolides D (107)

Sea pen

Four new briarane diterpenes, named Malayenolides A-D (**104-107**), were isolated from the sea pen *Veretillum malayense* collected in Indonesia. Malayenolides A-D (1-4) showed toxicity to brineshrimp, LC50 100 µg/mL, <2 µg/mL, 20 µg/mL, respectively (Fu *et al.*, 1999).

CONCLUSION

Marine invertebrates, which are plentiful in the Indo-Pacific regions including Indonesia, are rich in secondary metabolites and are becoming targets for the continuing search for bioactive compounds. The new metabolites from Indonesian marine organisms were mainly isolated from sponges. Octocorals are also being studied with promising results for secondary metabolites. Soft corals are the dominant reef dwelling octocorals in Indonesia. Since 1997–2014, more than 20 publications have reported on the bioactive compounds from Indonesian soft corals such as *Cladiella* sp., *Lobophytum* sp. and *Sinularia* sp. (Putra and Murniasih, 2016).

The discovery of novel compounds from Indonesian sea pens and gorgonians is much rarer, as indicated by the fact that until now only three publications have described novel compounds from them. Terpenes and sterols represented the two main chemical classes of compounds discovered from Indonesian marine organisms.

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