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Original article

Isolation of toxic compounds from wild *Phaeocystis globosa*

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ABSTRACT

Two new compounds, namely taenialactam C and globorin A (**1** and **2**), as well as six known compounds, cornoside (**3**), 2-phenylethyl-β-D-glucoside (**4**), 3-isopropyl-5-acetoxycyclohexene-2-one-1 (**5**), 4-methyl-phenol (**6**), 5-[(2S)-2-aminobutyl]-2-methyl-phenol (**7**), and 1-(4-methylphenyl)-1-propanone (**8**) were isolated from wild *Phaeocystis globosa*. The structures of the new compounds were established by detailed spectroscopic analysis and by comparison with spectral data of related known compounds. The structures of the known compounds were identified by comparing their spectroscopic data with those reported in the literature. This paper also reports toxicity properties of the eight compounds against the brine shrimp *Artemia salina* and juvenile *Epinephelus akaara* fish. Some of these compounds showed significant lethality on the brine shrimp *A. salina* and the juvenile *E. akaara* fish.
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1. Introduction

Harmful algal blooms (HABs) are reported to occur worldwide, which have been a serious problem for aquaculture and fisheries operation and have bad impacts on human health [1]. The blooms of *Phaeocystis globosa* have frequently occurred along coastal waters and exert serious impacts on ecological environments by releasing toxic hemolytic substances, forming nuisance foam, and causing oxygen depletion [2]. Due to these severe negative effects, several studies have described methods of controlling the bloom of *P. globosa* [3,4].

However, the investigation on the chemical constituents of wild *P. globosa* and its biological activities are not reported yet. In the course of our phytochemical studies on this specimen, two new compounds, namely, taenialactam C and globorin A (**1** and **2**), as well as six known compounds, cornoside (**3**) [5], 2-phenylethyl-β-D-glucoside (**4**) [6], 3-isopropyl-5-acetoxycyclohexene-2-one-1 (**5**) [7], 4-methylphenol (**6**) [8], 5-[(2S)-2-aminobutyl]-2-methyl-phenol (**7**) [9], and 1-(4-methylphenyl)-1-propanone (**8**) [10] (Fig. 1) were isolated from *P. globosa*. We also report toxicity properties of the eight compounds against the brine shrimp *Artemia salina* and juvenile *Epinephelus akaara* fish.

2. Experimental

2.1. General experimental procedures

UV spectra were recorded in MeOH on a Perkin–Elmer Lambda 35 UV–vis spectrophotometer. The IR spectra were measured in KBr on a WQF-410 FT-IR spectrophotometer. NMR spectra were recorded on a Bruker AV 600 MHz NMR spectrometer with TMS as an internal standard. HRESIMS data were obtained from a Bruker Maxis mass spectrometer. HPLC was performed on a Waters-2695 HPLC system, using a Sunfire^{TM} C_{18} column (150 × 10 mm i.d., 10 μm) coupled to a Waters 2998 photodiode array detector. Optical rotation data were measured by Perkin–Elmer Model 341 polarimeter. The silica gel GF_{254} used for TLC was supplied by

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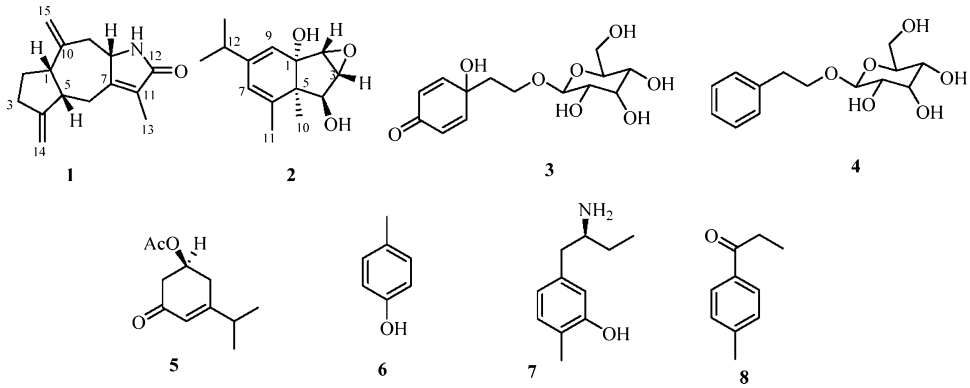

Fig. 1. Structures of compounds **1**–**8** isolated from wild *P. globosa*.

the Qingdao Marine Chemical Factory, Qingdao, China. Spots were detected on TLC under UV light or by heating after spraying with 5% H_2SO_4 in EtOH. All solvent ratios are measured v/v.

2.2. Plant material

Wild *P. globosa* was collected from the entry of Beilun River, Fangchenggang city, Guangxi province, China, in December 2012. The specimen was identified by Professor Songhui Lv from Jinan University. A voucher specimen (2012-GXAS-0015) was deposited in Guangxi Key Laboratory of Marine Environmental Science, Guangxi Academy of Sciences, China.

2.3. Extraction and isolation

P. globosa (35 L) was exhaustively extracted with EtOH (95%, 10 L) at 25 °C for 3 × 4 days. The solvent was evaporated *in vacuo* to afford a syrupy residue (45 g) that was suspended in distilled water (0.5 L) and fractionated successively with petroleum ether (3 × 0.5 L), ethyl acetate (3 × 0.5 L), and *n*-butanol (3 × 0.5 L). The ethyl acetate soluble portion (3.36 g) was subjected to column chromatography on silica gel, using $CHCl_3$-Me_2CO (from 10:0 to 0:10) as eluent, giving eleven fractions (A–K). Fraction C was subjected to column chromatography to afford ten sub-fractions (C1–C10). Sub-fraction C3 was subjected to Sephadex LH-20 column chromatography with $CHCl_3$-MeOH (1:1), then separated by HPLC, using the mixtures of MeOH-H_2O (35:65) to yield **5** (5.3 mg). Fraction D was subjected to column chromatography to afford eight sub-fractions (D1–D8). Sub-fraction D2 was subjected to Sephadex LH-20 column chromatography with $CHCl_3$-MeOH (1:1), then separated by HPLC, using the mixtures of MeOH-H_2O (70:30) to yield **1** (2.7 mg). Fraction F was subjected to column chromatography to afford ten sub-fractions (F1–F10). Sub-fraction F3 was subjected to Sephadex LH-20 column chromatography with $CHCl_3$-MeOH (1:1), then separated by HPLC, using the mixtures of MeOH-H_2O (60:40) to yield **8** (0.4 mg) and **3** (0.6 mg), respectively. Fraction H was subjected to column chromatography to afford ten sub-fractions (H1–H10). Sub-fraction H9 was subjected to Sephadex LH-20 column chromatography with $CHCl_3$-MeOH (1:1), then separated by HPLC, using the mixtures of MeOH-H_2O (15:85) to yield **7** (1.2 mg). The *n*-butanol soluble portion (7.83 g) was subjected to column chromatography on silica gel, using $CHCl_3$-Me_2OH (from 10:0 to 0:10) as eluent, giving seven fractions (B_a–B_g). Fraction B_f was subjected to column chromatography to afford ten sub-fractions (B_f1–B_f10). Sub-fraction B_f3 was subjected to Sephadex LH-20 column chromatography with $CHCl_3$-MeOH (0:100), then separated by HPLC, using the mixtures of MeOH-H_2O (10:90) to yield **2** (4.9 mg). Sub-fraction B_f4 was subjected to Sephadex LH-20 column chromatography with $CHCl_3$-MeOH (0:100), then separated by HPLC, using the mixtures of MeOH-H_2O (25:75) to yield **4** (1.8 mg). Fraction B_g was subjected to column chromatography to afford five sub-fractions (B_g1–B_g5). Sub-fraction B_g3 was subjected to Sephadex LH-20 column chromatography with $CHCl_3$-MeOH (0:100), then separated by HPLC, using the mixtures of MeOH-H_2O (30:70) to yield **6** (1.2 mg).

Taenialactam C (**1**): White solid; CD (MeOH) $\Delta\varepsilon_{220nm}$ + 4.58; ^{1}H NMR ($CDCl_3$, 600 MHz) and ^{13}C NMR ($CDCl_3$, 150 MHz), see Table 1; HRESIMS *m/z* 230.1549 (calcd. for $C_{15}H_{19}NO$ + H, 230.1539).

Globorin A (**2**): Yellow oil; CD (MeOH) $\Delta\varepsilon_{215nm}$ + 6.2; ^{1}H NMR (CD_3OD, 600 MHz) and ^{13}C NMR (CD_3OD, 150 MHz), see Table 3; HRESIMS: *m/z* 259.1298 (calcd. for $C_{14}H_{20}O_3$ + Na, 259.1305).

2.4. Toxicity test for marine organisms

The toxicity test was conducted with brine shrimp *A. salina* and grouper *E. akaara*. The procedures of the toxicity tests followed those described by the US Environmental Protection Agency for toxicity tests for aquatic organisms [11].

The shrimp lethality of the isolates was assessed following the reported method [12–14]. Brine shrimp eggs (Ocean Star International, Inc., USA) were hatched in a large beaker of natural seawater (South China Sea), then incubated at room temperature

Table 1
^{1}H NMR and ^{13}C NMR data of **1** and **2** (δ in ppm, *J* in Hz).

No.	**1**[a]		**2**[b]	
	δ_C	δ_H	δ_C	δ_H
1	45.9, CH	2.72–2.78 (m)	78.3, C	
2α	28.8, CH_2	1.29–1.33 (m)	72.8, CH	4.93 (d, 6.3)
β		1.25–1.30 (m)		
3α	31.8, CH_2	2.37 (dd, 10.2, 7.1)	67.3, CH	3.33 (dd, 6.7, 6.3)
β		2.30 (dd, 10.2, 3.4)		
4	156.4, C		79.6, CH	3.84 (d, 6.7)
5	50.0, CH	2.62 (m)	44.5, C	
6α	32.7, CH_2	2.37 (dd, 11.2, 6.4)	152.4, C	
β		2.30 (dd, 11.2, 3.4)		
7	157.2, C		129.7, CH	7.06 (s)
8	61.5, CH	3.84 (dd, 8.5, 3.0)	147.5, C	
9α	40.7, CH_2	2.76 (dd, 11.0, 8.5)	119.5, CH	7.15 (s)
β		2.72 (dd, 11.0, 3.0)		
10	145.5, C		18.4, CH_3	1.28 (s)
11	126.6, C		24.4, CH_3	2.23 (s)
12	174.8, C		36.7, CH	2.91–2.95 (m)
13	8.0, CH_3	1.82 (s)	24.6, CH_3	1.18 (d, 6.7)
14α	114.7, CH	5.04 (d, 9.0)	24.6, CH_3	1.18 (d, 6.7)
β		5.01 (d, 9.0)		
15α	107.1, CH	5.02 (d, 8.4)		
β		4.95 (d, 8.4)		

[a] In $CDCl_3$, 600 MHz for ^{1}H NMR band 150 MHz for ^{13}C NMR.
[b] In CD_3OD, 600 MHz for ^{1}H NMR and 150 MHz for ^{13}C NMR.

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