



Novel triterpenoids and glycosides from durian exert pronounced anti-inflammatory activities



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ABSTRACT

Durian, known for its abundant nutrition, is a delicious fruit from Southeast Asia with increasing popularity worldwide. In this study, a series of chromatographic methods and bioactivity assays were applied to identify major compounds from durian shells. Two new triterpenoids, two new phenolics, and seven new glycoside esters, as well as sixteen known compounds, were isolated and identified. Anti-inflammatory activities were assayed and evaluated for the isolated compounds. Most of the isolated compounds exhibited pronounced inhibitory activities on lipopolysaccharides-induced NO production in RAW 264.7 cells. The results indicated that durian shells could serve as anti-inflammatory agents for functional food or medicinal use. This study additionally provided motivation for the ecological protection of durian shells.

1. Introduction

Durian (*Durio zibethinus* Murr.) is a species of the genus *Durio* and is cultivated in Southeast Asia. Known as a typical tropical fruit, it is native to Southeast Asia and has been introduced worldwide. The fruit of durian has an oval or spherical shape and is approximately 15–30 cm long. With an overpowering aroma, durian has a special attraction for some people (Li, Schieberle, & Steinhaus, 2012) and has been regarded as the king of fruits for its abundant nutrition. However, the spiny and hard shell of durian is difficult to dispose and is treated as agricultural waste. With increasing environmental awareness, the potential value of these shells are being evaluated. Currently, durian shells have been utilized in architectural engineering due to their hardness. As found in previous phytochemical studies, the genus *Durio* contain bioactive components, such as triterpenoids, coumarins, phenolics, lignans, flavonoids, sulfur-containing compounds, and some esters (Lambert & Garson, 2010; Moses et al., 2012; Rudiyanisya & Garson, 2006).

Epidemiology studies have verified that high fruit intake plays an important role in preventing chronic diseases, such as inflammatory, atherosclerosis, cancer, diabetes, and coronary heart diseases (Zineb, Boukouada, Djeridane, Saidi, & Yousfi, 2012). The pronounced anti-inflammatory activities of fruits and their byproducts have been

confirmed by many studies (Hasnaoui, Wathelet, & Jimenez-Araujo, 2014; Mueller, Triebel, Rudakovski, & Riching, 2013). Presently, a growing number of studies have concentrated on revealing anti-inflammatory factors from fruits, resulting in the identification of novel anti-inflammatory compounds. In the process of seeking bioactive components from medicinal plants, fruits, and vegetables (Cheng, Yi, Wang, Huang, & He, 2017; Feng, Wang, Yi, Yang, & He, 2016; Pan, Yi, Wang, Chen, & He, 2016), the systemic phytochemical characterization of durian shell extracts was conducted. Eleven new and sixteen known compounds were purified and identified. The potent anti-inflammatory activities of these isolates were also evaluated and discussed.

2. Materials and methods

2.1. General experimental procedures

Melting points were measured on a Beijing Taike X-5 micromelting point apparatus (Beijing, China). Optical rotations were acquired on a Jasco P-2200 digital polarimeter (Jasco Inc., Tokyo, Japan). IR spectra were obtained on a PerkinElmer 100 IR spectrometer (PerkinElmer Inc., Waltham, MA, USA), and a Bruker Avance III-400 NMR spectrometer (Bruker Inc., Fällanden, Switzerland) was used to acquire NMR spectra. High-resolution ESI-MS (HR-ESI-MS) data were obtained by a Waters

Abbreviations: CC, column chromatography; ¹H–¹H COSY, ¹H–¹H correlation spectroscopy; HMBC, heteronuclear multiple-bond correlation; HR-ESI-MS, high-resolution electrospray ionization mass spectrometry; HSQC, heteronuclear single-quantum coherence; LPS, lipopolysaccharides; MPLC, medium-pressure liquid chromatography; MTT, 3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide; NO, nitric oxide; RAW 264.7, murine macrophage

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Table 1
NMR data of compounds **1** and **2** (δ in ppm and J in Hz).^{a–c}

Position	1		2		Position	1		2	
	δ_{H}	δ_{C}	δ_{H}	δ_{C}		δ_{H}	δ_{C}	δ_{H}	δ_{C}
1	1.32 m, 2.30 m	45.0	1.30 m, 2.27 m	45.0	21	1.10–1.48 ov	34.5	1.08–1.46 ov	34.6
2	5.83 m	74.1	5.77 m	74.0	22	1.10–1.48 ov	30.3	1.08–1.46 ov	30.4
3	4.53 d (9.9)	74.3	4.47 d (9.9)	74.2	23	3.75 d (10.6)	65.8	3.72 d (10.6)	65.9
4		44.7		44.7		4.26 d (10.6)		4.23 d (10.6)	
5	1.58–2.33 ov	47.7	1.55–2.30 ov	47.9	24	1.10 s	14.7	1.09 s	14.8
6	1.58–2.33 ov	18.8	1.55–2.30 ov	18.8	25	1.18 s	17.5	1.17 s	17.5
7	1.58–2.33 ov	33.1	1.55–2.30 ov	33.1	26	1.05 s	17.8	1.04 s	17.8
8		40.2		40.2	27	1.26 s	26.5	1.25 s	26.6
9	1.58–2.33 ov	48.4	1.55–2.30 ov	48.4	28		180.6		180.6
10		38.9		38.9	29	0.91 s	33.5	0.91 s	33.6
11	1.58–2.33 ov	24.0	1.55–2.30 ov	24.0	30	0.98 s	24.1	0.97 s	24.1
12	5.52 br s	122.7	5.50 br s	122.7	1'		126.5		127
13		145.3		145.3	2'	7.26 d (8.5)	130.9	8.11 d (8.5)	134.1
14		42.6		42.6	3'	7.16 d (8.5)	117.1	7.14 d (8.5)	116.3
15	1.58–2.33 ov	28.6	1.55–2.30 ov	28.7	4'		161.7		160.9
16	1.58–2.33 ov	24.3	1.55–2.30 ov	24.3	5'	7.16 d (8.5)	117.1	7.14 d (8.5)	116.3
17		47.0		47.0	6'	7.26 d (8.5)	130.9	8.11 d (8.5)	134.1
18	3.31 m	42.3	3.31 m	42.3	7'	7.96 d (15.9)	144.9	6.85 d (12.9)	143.9
19	1.10–1.48 ov	46.7	1.08–1.46 ov	46.8	8'	6.55 d (15.9)	116.4	5.97 d (12.9)	117.4
20		31.3		31.3	9'		168.0		167.3

^aMeasured in DMSO- d_6 .

^bAssignments were based on HSQC, HMBC, and ^1H - ^1H COSY experiments.

^cm, signal multiplicity pattern is unclear; ov, signals overlapped.

Aquity UPLC/Q-TOF mass spectrometer (Milford, MA, USA). Semi-preparative HPLC was applied with a Rainin pump (Rainin Instrument Co. Inc., Woburn, MA, USA), a refractive index detector, and a Cosmosil HPLC column (5C18-MS-II, 10ID \times 250 mm, Nacal Tesque, Kyoto, Japan). Silica gel for column chromatography and TLC was purchased from Anhui Liangchen Silicon Material Co. Ltd. (Lu'an, China). (*S*)-(+)-2-Methylbutyric acid was purchased from Acoros organics (Belgium), and the sugar reagents for GC-MS analysis were purchased from Sigma (St. Louis, MO, USA). HPLC analysis was applied with a Waters 600 and 996 photodiode array detector (Milford, MA, USA). GC for sugar analysis was analysed on a Varian CP-3800 (Palo Alto, CA, USA).

2.2. Plant material

Ripe durian shells were collected in Sep. 2014 from the local markets of Guangzhou (Guangzhou, China) and were stored at room temperature. The shells were identified by Prof. X.J. He of Guangdong Pharmaceutical University. A voucher specimen (No. GDU-201403) was deposited in the Lead Compounds Laboratory, School of Pharmacy, Guangdong Pharmaceutical University.

2.3. Extraction, isolation, and purification procedures

Air-dried shells (20 kg) were pulverized to 40 mesh and extracted four times with 70% EtOH under reflux. After filtration and concentration, the extract was suspended in 15 L distilled water and successively extracted with cyclohexane, chloroform, ethyl acetate, and *n*-butanol to yield a CHCl_3 -soluble fraction (50.0 g), an EtOAc-soluble fraction (68.1 g), and an *n*-BuOH-soluble fraction (280.0 g), respectively.

The CHCl_3 -soluble fraction (50.0 g) was fractionated by silica gel column chromatography (CC) and eluted with $\text{CHCl}_3/\text{MeOH}$ (100:1 to 1:1). Ten subfractions (A1–A10) were obtained on the basis of TLC analysis. Fraction A2 (1.5 g) was further separated by a silica gel CC to yield 5 subfractions (A2-1 to A2-5). Fraction A2-3 was subjected to ODS MPLC and eluted with $\text{MeOH-H}_2\text{O}$ (5:5 to 9:1), followed by a semi-preparative C_{18} -HPLC using 75% $\text{MeOH-H}_2\text{O}$ as mobile phase to obtain compounds **5** (12.1 mg), **7** (8.2 mg), and **8** (17.1 mg). Fraction A3

(6.33 g) was subjected to a silica gel column to obtain 8 fractions (A3-1 to A3-8). Fraction A3-3 was further fractionated over ODS MPLC eluted with $\text{MeOH-H}_2\text{O}$ (5:5 to 9:1) to obtain 7 fractions. The third subfraction was purified by a semi-preparative C_{18} -HPLC using 65% $\text{MeOH-H}_2\text{O}$ as mobile phase to obtain compounds **6** (7.3 mg), **9** (24.3 mg), and **10** (18.3 mg).

The ethyl acetate-soluble fraction (68.1 g) was fractionated by silica gel CC and eluted with $\text{CHCl}_3/\text{MeOH}$ by increasing polarity to yield 8 fractions (B1–B8). Fraction B3 (18.2 g) was further separated by silica gel CC to obtain 9 fractions (B3-1 to B3-9). Fraction B3-3 was subjected to ODS MPLC and eluted with $\text{MeOH-H}_2\text{O}$ (5:5 to 8:2) to obtain 4 fractions. The second fraction was purified by semi-preparative HPLC and eluted with 60% $\text{MeOH-H}_2\text{O}$ to obtain compounds **1** (8.7 mg) and **3** (14.8 mg). Compounds **2** (9.1 mg) and **4** (6.3 mg) were isolated from the third fraction by semi-preparative HPLC using 60% $\text{MeOH-H}_2\text{O}$ as the mobile phase. Fraction B4 was purified by a similar method to obtain compound **19** (15.7 mg).

The *n*-butanol-soluble fraction (280 g) was fractionated by a D101 macroporous resin column and was eluted with H_2O , 20% MeOH , 40% MeOH , 70% MeOH , and 100% MeOH to obtain 5 fractions (C1–C5). Fraction C2 (25.3 g) was subjected to a silica gel CC and gradient eluted with $\text{CHCl}_3/\text{MeOH}$ to obtain 8 fractions (C2-1 to C2-8). Fraction C2-4 (5.3 g) was further isolated by ODS MPLC using a Sephadex LH-20 column (cyclohexane/ $\text{CHCl}_3/\text{MeOH}$, 5:5:1, v/v/v) and was purified using a semi-preparative C_{18} -HPLC with 30% $\text{MeOH}/\text{H}_2\text{O}$ as the mobile phase to obtain compounds **11** (13.6 mg), **12** (8.8 mg), **13** (3.3 mg), **14** (13.0 mg), **20** (15.7 mg), and **21** (14.8 mg). Fraction C3 (17.1 g) was subjected to silica gel CC and eluted with $\text{CHCl}_3/\text{MeOH}$ (60:1 to 1:1) to obtain 7 subfractions (C3-1 to C3-7). Subfraction C3-4 (1.1 g) was subjected to a Sephadex LH-20 column (cyclohexane/ $\text{CHCl}_3/\text{MeOH}$, 5:5:1, v/v/v), followed semi-preparative HPLC ($\text{MeOH}/\text{H}_2\text{O}$, 20:80, v/v) to obtain compounds **15** (13.1 mg), **16** (3.4 mg), **17** (6.4 mg), and **18** (13.2 mg). Compound **22** (14.3 mg) was isolated from the subfraction C3-6.

2a-Trans-p-coumaroyloxy-2a,3 β ,23a-trihydroxy-olean-12-en-28-oic acid (**1**). $[\alpha]_{\text{D}}^{25} + 10.2$ (c 0.30, MeOH); UV (MeOH) λ_{max} 249, 312 nm; IR (KBr) ν_{max} 3392 (OH), 2928 (CH_2), 1686 (C=O), 1605, 1514 cm^{-1} ; ^1H and ^{13}C NMR data, see Table 1; HR-ESI-MS m/z 657.3800 [$\text{M} + \text{Na}$]⁺ (calcd for $\text{C}_{39}\text{H}_{54}\text{O}_7$ Na, 657.3736).

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