Food Sci. Biotechnol. 23(4): 1081-1085 (2014)
DOI 10.1007/s10068-014-0148-7

RESEARCH NOTE

Two Novel Glycosyl Cinnamic and Benzoic Acids from Korean Black Raspberry (*Rubus coreanus*) Wine

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Received October 19, 2013; revised January 7, 2014; accepted January 12, 2014; published online August 31, 2014

Abstract Korean black raspberry (*Rubus coreanus*) wine was solvent-fractionated with *n*-hexane, ethyl acetate, and *n*-butanol and the *n*-butanol layer was purified by various column chromatographic procedures, including Amberlite XAD-2, Sephadex LH-20, and octadecylsilane resins as well as high performance liquid chromatography. Two novel glycosyl cinnamic and benzoic acids were isolated and their structures were (*E*)-8-*O*-β-D-glucopyranosylcinnamic acid (**1**) and 3'-[*O*-β-D-glucopyranosyl)(1''→6')-α-D-psicofuranosyl] benzoate (**2**) based on the spectroscopic data obtained by high resolution fast-atom bombardment mass spectroscopy and nuclear magnetic resonance analyses.

Keywords: Korean black raspberry wine, *Rubus coreanus*, (*E*)-8-*O*-β-D-glucopyranosylcinnamic acid, diglycosyl benzoic acid, glucopyranosyl psicofuranose

Introduction

Korean black raspberry (KBR, *Rubus coreanus* Miquel.) is a member of Rosaceae family and has been used in traditional folk remedies for liver protection as well as for treating enuresis, asthma, and spermatorrhea in Korea (1,2). In addition, many studies have reported that the fruit exerts various biological effects including anticancer (3), antimicrobial (4), antioxidant (5-7), hepatitis B virus inhibitory (8), antinociceptive, and anti-inflammatory (9) activities. Moreover, the bioactive components of KBR fruit including anthocyanins (10,11), triterpenoids (12,13), flavonoids (6,7), and tannins (14,15) have been identified. KBR fruit has been used in various processed foods in Korea (16). In particular, KBR wine (*bokbunja ju*), which is made by fermenting the ripened fruit, has a deep red color and sweet taste and is a popular Korean alcoholic beverage (16). KBR wine also exerts biological activities including reducing serum cholesterol (17) as well as antioxidant and anti-inflammatory (18), and anticancer (16) effects. Various metabolites from the constituents of KBR fruit produced during KBR wine preparation may contribute to its biological activities. Understanding the constituents of KBR wine is very important in regards to acquiring basic information on processing, preservation, and the biological function of KBR wine. However, studies on the chemical constituents of KBR wine are limited in comparison to those of KBR fruit. Therefore, the chemical constituents contained in KBR wine were investigated. In our previous study, 10 phenolic and volatile compounds were found from the ethyl acetate (EtOAc) layer of KBR wine (19,20). In addition, changes in total phenolics, total anthocyanin, gallic acid (GA), and 3,4-dihydroxybenzoic acid contents and antioxidative activity during KBR wine manufacturing were investigated (21). In the course of our investigation on chemical constituents from KBR wine, two novel phenolic glycosides were additionally isolated. This study describes the isolation and structural elucidation of the two novel glycosyl cinnamic and benzoic acids from the water-saturated *n*-butanol (BuOH) layer of KBR wine based on the NMR and high resolution-fast-atom bombardment mass spectroscopy (HR-FAB-MS) analyses.

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Materials and Methods

General experimental procedures NMR spectra were obtained with an ^{unit}INOVA 500 spectrometer (Varian, Walnut Creek, CA, USA) using the solvent as the internal

standard. The isolated compounds were dissolved in CD_3OD. Mass spectral data were obtained using HR-FAB-MS (JMS-HX100; Jeol, Tokyo, Japan) with a matrix ingredient (3-nitrobenzyl alcohol). Column chromatography was performed with Amberlite XAD-2 (20-60 mesh; Sigma, St. Louis, MO, USA), Sephadex LH-20 (25-100 mesh; GE Healthcare Bio-Sciences AB, Uppsala, Sweden), and octadecylsilane (ODS, 170 mesh; YMC, Kyoto, Japan) resins. HPLC separations were performed on a SPD-M20D system (Shimadzu, Kyoto, Japan) with ODS columns [ODS-80Ts column (4.6 i.d.×250 mm, 5 μm; Tosoh, Kyoto, Japan) and μBondapak (7.8 i.d.×300 mm, 10 μm; Waters, Milford, MA, USA)] and RP-amide C_{16} (4.6 i.d.×250 mm; Supelco, Bellefonte, PA, USA). Detection was accomplished at 280 nm with a flow rate of either 1.0 or 2.0 mL/min. TLC was performed on silica gel 60 F_{254} (0.25-mm thickness; Merck, Darmstadt, Germany).

Materials and chemicals The fruits of *R. coreanum* were collected in June 2009 from Naju city, Korea. A voucher sample was deposited in the warm-temperate forest arboretum located on Bogil Island, Chonnam National University (Gwangju, Korea).

Preparation of KBR wine The manufacture of the KBR wine was carried out according to the mass-production method (19) of Yeon-Su Dang Manufacturing Co. (Gwangju, Korea). Briefly, KBR wine (11.7 L) was prepared from ripened KBR fruit (15.7 kg) without the addition of other ingredients. The alcoholic content of KBR wine was about 13.0%.

Extraction and purification The KBR wine (11.7 L, 15.7 kg fresh fruit equivalent) was concentrated *in vacuo* to remove the ethanol. The concentrated aqueous layer (2.8 L) was successively partitioned with *n*-hexane (3 L, 3 times), EtOAc (3 L, 3 times), and BuOH (3 L, 3 times). The BuOH layer (138.5 g) was fractionated by chromatography on an Amberlite XAD-2 column (8.0 i.d.×58 cm) and eluted with H_2O/MeOH=8:2, 6:4, 4:6, 2:8, and 0:10 (v/v, each 5.6 L). Each column chromatographic fraction was spotted on a silica gel TLC plate and developed using a mixture of MeOH/acetic acid/water (7:1:2, v/v/v). The spots were detected by UV light (254 and 365 nm). Thirty-one fractions (BL1-HL31) were separated by Amberlite XAD-2 column chromatography of the BuOH layer. Fraction BL8 (4.9 g, H_2O/MeOH=8:2, v/v) was chromatographed on a Sephadex LH-20 column (2.0 i.d.×132 cm, bed volume 480 mL) using 100% MeOH as an elution solvent to obtain 5 fractions (BL8.1-BL8.5). BL8.3 (2.17 g) was further fractionated by Sephadex LH-20 (1.5 i.d.×80 cm, bed volume 480 mL) column chromatography using 10% MeOH as an elution solvent to afford 3 fractions (BL8.3a-BL8.3c). Fraction BL8.3c (464 mg) was further fractionated with an ODS column (2.3 i.d.×53 cm) and eluted with H_2O/MeOH (90:10, 80:20, 75:25, 70:30, 65:35, 60:40, 55:45, 50:50, v/v, each 350 mL) to give 5 fractions (BL8.3c1-BL8.3c5). Fraction BL8.3c4 (H_2O/MeOH= 65:35, v/v, 25 mg) was subjected to HPLC using an ODS-80Ts column and a gradient system of 20% MeOH [pH 2.6, by trifluoroacetic acid (TFA), eluent A] to 80% MeOH (eluent B): 100% A was started, increased to 100% B over 40 min, and held at 100% B until 50 min. After repeated purification, compound **1** (t_R 14.8 min, 1.6 mg, white amorphous powder) was isolated.

Fraction BL10 (4.9 g, H_2O/MeOH=6:4, v/v), obtained after Amberlite XAD-2 column chromatography, was fractionated on a Sephadex LH-20 column (1.5 i.d.×80 cm, bed volume 480 mL) using 100% MeOH as the eluent to obtain 11 fractions (BL10.1-BL10.11). Fraction BL10.4 (48.9 mg) was subjected to HPLC equipped with a μBondapak column with a gradient system of 20% MeOH (pH 2.6, TFA) to 100% MeOH; 100% A was held for 5 min, increased to 100% B over 35 min, and held at 100% B until 50 min. After repeated purification, 11 fractions including BL10.3d were purified. Finally, fraction BL10.3d (19.0 mg) was subjected to HPLC using an RP-amide C_{16} (4.6 i.d.×250 mm) column and eluting with 5% MeOH (pH 2.6, TFA) as a mobile phase to obtain **2** (t_R 15.3 min, 2.4 mg).

Results and Discussion

After solvent fractionation of the KBR wine (11.7 L, 15.7 kg fresh fruit equivalent), *n*-hexane (0.2 g), EtOAc (56.2 g), BuOH (138.5 g), and aqueous (731.5 g) layers were obtained. Of them, the EtOAc layer has been investigated in our previous studies to isolate and identify the chemical constituents in KBR wine (19,20). In the present study, the BuOH layer was used for a series of investigations on screening of useful compounds from KBR wine. The BuOH layer (138.5 g) of the KBR wine (11.7 L, 15.7 kg fresh fruit equivalent) was fractionated by various column chromatographic procedures, including Amberlite XAD-2, Sephadex LH-20, and ODS as well as HPLC to obtain two novel compounds (**1**, 1.6 mg; **2**, 2.4 mg). Compounds **1** and **2** were structurally elucidated as novel glycosyl cinnamic and benzoic acids based on NMR and MS spectroscopic data.

Compound **1** was isolated as a white amorphous powder. The molecular formula ($C_{15}H_{18}O_8$) of **1** was determined by HR-FAB-MS (positive) spectrum (*m/z* 327.1077 $[M+H]^+$; calculated for $C_{15}H_{19}O_8$, *m/z* 327.1077, 0 *mmu*). The ^{13}C-NMR (125 MHz, CD_3OD) spectrum revealed the presence of nine carbon signals assignable to a phenylpropanoid

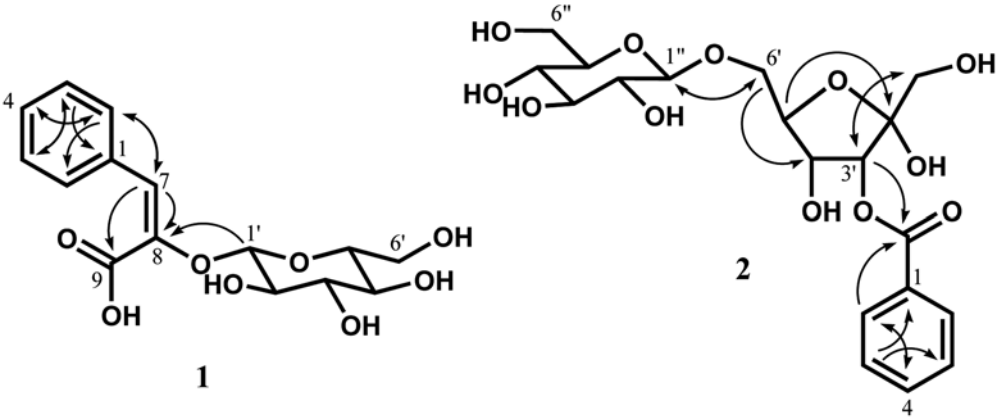

Fig. 1. Structures and HMBC correlations (arrows) of the compounds isolated from KBR wine.

including a carbonyl carbon signal at δ 167.3 (C-9) and eight other carbon signals at δ 142.9-126.0, and in addition to six carbon signals assignable to a sugar moiety at δ 103.0 (C-1'), 78.2 (C-2'), 75.8 (C-3'), 71.5 (C-4'), 78.8 (C-5'), and 62.7 (C-6'). That is, the ^{1}H-NMR (500 MHz, CD_3OD) spectrum of **1** showed the presence of mono-substituted benzene ring proton signals [δ 7.86 (2H, d, *J*=7.5 Hz, H-2, 6), 7.35 (2H, t, *J*=7.5 Hz, H-3, 5), and 7.30 (1H, d, *J*=7.5 Hz, H-4)] and a proton signal [δ 7.05 (1H, s, H-7)] of a double bond, suggesting that aglycone of **1** was hydroxylcinnamic acid. Sugar proton signals including an anomeric proton signal [δ 5.21 (1H, d, *J*=8.0 Hz, H-1')] and other proton signals [δ 3.75-3.23 (6H, H-2'~H-6')] were also found. The configuration of β-D-glucopyranose was indicated by the proton-proton correlations in the ^{1}H-^{1}H correlation spectroscopy (COSY) spectrum and the coupling constant values (*J*=8.0-9.0 Hz) of the sugar protons in the ^{1}H-NMR spectrum. In addition, all carbons were assigned according to the results of the heteronuclear single quantum correlation (HSQC) analysis. The aglycone moiety was assigned as 8-hydroxycinnamic acid on the basis of ^{1}H-^{13}C long-range correlations in the heteronuclear multi-bond correlation (HMBC) spectrum (Fig. 1). In addition, the correlation between H-1' (δ 5.21) and C-8 (δ 142.9) indicated that glucose was etherified at the C-8 position of 8-hydroxycinnamic acid. Therefore, the structure of **1** was elucidated to be 8-*O*-β-D-glucopyranosylcinnamic acid. However, the stereoisomeric structure in the olefinic double bond of the aglycone was not yet clear. Therefore, compound **1** was additionally analyzed by ^{1}H-NMR with D_2O to compare with data of a known compound. That is, the chemical shift (δ 7.07, 1H, s, H-7) of the olefinic double bond proton signal in the ^{1}H-NMR spectrum (500 MHz, D_2O, Table 1) of **1** was observed down-field at 0.35 ppm when compared to that (δ 6.72) of (*Z*)-8-*O*-β-D-glucopyranosylcinnamic acid previously identified from the leaves of *Onobrychis viciifolia* (22). Consequently, the structure of **1** was elucidated as (*E*)-8-*O*-β-D-glucopyranosylcinnamic acid (Fig. 1).

Compound **2** was isolated as a white amorphous powder. Its molecular formula ($C_{19}H_{26}O_{12}$) was obtained from the HR-FAB-MS (*m/z* 469.1327 $[M+Na]^+$; calculated for $C_{19}H_{26}O_{12}Na$, *m/z* 469.1327, 0.0 *mmu*) data. The ^{1}H-NMR (500 MHz, CD_3OD) spectrum of **2** revealed the presence of mono-substituted benzene ring proton signals [δ 8.08 (2H, δ, *J*=7.5 Hz, H-2, 6), 7.49 (2H, t, *J*=7.5 Hz, H-3, 5), and 7.61 (1H, t, *J*=7.5 Hz, H-4)] (Table 2). This was supported by the ^{13}C-NMR (125 MHz, CD_3OD) spectrum, which showed the presence of carbon signals assignable to a benzoyl moiety at δ 168.0 (C-7), 134.4 (C-4), 131.8 (C-1), 130.0 (C-2, 6), and 129.7 (C-3, 5). In addition, 12 carbon signals of two sugar moieties at δ 104.8-62.4 were observed. The configuration of β-D-glucopyranose was assigned by the proton-proton correlations in the ^{1}H-^{1}H COSY experiments and their coupling constant values (*J*=7.5-9.0 Hz) in the ^{1}H-NMR spectrum. The other sugar was assigned as α-D-psicofuranose by the ^{1}H-NMR, ^{1}H-^{1}H COSY, HSQC, and nuclear Overhauser effect (NOE) experiments. That is, a series of correlations in cross peaks of H-3'/H-4', H-4'/H-5', and H-5'/H-6' in the ^{1}H-^{1}H COSY spectrum were found. The coupling constant (*J*) of H-3' (δ 5.31) and H-4' (δ 4.10) was 3.5 Hz. The spatial interactions between δ 4.17 (H-1') and δ 5.31 (H-3'), 4.10 (H-4'), and 3.96 (H-6') were observed in the NOE experiment (data not shown). In addition, the HMBC spectrum of **2** showed correlations between the anomeric proton (δ 4.43, C-1") of glucose and the methylene carbon signal (δ 72.9, C-6') of psicose (Fig. 1). The disaccharide moiety of **2** was assigned as α-D-(*O*-β-D-glucopyranosyl)(1"→6')-psicofuranose. Moreover, the cross peaks of H-3' (δ 5.31) and the carbonyl carbon signal (δ 168.0, C-7) indicated that benzoic acid was esterified with the C-3' position of psicose. Therefore, the structure of **2** was determined to be 3'-[*O*-β-D-glucopyranosyl) (1"→6')-

Table 1. ^{1}H- (500 MHz) and ^{13}C- (125 MHz) NMR data of compound 1

Position	δ_C[1)]	δ_H (*rel. int.*, *multi*, *J* in Hz)[1)]	δ_H (*rel. int.*, *multi*, *J* in Hz)[2)]
1	134.9	-	-
2, 6	131.8	7.86 (2H, d, 7.5)	7.78 (2H, d, 7.0)
3, 5	129.5	7.35 (2H, t, 7.5)	7.37 (2H, t, 7.0)
4	130.2	7.30 (1H, t, 7.5)	7.35 (1H, t, 7.0)
7	126.0	7.05 (1H, s)	7.07 (1H, s)
8	142.9	-	-
9	167.3	-	-
1'	103.0	5.21 (1H, d, 8.0)	5.00 (1H, d, 7.8)
2'	78.2	3.49 (1H, dd, 9.0, 8.0)	3.49 (1H, dd, 9.0, 7.8)
3'	75.8	3.41 (1H, dd, 9.0, 9.0)	3.43 (1H, dd, 9.0, 9.0)
4'	71.5	3.36 (1H, dd, 9.0, 9.0)	3.36 (1H, dd, 9.0, 9.0)
5'	78.8	3.23 (1H, ddd, 12.0, 5.5, 2.5)	3.28 (1H, m)
6'a	62.7	3.75 (1H, dd, 12.0, 2.5)	3.69 (1H, dd, 12.0, 2.5)
6'b		3.61 (1H, dd, 12.0, 5.5)	3.56(1H, dd, 12.0, 5.5)

[1)] ^{1}H- and ^{13}C-NMR spectra were determined in CD_3OD.
[2)] ^{1}H-NMR spectrum was determined in D_2O.

Table 2. ^{1}H- (500 MHz) and ^{13}C- (125 MHz) NMR data of compound 2 in CD_3OD

Position	δ_H (*rel. int.*, *multi*, *J* in Hz)	δ_C
1	-	131.8
2, 6	8.08 (2H, d, 7.5)	130.0
3, 5	7.49 (2H, t, 7.5)	129.7
4	7.61 (1H, t, 7.5)	134.4
7	-	168.0
1'a	4.17 (1H, d, 12.8)	62.4
1'b	3.79 (1H, d, 12.8)	
2'	-	99.2
3'	5.31 (1H, d, 3.5)	74.7
4'	4.10 (1H, dd, 10.0, 3.5)	70.0
5'	3.94 (1H, d, 10.0)	70.8
6'a	3.96 (1H, br. d., 12.5)	72.9
6'b	3.84 (1H, br. d, 12.5)	
1"	4.43 (1H, d, 7.5)	104.8
2"	3.26 (1H, dd, 9.0, 7.5)	75.3
3"	3.37 (1H, dd, 9.0, 7.5)	78.1
4"	3.31 (1H)[1)]	71.6
5"	3.28 (1H, m)	78.2
6"a	3.86 (1H, dd, 12.0, 1.2)	62.8
6"b	3.65 (1H, dd, 12.0, 5.0)	

[1)] Chemical shifts of H-4" and solvent overlapped.

α-D-psicofuranosyl] benzoate (Fig. 1).

We isolated two novel glycosyl cinnamic and benzoic acids from the BuOH layer of KBR wine. These novel compounds were structurally elucidated as (*E*)-8-*O*-β-D-glucopyranosylcinnamic acid (**1**) and 3'-[*O*-β-D-glucopyranosyl)(1"→6')-α-D-psicofuranosyl] benzoate (**2**) (Fig. 1). These compounds have not been identified in nature. However, (*Z*)-8-*O*-β-D-glucopyranosylcinnamic acid, which is an isomer of **1**, has been previously identified from the leaves of *O. viciifolia.* Therefore, compound **1** might have been produced during wine manufacturing or occurred during isolation of the *Z* form. In addition, glucopyranosylpsicofuranose contained as a partial structure in **2** is produced by glucosyltransferase from microorganisms (23). Therefore, compound **2** might be produced by the action of microorganisms during wine manufacturing. However, we do not know whether the two novel glycosyl cinnamic and benzoic acids originated in KBR wine or its fresh fruit. Therefore, the occurrence of the two novel glycosyl cinnamic and benzoic acids in KBR wine or its fresh fruit as well as their biological effects should be further studied.

Disclosure The authors declare no conflict of interest.

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