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ORIGINAL ARTICLE

Constituents from *Bupleurum montanum* (Coss. & Dur.) (Apiaceae)

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KEYWORDS

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Abstract A chemical investigation of the aerial parts of *Bupleurum montanum* (Coss. & Dur.) (Apiaceae) afforded five compounds, quercitin **1**, tamarexetin **2**, isorhamnetin-3-rutinoside **3**, kaempferol-3-*O*-β-rutinoside **4**, and 3,4-dihydroxybenzoic acid (Protocatechuic acid) **5**. The structural elucidation was performed mainly by MS, 1D and 2D NMR spectrum data.

1. Introduction

The genus *Bupleurum*, family Apiaceae, is widespread in the Mediterranean. The flora of Algeria contains 14 species of *Bupleurum* with 5 endemic species (*Bupleurum plantagineum* Desf., *Bupleurum atlanticum* Murb., *Bupleurum montanum* Coss., *Bupleurum balansae* Boiss. et Reut., *Bupleurum oligactis* Boiss.) (Quezel and Santa, 1963).

In previous phytochemical studies of *B. montanum*, we have reported the study of the chemical composition and antimicrobial activity of essential oil (Laouer et al., 2009). In the present and in continuation of our studies on Algerian Apiaceous plants (Bousetla et al., 2005; Benahmed et al., 2006, 2008) we describe the isolation and structural determination of five compounds from aerial parts of *B. montanum*. Compounds **1**–**5** were identified by GC–MS analysis and (UV, MS, ^{1}H NMR, and ^{13}C NMR spectroscopy) as: quercitin **1**, tamarexetin **2**, isorhamnetin-3-rutinoside **3**, kaempferol-3-*O*-β-rutinoside **4**, and 3,4-dihydroxybenzoic acid **5** (see Fig. 1).

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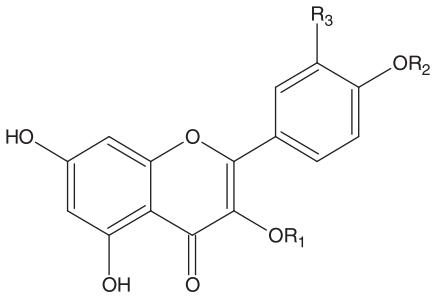
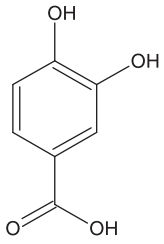


	R_1	R_2	R_3
1	H	H	OH
2	H	CH_3	OH
3	Rha(1→6) Glc	H	OCH_3
4	Rha(1→6) Glc	H	H



5

Fig. 1 Chemical structures of compounds **1**–**5**.

2. Experimental

2.1. General procedures

Ultra-violet absorption spectrum was recorded on Perkin–Elmer Lambda 2 UV spectrophotometer. NMR spectra were recorded on Bruker DMX 300 and chemical shifts are shown in δ values (ppm) with tetramethylsilane (TMS) as an internal reference. EI-MS data were obtained on a Hewlett Packard 5973 A quadripole mass spectrometer. Silica gel F_{254} was used for TLC. Spots were detected on TLC under UV light. Column chromatography was carried out on silica gel 60 Merck (6–35 μm, 20–45 μm, 70–200 μm).

2.2. Plant material

Aerial parts of *B. montanum* (Coss. & Dur.) (Apiaceae) were collected from Megress Mountain, Sétif, (Eastern Algeria) at 1500 m above sea level during the flowering period in Jun (2006), and identified by Pr. H. Laouer (Department of Biology, University Ferhat Abbas, Sétif, Algeria). A voucher specimen (B-6307) has been deposited in the Muséum d'Histoire naturelle de la ville de Nice, France.

2.3. Extraction and isolation

The air dried aerial parts of *B. montanum* (1000 g) were extracted three times with boiling 70% MeOH. The hydro-alcoholic solutions were concentrated under reduced pressure to dryness and the residue was dissolved in hot water and kept in cold overnight. After filtration, the aqueous solution was successively treated with ethylacetate and *n*-butanol, then the EtOAc and *n*-BuOH extracts were concentrated to dryness (Benahmed et al., 2006, Akkal et al., 2010).

The EtOAc extract (5 g) was chromatographed on a silica gel column by gradient elution with CH_2Cl_2/MeOH with increasing polarity. Four main fractions (A1–A4) were collected. Each fraction was then subjected to repeated chromatography on silica gel by column chromatography and thin layer chromatography to yield compounds **1** (8 mg), **2** (9.3 mg) and **3** (8.2 mg). The *n*-BuOH extract was subjected to a silica gel column chromatography by elution with a gradient of CH_2Cl_2/MeOH with increasing polarity to give five fractions (B1–B5). Fraction B2 (3.5 g) was further separated by silica gel column chromatography eluting with EtOAc/MeOH/H_2O (25:3:1) to obtain **4** (40 mg). Compound **5** was obtained from fractions B4 and B5 which were combined and subjected to a silica gel column chromatography then to TLC on silica gel using EtOAc/MeOH/H_2O (25:3:1) as eluant.

2.3.1. Quercitin (*1*)

^{1}H NMR (300 MHz, in ppm, DMSO-d_6): δ 7.5 (^{1}H, d, J = 2.1 Hz, H-2′), 7.3 (1H, dd, J = 8.5 Hz, J = 2.1 Hz, H-6′), 6.7 (1H, d, J = 8.5 Hz, H-5′), 6.3 (1H,d, J = 1.9 Hz, H-8), 6.1 (1H, d, J = 1.9 Hz, H-6). ^{13}C NMR (100 MHz, in ppm, DMSO-d_6): δ 176.2 (C-4), 164.2 (C-7), 161.0 (C-5), 156.5 (C-8a), 148.0 (C-4′), 147.1 (C-2), 145.4 (C-3′), 136.1 (C-3), 122.3 (C-1′), 120.3 (C-6′), 115.9 (C-5′), 115.4 (C-2′), 103.3 (C-4a), 98.5 (C-6), 93,7 (C-8). EI-MS m/z $[M]^{+\cdot}$ at 302 (100%).

2.3.2. Tamarixetin (*2*)

UV (λ_{max} in MeOH): gives bands at 371 and 254 nm for band I and II, addition of NaOH; 411, 322 and 274, $AlCl_3$; 428 and 264 while HCl; 427 and 263. ^{1}H NMR (300 MHz, in ppm, DMSO-d_6): δ 7.8 (1H, d, J = 1.54 Hz, H-2′), 7.8 (1H, dd, J = 7.5 Hz, J = 1.5 Hz, H-6′), 7.01 (1H, d, J = 7.5 Hz, H-5′), 6.54 (1H,d, J = 1.8 Hz, H-8), 6.25 (1H, d, J = 1.8 Hz, H-6), and 3.9 (3H, S, OMe). ^{13}C NMR (100 MHz, in ppm, DMSO-d_6): δ 175.8 (C-4), 163.8 (C-7), 160.6 (C-5), 156.1 (C-8a), 148.7 (C-4′), 146.6 (C-2), 146.6 (C-3′), 135.7 (C-3), 121.9 (C-1′), 121.6 (C-6′), 115.5 (C-5′), 111.5 (C-2′), 102.9 (C-4a), 98.2 (C-6), 93,6 (C-8). EI-MS m/z $[M]^{+\cdot}$ at 316 (100%).

2.3.3. Isorhamnetin-3-rutinoside (*3*)

UV (λ_{max} in MeOH): gives bands at 356 and 254 nm for band I and II, addition of NaOH; 412, 326 and 273, $AlCl_3$; 403 and 268, as like as HCl; 403 and 268. ^{1}H NMR (300 MHz, in ppm, DMSO-d_6): δ 7.86 (1H, d, J = 1.7 Hz, H-2′), 7.53 (1H, dd, J = 8.5 Hz, J = 1.7 Hz, H-6′), 6.5 (1H, d, J = 8.5 Hz, H-5′), 6.43 (1H,d, J = 1.7 Hz, H-8), 6.2 (1H, d, J = 1.7 Hz, H-6), 5.45 (1H, d, J = 7.2, H-1″), 4.41 (1H, H-1‴), 3.83 (3H, S, OMe) and 0.98 (3H, d, J = 6.0, H-6‴). ^{13}C NMR (100 MHz, in ppm, DMSO-d_6): δ 177.2 (C-4), 164.3 (C-7), 161.1 (C-5), 156.4 (C-8a), 149.3 (C-4′), 156.4 (C-2), 146.2 (C-3′), 132.9 (C-3), 120.9 (C-1′), 122.2 (C-6′), 115.2 (C-5′), 113.2

(C-2′), 103.9 (C-4a), 101.1 (C-1″), 100.8 (C-1‴), 98.7 (C-6), 93,8 (C-8), 76.3 (C-3″), 75.8 (C-5″), 74.2 (C-2″), 71.7 (C-4‴), 70.5 (C-3‴), 70.2 (C-2‴), 70.0 (C-4″), 68.2 (C-5‴), 66.8 (C-6″), 55.6 (C-OMe) and 17.6 (C-6‴). ES-MS m/z $[M+Na]^+$ at 647.

*2.3.4. Kaempferol-3-O-β-rutinoside (**4**)*

UV (λ_{max} in MeOH): gives bands at 350 and 266 nm for band I and II, addition of NaOH; 401, 325 and 274, NaOAc; 377, 305 and 274, H_3BO_3; 353 and 277, $AlCl_3$; 398 and 274, and HCl; 395 and 274. ^{1}H NMR (300 MHz, in ppm, DMSO-d_6): δ 7.97 (2H, d, J = 8.3 Hz, H-2′ and H-6′), 6.88 (2H, d, J = 8.3 Hz, H-3′ and H-5′), 6.27 (1H, d, H-8), 6.07 (1H,d, H-6), 5.26 (1H, d, J = 7.1, H-1″), 4.39 (1H, H-1‴), and 0.98 (3H, d, J = 6, H-6‴). ^{13}C NMR (100 MHz, in ppm, DMSO-d_6): δ 177.6 (C-4), 164.3 (C-7), 160.9 (C-5), 160.1 (C-4′), 156.7 (C-8a), 156.2 (C-2), 132.9 (C-3), 130.7 (C-2′ and C-6′), 120.7 (C-1′), 115.1 (C-3′ and C-5′), 105.1 (C-4a), 101.8 (C-1″), 100.8 (C-1‴), 97.7 (C-6), 94,2 (C-8), 76.3 (C-3″), 75.6 (C-5″), 74.1 (C-2″), 71.8 (C-2‴), 71.7 (C-4‴), 70.5 (C-3‴), 70.3 (C-4″), 68.2 (C-5‴), 66.8 (C-6″), and 17.7 (C-6‴). ES-MS m/z $[M+Na]^+$ at 617.

*2.3.5. 3,4-Dihydroxybenzoic acid (**5**)*

^{1}H NMR (300 MHz, in ppm, MeOD): δ 7.33 (1H, d, J = 1.8, H-2), 7.29 (1H, dd, J = 8.1 and 1.8, H-6) and 6.67 (1H, d, J = 8.1, H-5). ^{13}C NMR (100 MHz, in ppm, MeOD): δ: 173.7 (C-1′), 150.5 (C-4), 147.02 (C-3), 127.6 (C-1), 123.8 (C-6), 118.2 (C-2) and 115.8 (C-5). EI-MS m/z $[M]^{+\cdot}$ at 154.

3. Results and discussion

Compound (**1**), (**2**), (**3**), and (**4**) displayed UV absorption and ^{1}H, ^{13}C NMR data typical of flavonoids (Mabry et al., 1970). All compounds showed carbon signal at δ 176.15 (**1**), 175.8 (**2**), 177.2 (**3**) and 177.6 (**4**). This indicated that there are flavones with a hydroxyl group at C-5. The UV spectral data of both flavones with diagnostic shifts reagents indicated flavonols with free 5 and 7 hydroxyl groups.

3.1. Compound *1*

The molecular formula of (**1**) was determined to be $C_{15}H_{10}O_7$ on the basis of the ion IE-MS (m/z 302 $[M^+]$. The MS-fragmentation ion at m/z 153 $[A_1+H]^+$ and at m/z 137 $[B_2]$.

The ^{1}H NMR spectrum of (**1**) displayed signals for two meta-coupled protons at δ 6.27 (1H, d, J = 1.9 Hz, H-8) and 6.04 (1H, d, J = 1.9 Hz, H-6). The multiplicities and the weak coupling constants of H-6 and H-8 were in agreement with the existence of the hydroxyl group at C-7 (δ 164.2). The chemical shifts at δ 7.53, 7.38 and 6.75 were assigned to protons in the B ring and the presence of the pick at m/z 137 $[B_2]$ was in accordance with a B ring with two hydroxyl groups at 4′ and 3′ position. Consequently, the structure of flavonoid (**1**) was established as known flavonol 3,5,7,3′,4′-pentahydroxy flavones or quercitin (Bilia et al., 1996).

3.2. Compound *2*

The molecular formula was established as $C_{16}H_{12}O_7$ on the basis of positive ion EI-MS (m/z 316 $[M^+]$). The MS-fragmentation ion at m/z = 153 as well as the absence of m/z = 137 [if a-di-OH ring B] were in accordance with a B ring with one hydroxyl and one methoxyl group (Harborne et al., 1975).

The UV spectrum exhibited absorption maxima at 371 and 254 nm that are characteristic absorption bands of a flavones skeleton (Mabry et al., 1970). The UV spectral data showed the presence of a free 7-OH group and there are no ortho hydroxyl groups on the ring A and B.

The ^{1}H NMR spectrum of (**2**) displayed signals for two meta-coupled protons at δ 6.54 (1H, d, J = 1.8 Hz, H-8) and δ 6.25 (1H, d, J = 1.8 Hz, H-6). The multiplicities and the weak coupling constants of H-6 and H-8 were in accordance with the existence of the hydroxyl group at C-7 (δ 163.8). The ^{1}H NMR also demonstrated two one proton doublets at δ 7.81 (1H, d, J = 1.5 Hz), and 7.01 (1H, d, J = 7.5 Hz,) and one double doublet 7.76 (1H, dd, J = 7.5, 1.5 Hz), assignable to H-2′, H-5′ and H-6′ protons, respectively. The appearance of two doublets and their coupling constant values are further in agreement with the hydroxy group and one methoxyl group at C-3′ and C-4′. The presence of a methoxyl group was shown by a singlet representing three protons at δ 3.9 ppm. The HMBC spectrum of compound (**2**) suggested that this methoxyl group was attached to the 4′-position. On the basis of the UV shifts, and ^{1}H ^{13}C NMR and by comparison with those reported in the literature, the compound is identified as: 3,5,7,3′-tetrahydroxy-4′-methoxyflavone or tamarixetin (Barrero et al., 1998).

3.3. Compound *3*

Compound (**3**), exhibited a molecular ion peak at m/z 647 $[M+Na]^+$ in its electrospray mass spectrum corresponded to the molecular formula $C_{28}H_{32}O_{16}$. The UV spectrum in MeOH gave maxima at 254 and 356 nm, respectively, indicating that the compound belongs to the flavone groups. No shift in band I of compound (**3**) was observed after the addition of $AlCl_3$ and $AlCl_3$/HCl. These UV data indicated an absence of an orthodihydroxyl pattern in B ring (Markham, 1982). The UV spectral data showed also the presence of a free 7-OH group. The ^{1}H NMR spectrum of the compound exhibited signal at δ 12.57 (1H, s) attributed to a chelated hydroxyl group. The presence of a methoxyl group was shown by a singlet representing three protons at δ 3.83 ppm. The assignment of C-3′ is confirmed by the correlation between the singlet at δ 3.83 ppm (3H, methoxyl) and this carbon in the HMBC experiment which indicates C-3′ methoxylated flavonoid. The ^{1}H NMR also demonstrated two one proton doublets at δ 7.86 (1H, J = 1.7 Hz) and δ 6.5 (1H, J = 8.5 Hz) and one double doublet 7.53 (1H, J = 1.7, 8.5 Hz) assignable to H-2′, H-5′ and H-6′ protons, respectively. The appearance of two doublets and their coupling constant values are further in agreement with the hydroxyl and the methoxyl groups at C-3′ and C-4′. The ^{1}H NMR spectrum of (**3**) displayed signals for two meta-coupled protons at δ 6.2 (1H, d, J = 1.7 Hz, H-6) and δ 6.43 (1H, d, J = 1.7 Hz, H-8).

The resonances of the signals observed in the low-field region in the ^{1}H NMR spectra at δ 5.45 (1H, d, J = 7.2 Hz) and at δ 4.41 (1H, br s) were attributed to the anomeric protons of glucose and rhamnose, respectively. This was confirmed by the ^{13}C NMR spectrum which exhibited most of signals of saccharide between 65 and 78 ppm and the anomeric at 100.8 and 101 ppm. The β-anomeric configuration for the

glucose was judged from its large coupling constants (J = 7.2 Hz) (Agrawal, 1992). The appearance of the doublet at δ 0.98 (3H, d, J = 6.0 Hz) was assignable to H-6 of rhamnose. The rhamnose was attached to the hydroxyl group at the C-6 position of glucose as judged from the downfield shift (6.6 ppm) of the C-6 signal (Markham et al., 1978). The HMBC spectrum of compound (**3**) suggested that the saccharide moiety was attached to the 3-position. The NMR data of (**3**) were identical with those of 3-*O*-methylquercitin 3-*O*-α-L-rhamnopyranosyl-(1→6)-β-D-glucopyranoside (Sang et al., 2002; Senatore et al., 2000), which have been confirmed by 2D NMR. Thus, the structure 3-*O*-methylquercitin 3-*O*-α-L-rhamnopyranosyl-(1→6)-β-D-glucopyranoside was assigned to (**3**), originally isolated from the flowers of *Narcissus tazetta* L. (Kubota and Hase, 1956) and from the pollen of *Lilium auratum*. (Kotake and Arakawa, 1956).

3.4. Compound 4

The spectral data of **4** showed that it possessed the same saccharide structure as that of **3** but differed from the aglycone part. The aglycone of **4** is the very common flavonol kaempferol. Therefore, the structure kaempferol-3-*O*-α-L-rhamnopyranosyl-(1→6)-β-D-glucopyranoside was assigned to **4** (Sang et al., 2002).

3.5. Compound 5

The ^{1}H NMR spectrum demonstrated two one proton doublets at δ 6.67 (1H, J = 8.1 Hz) and δ 7.33 (1H, J = 1.8 Hz) and one double doublet at 7.29 (1H, J = 1.8, 8.1 Hz). The appearance of two doublets and their coupling constant values are further in agreement with the presence of a trisubstituted aromatic nucleus in position 1, 3 and 4. The ^{13}C NMR exhibited 7 carbon signals. The DEPT 135 spectrum showed that it possessed 4 quaternary carbons. The compound exhibited a molecular ion peak at m/z 154 $[M]^+$ in its electrospray mass spectrum corresponded to the molecular formula $C_7H_6O_{11}$. The MS-fragmentation ion at m/z 137 $[M-OH]^+$, and at m/z 110 $[M-COOH]^+$.

The ^{13}C NMR spectrum showed a downfield signal at δ 173.6 clearly assigned to carbonyl carbon. The HMBC spectrum of compound (**5**) suggested that this carbonyl was attached to the 1-position of the aromatic nucleolus. Therefore, the structure of (**5**) was established to be 3,4-dihydroxybenzoic acid.

4. Conclusion

The genus *Bupleurum* is known for the presence of variety of compounds. Previous investigations led to the isolation of saponosids (Ebata et al., 1996; Tan et al., 1999; Zhao et al., 1996), coumarins (Pistelli et al., 1996; Banerji et al., 1977; Nakabayashi et al., 1964; Gonzalez et al., 1975), isoflavones (Tan et al., 1998) and flavonoids (Zhang et al., 2007). The present study reported for the first time quercitin (1), tamarixetin (**2**), isorhamnetin-3-rutinoside (**3**), kaempferol-3-*O*-β-rutinoside (**4**) and 3,4-dihydroxybenzoic acid (**5**) in the aerial parts of *B. montanum*. The presence of flavonoids in the genus is in accordance with the placement of the genus in the Apiaceae family that includes several genera, Flavum, Gibraltaricum, Polyclonum, Sibiricum and Chinense known for their ability to produce flavonoids (Zhang et al., 2007; Song et al., 1992; Luo and Jin, 1991; Barrero et al., 1998; Gevrenova et al., 1997, and Crowden et al., 1969).

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