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## **New Flavonol Glycoside from the Leaves of *Ventilago africana***

Diane Patricia Apie Gossan, Abdulmagid Alabdul Magid, Philomène Akoua Yao-Kouassi, Damien Le Faucheur, Antoine Ahibo Coffy, Dominique Harakat, Laurence Voutquenne-Nazabadioko

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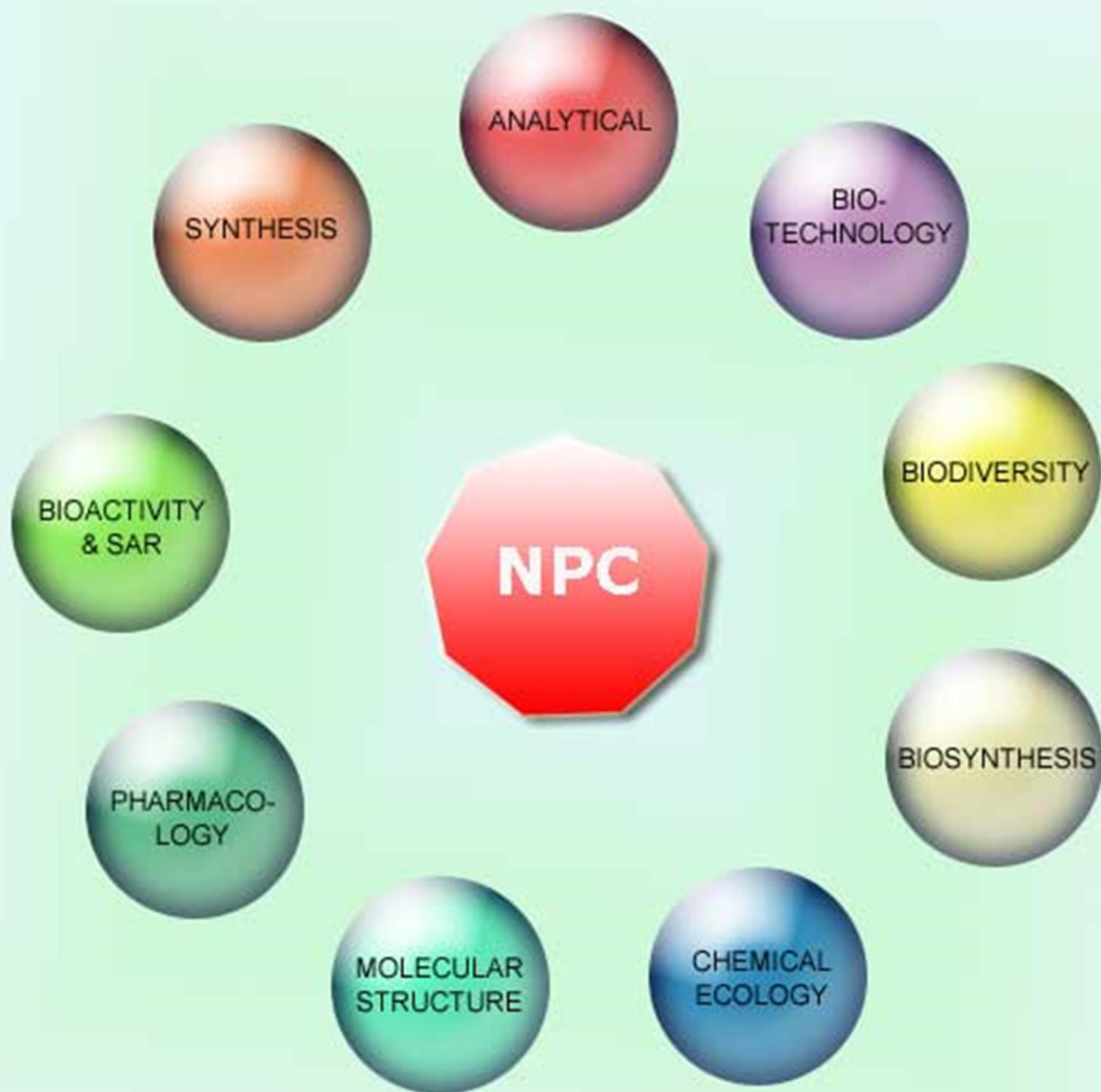
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New Flavonol Glycoside from the Leaves of *Ventilago africana*Diane Patricia Apie Gossan<sup>a,b</sup>, Abdulmagid Alabdul Magid<sup>\*a</sup>, Philomène Akoua Yao-Kouassi<sup>b</sup>, Damien Le Faucheur<sup>a</sup>, Antoine Ahibo Coffy<sup>b</sup>, Dominique Harakat<sup>c</sup> and Laurence Voutquenne-Nazabadioko<sup>a</sup><sup>a</sup>ICMR-UMR CNRS 7312, Groupe Isolement et Structure, Campus Sciences, Bat. 18, BP 1039, 51687 Reims Cedex 2, France<sup>b</sup>Laboratoire de Chimie Organique Biologique, 01 BPV 34 Abidjan 01, Université Félix-Houphouët Boigny d'Abidjan-Cocody, Côte d'Ivoire<sup>c</sup>ICMR-UMR CNRS 7312, Service Commun d'Analyses, Campus Sciences, Bat. 18, BP 1039, 51687 Reims Cedex 2, France

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A new flavonol diglycoside, rhamnocitrin-3-*O*- $\alpha$ -L-rhamnopyranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-galactopyranoside, was isolated from the leaves of *Ventilago Africana*, in addition to 11 known flavonoids. Their structures were determined by spectroscopic methods including 1D- and 2D-NMR, and HR-ESI-MS analysis. The isolated compounds were evaluated for their antioxidant activity by using DPPH radical-scavenging assay. Compounds 4, 7-9 have discrete to good antioxidant potential with EC<sub>50</sub> values ranging from 20.9 to 40.4  $\mu$ M, compared with ascorbic acid (EC<sub>50</sub> 60  $\mu$ M) used as positive control.

**Keywords:** *Ventilago africana*, Rhamnaceae, Flavonoids; DPPH free radical scavenging activity.

*Ventilago africana* Exell is a liane with a thick, soft, woody stem belonging to the family Rhamnaceae [1]. It is distributed in evergreen forests, especially marshy sites and mangroves, from Guinea-Bissau to Nigeria, and across the Congo basin to Uganda and Angola [2]. *V. africana* has been used in traditional medicine for the treatment of *dysmenorrhoea* and as a febrifuge [2]. Previous chemical studies of the genus *Ventilago* have shown the presence of a variety of anthraquinones, naphthoquinones, quinones, and benzisochromanquinones [3]. In the course of our search for interesting bioactive substances in nature, the 80% aqueous methanol extract of the leaves of *V. africana* was investigated. As a result, a new flavonol diglycoside was identified, together with eleven known flavonoids. Free radical scavenging activities of the isolated compounds are also described in this paper.

The *n*-BuOH-soluble fraction of the 80% MeOH extract of the leaves of *V. africana* was separated by a combination of chromatographic methods to obtain a new flavonol diglycoside **1**, in addition to eleven known compounds (**2-12**) (Figure 1). Compounds **2-12** were elucidated as kaempferol-3-*O*- $\alpha$ -L-rhamnopyranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-glucopyranoside (**2**) [4], kaempferol-3-*O*-(2,6-di-*O*- $\alpha$ -L-rhamnopyranosyl- $\beta$ -D-glucopyranoside (**3**) [5], kaempferol-3-*O*- $\alpha$ -L-rhamnopyranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-galactopyranoside (**4**) [6], rhamnocitrin-3-*O*- $\alpha$ -L-rhamnopyranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-glucopyranoside (**5**) [6], rhamnocitrin-3-*O*-(2,6-di-*O*- $\alpha$ -L-rhamnopyranosyl- $\beta$ -D-glucopyranoside (**6**) [7], quercetin (**7**) [8], quercetin-3-*O*- $\alpha$ -L-rhamnopyranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-glucopyranoside (**8**) [9], quercetin-3-*O*-(2,6-di-*O*- $\alpha$ -L-rhamnopyranosyl- $\beta$ -D-glucopyranoside (**9**) [9], rhamnetin-3-*O*- $\alpha$ -L-rhamnopyranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-glucopyranoside (**10**) [10], isorhamnetin 3-*O*- $\alpha$ -L-rhamnopyranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-glucopyranoside (**11**) [10], and rhamnazin-3-*O*- $\alpha$ -L-rhamnopyranosyl-(1 $\rightarrow$ 6)- $\beta$ -D-glucopyranoside (**12**) [11]. To the best of our knowledge, this is the first report of flavonoids in the genus *Ventilago*.

Compound **1** was isolated as an amorphous, yellow powder, and its molecular formula was determined to be C<sub>28</sub>H<sub>32</sub>O<sub>15</sub> by HR-ESI-MS

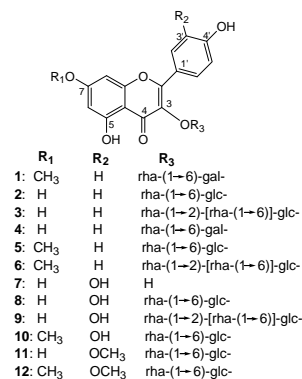


Figure 1: Chemical structure of compounds **1-12**, isolated from *V. africana*.

from the ion at  $m/z$  631.1636 [M + Na]<sup>+</sup> (C<sub>28</sub>H<sub>32</sub>O<sub>15</sub>Na, calcd 631.1639). The UV spectrum revealed absorption bands at 266 and 346 nm, suggesting a flavonol skeleton. The <sup>1</sup>H and <sup>13</sup>C NMR spectra of **1** comprised resonances corresponding to aromatic and glycosidic protons and carbons, and an OCH<sub>3</sub> ( $\delta_C$  56.5;  $\delta_H$  3.92). Four aromatic doublets corresponded to H-6 [ $\delta_H$  6.38 (1H,  $J$  = 1.1 Hz)], *meta*-coupled with H-8 [ $\delta_H$  6.65 (1H,  $J$  = 1.1 Hz)] on the A ring, and to H-2', H-6' [ $\delta_H$  8.14 (2H,  $J$  = 8.0 Hz)], *ortho*-coupled with H-3',H-5' [ $\delta_H$  6.92 (2H,  $J$  = 8.0 Hz)] on the B ring. Full identification of the aglycone was finally achieved by 2D-NMR spectroscopy. The HMBC correlation between  $\delta_H$  3.92 (OCH<sub>3</sub>) and  $\delta_C$  167.5 (C-7) placed the OCH<sub>3</sub> at C-7 of the aglycone, which led to the rhamnocitrin structure (7-methoxy 3,5,4'-trihydroxy-flavone) [7]. Furthermore, two anomeric proton resonances corresponding to *O*-linked sugars were displayed in the <sup>1</sup>H NMR spectrum as two doublets at  $\delta_{H-1''}$  5.10 (d,  $J$  = 7.8 Hz) and  $\delta_{H-1''}$  4.54,  $J$  = 1.2 Hz. Based on the results of acid hydrolysis of the flavonoids mixture, the magnitudes of their  $J_{1,2}$  coupling constants and the analysis of 1D and 2D-NMR data, the sugar units were elucidated as  $\beta$ -D-galactopyranose ( $\delta_{H-1''}$  5.10;  $\delta_{C-1''}$  105.3) and  $\alpha$ -L-rhamnopyranose ( $\delta_{H-1''}$  4.54;  $\delta_{C-1''}$  101.9) [12]. The  $\beta$ -D-gal was characterized by the

large coupling constants  $J_{H-1,H-2}$  and  $J_{H-2,H-3}$  ( $> 7.8$  Hz) and the small coupling constant  $J_{H-3,H-4}$  (3.3 Hz), whereas the  $\alpha$ -L-rha was characterized by the small coupling constants  $J_{H-1,H-2}$  (1.2 Hz) and its methyl group at  $\delta_{H-6}$  1.20 (d,  $J = 6.0$  Hz) and  $\delta_{C-6}$  18.0. The glycosidic linkage was established by the HMBC correlations between  $\delta_{H-1''}$  5.10 and  $\delta_C$  135.8 (C-3) and between  $\delta_{H-1''}$  4.54 and  $\delta_{C-6''}$  67.5, indicating that the galactose unit was located at C-3 of the aglycone and the rhamnose unit was attached to C-6 of galactose. Thus, **1** was characterized as rhamnocitrin-3-*O*- $\alpha$ -L-rhamnopyranosyl-(1 $\rightarrow$ 6)]- $\beta$ -D-galactopyranoside.

In order to assess the antioxidative properties of **1-12**, the radical scavenging activity of these compounds on DPPH was measured. According to the results, summarized in Table 1, compounds **4**, **7**, **8**, and **9** exhibited a good antioxidant potential ( $EC_{50}$  values ranging from 20.9 to 40.4  $\mu$ M) compared with ascorbic acid, which was used as a positive control ( $EC_{50}$  60  $\mu$ M) (Table 1).

Compounds **7-10** were found to be active, which indicated that the di-OH substitution at 3' and 4' of the B-ring is particularly important to the antiradical activity of a flavonoid. Compounds **2** and **4** shared a common aglycone mono-OH substituted in the B-ring. The only difference was the nature of the disaccharide chain linked at C-3, rha-(1 $\rightarrow$ 6)-glc- (**2**) and rha-(1 $\rightarrow$ 6)-gal- (**4**), suggesting that the galactose might contribute to the antioxidant activity.

**Table 1:** DPPH radical scavenging activity of compounds **1-12**.

Comps	$EC_{50} \pm S.D.$ ( $\mu$ M)	Comps	$EC_{50} \pm S.D.$ ( $\mu$ M)
<b>1</b>	- <sup>a</sup>	<b>8</b>	40.2 $\pm$ 0.7
<b>2</b>	- <sup>a</sup>	<b>9</b>	36.5 $\pm$ 0.6
<b>3</b>	- <sup>a</sup>	<b>10</b>	65.5 $\pm$ 1.9
<b>4</b>	40.4 $\pm$ 0.9	<b>11</b>	- <sup>a</sup>
<b>5</b>	- <sup>a</sup>	<b>12</b>	- <sup>a</sup>
<b>6</b>	- <sup>a</sup>	Ascorbic acid <sup>b</sup>	60.2 $\pm$ 1.2
<b>7</b>	20.9 $\pm$ 0.5		

<sup>a</sup> 50% inhibition not achieved at the concentration of 200  $\mu$ g/mL. <sup>b</sup> Used as a positive control.

Comparison of the antioxidant activity of compounds **1** with **4** and **8** with **10** indicated that the methoxy group at C-7 in **1** or **10** decreased activity. The free 3'-OH was also found to be important for antioxidant activity by comparison of the antioxidant activity of **8** (3'-OH) with **11** (3'-OCH<sub>3</sub>). The same result was found by comparison of activity of **10** and **12**. Thus, the hindrance effect (methoxy groups at C-7 and/or C-3'), the nature of the saccharide chain linked at C-3, and the B ring OH substitution (4'-OH or 3',4'-di-OH) in the flavonoid structures are determinant in the scavenging of free radicals.

## Experimental

**General:** NMR, Bruker Avance DRX III 500; HR-ESI-MS, Micromass Q-TOF micro instrument; Optical rotations, Perkin-Elmer 341 polarimeter; UV, Shimadzu UV-2450 spectrophotometer in MeOH; TLC, pre-coated silica-gel 60 F<sub>254</sub>; CC, Kieselgel 60 (63-200 mesh) or LiChroprep RP-18 (40-63  $\mu$ m); Flash chromatography, Grace Reveleris system with dual UV and ELSD detection equipped with a 12g RP-C<sub>18</sub> column. The mobile phase was water and methanol with a flow rate of 30 mL/min and the effluents were monitored at 205, 225, 250, and 360 nm. HPLC was performed on a Dionex apparatus equipped with an ASI-100 autosampler, an Ultimate 3000 pump, a diode array detector UVD 340S and a Chromeleon software. RP-C<sub>18</sub> column (Phenomenex 250x15 mm, Luna 5 $\mu$ ) was used for semi preparative HPLC with a binary gradient eluent (H<sub>2</sub>O (pH 2.4 with TFA); MeCN) and a flow rate of 6 mL/min; the chromatogram was monitored at 205, 225, 250, and 350 nm. Absorbance values in the DPPH free radical

scavenging assay were read on a Fluostar omega microplate reader (BMG labtech).

**Plant material:** The leaves of *V. africana* were collected in Adiopodoumé, Ivory Coast, in January 2011. A voucher specimen (Aké-Assi-21254) has been deposited in the herbarium of the National Center of Floristics of FHB University of Cocody (Ivory Coast).

**Extraction and isolation:** The powdered dry leaves of *V. africana* (800 g) were percolated with 14 L of 80% MeOH. After methanol evaporation and concentration to 2 L, the aqueous residue was successively partitioned with light petroleum, CH<sub>2</sub>Cl<sub>2</sub>, and *n*-BuOH. The *n*-BuOH extract (43.8 g) was subjected to Diaion HP20 macroporous adsorbent resin CC using a MeOH-H<sub>2</sub>O gradient (25, 50, 75, and 100% MeOH) to yield fractions I-IV, successively. Fraction II (9.7 g) was subjected to RP-C<sub>18</sub> CC with a gradient system of MeOH-H<sub>2</sub>O (25-45% MeOH) to afford 12 fractions (B<sub>1</sub>-B<sub>12</sub>). Further purification of B<sub>4</sub> (0.42 g) (eluted with MeOH-H<sub>2</sub>O, 3:7) by RP-C<sub>18</sub> chromatography using a MeOH-H<sub>2</sub>O gradient (6:4-7:3) and then by semi-prep HPLC (14% MeCN) afforded compounds **9** (*rt* 10.9 min, 9 mg), **8** (*rt* 11.9 min, 10 mg), and **3** (*rt* 14.0 min, 19 mg). Further purification of B<sub>7</sub> (0.61 g) (eluted with MeOH-H<sub>2</sub>O, 35:65) by RP-C<sub>18</sub> chromatography using a MeOH-H<sub>2</sub>O gradient (5:5-6:4) and then by semi-prep HPLC using a binary gradient (16-20% MeCN in 30 min) yielded compounds **4** (*rt* 16.5 min, 5 mg), **2** (*rt* 17.5 min, 26 mg), and **11** (*rt* 20.4 min, 4 mg). Fraction III (12 g) was subjected to silica gel VLC eluted successively with CHCl<sub>3</sub>-MeOH-H<sub>2</sub>O (95:5:0, 9:1:0, 85:15:0, 8:2:0, 75:25:0, 7:3:0.5) to yield fractions C<sub>1</sub>-C<sub>6</sub>. Fraction C<sub>2</sub> (0.28 g) was further separated by RP-C<sub>18</sub> CC eluted with a gradient of MeOH:H<sub>2</sub>O (4:6-8:2) to afford 16 mg of **13** and 33 mg of **7**. Fraction C<sub>4</sub> (0.84 g) was further purified by RP-C<sub>18</sub> flash chromatography using a binary gradient (30-80% MeOH in 60 min) and then by semi-prep HPLC (27-32% MeCN in 50 min) to afford compound **5** (*rt* 12.5 min, 11 mg). Fraction C<sub>6</sub> (1.1 g) was further purified by RP-C<sub>18</sub> VLC and then by semi-prep HPLC (24-26% MeCN in 50 min) to yield compounds **6** (*rt* 11.1 min, 12 mg) and **12** (*rt* 13.3 min, 8 mg) or isocratic elution with 14% MeCN to give compounds **10** (*rt* 13.8 min, 10 mg) and **1** (*rt* 21.3 min, 11 mg).

## Rhamnocitrin-3-*O*- $\alpha$ -L-rhamnopyranosyl-(1 $\rightarrow$ 6)]- $\beta$ -D-galactopyranoside (**1**)

$[\alpha]_D$ : -27.2 (*c* 0.13, MeOH).

UV/Vis  $\lambda_{max}$  (MeOH) nm (Abs.): 266 (0.58), 348 (0.50).

<sup>1</sup>H NMR (500 MHz, MeOH-*d*<sub>4</sub>): 1.20 (1H, d,  $J = 6.0$  Hz, H-6''), 3.30 (1H, t,  $J = 9.4$  Hz, H-4''), 3.42 (1H, dd,  $J = 10.3$ -6.1 Hz, H-6''), 3.51 (1H, dd,  $J = 9.4$ -3.2 Hz, H-3''), 3.54 (1H, m, H-5''), 3.56 (1H, dd,  $J = 9.0$ -3.3 Hz, H-3''), 3.60 (1H, dd,  $J = 3.2$ -1.2 Hz, H-2''), 3.64 (1H, brdt,  $J = 6.1$  Hz, H-5''), 3.74 (1H, dd,  $J = 10.3$ -5.5 Hz, H-6''), 3.79 (1H, d,  $J = 3.2$  Hz, H-4''), 3.82 (1H, dd,  $J = 9.0$ -7.8 Hz, H-2''), 3.92 (3H, s, 7-OMe), 4.54 (1H, d,  $J = 1.2$  Hz, H-1''), 5.10 (1H, d,  $J = 7.8$  Hz, H-1''), 6.38 (1H, s, H-6), 6.65 (1H, s, H-8), 6.92 (2H, d,  $J = 8.0$  Hz, H-3',5'), 8.14 (2H, d,  $J = 8.0$  Hz, H-2',6').

<sup>13</sup>C NMR (125 MHz MeOH-*d*<sub>4</sub>): 18.0 (CH<sub>3</sub>, C-6''), 56.5 (7-OCH<sub>3</sub>), 67.5 (CH<sub>2</sub>, C-6''), 69.7 (CH, C-5''), 70.2 (CH, C-4''), 72.1 (CH, C-2''), 72.3 (CH, C-3''), 73.0 (CH, C-2''), 73.9 (CH, C-4''), 75.0 (CH, C-3''), 75.4 (CH, C-5''), 93.3 (CH, C-8), 99.2 (CH, C-6), 101.9 (CH, C-1''), 105.3 (CH, C-1''), 106.5 (C, C-10), 116.2 (CH, C-3',5'), 122.6 (C, C-1'), 132.6 (CH, C-2',6'), 135.8 (C, C-3), 158.5 (C, C-9), 159.7 (C, C-2), 162.0 (C, C-4'), 163.8 (C, C-5), 167.5 (C, C-7), 179.8 (C, C-4).

HR-ESI-MS: *m/z* [M + Na<sup>+</sup>] calcd for C<sub>28</sub>H<sub>32</sub>O<sub>15</sub>Na: 631.1639; found: 631.1636.

**Acid hydrolysis:** Acid hydrolysis of the *n*-BuOH soluble fraction was realized as previously described [13]. Briefly, 1 g was refluxed with 50 mL of 2M HCl for 4 h. After extraction with ethyl acetate (3 x 30 mL), the aqueous layer was evaporated to furnish the monosaccharide residue (300 mg). Three sugars were identified as D-glucose, D-galactose and L-rhamnose by comparison with authentic samples on TLC and by measurement of the optical rotation of each purified sugar.

**DPPH free radical scavenging assay:** The scavenging activity of isolated compounds against DPPH was investigated by spectrophotometric methodology, as previously described [13]. Briefly, 5  $\mu$ L of either the standard or sample solutions (dissolved in DMSO) was mixed with 95  $\mu$ L of DPPH solution (158  $\mu$ M, dissolved in absolute EtOH). After mixing gently and incubating for

30 min at 37°C, the optical density was measured at  $\lambda$  515 nm. The percentage of absorbance inhibition at  $\lambda$  515 nm was calculated using the following equation: % inhibition  $[(Ab_{\text{control}} - Ab_{\text{sample}})/Ab_{\text{control}}] \times 100$ . DPPH solution in EtOH was used as a control. The curve of the % scavenging activity against the concentration of sample was prepared by the MSeExcel based program to obtain the EC<sub>50</sub>. All the tests were conducted in triplicate. The experimental data were expressed as mean  $\pm$  standard deviation.

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