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CONTRIBUTION TO THE STUDY ON POLAR CONSTITUENTS FROM THE BUDS OF *RHODOMYRTUS TOMENTOSA* (AIT.) HASSK. (MYRTACEAE)

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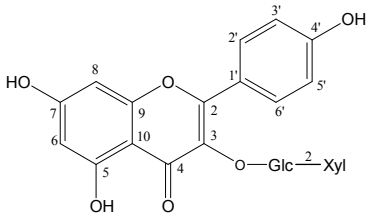
SUMMARY

*A flavonol glycoside, kaempferol 3-O-β-sambubioside (**1**) was isolated in a study on the polar constituents of the buds of Rhodomyrtus tomentosa Ait. Hassk. (Myrtaceae). Its structure was determined by spectroscopic methods.*

Keywords: *Rhodomyrtus tomentosa*; *Myrtaceae*; flavonol glycoside.

Rhodomyrtus tomentosa Ait. Hassk. (Myrtaceae) [1] (Vietnamese name: Sim) is a small shrub, 1-2 m high. The plant occurs rather widely in Asian tropical and subtropical regions. In Vietnam, *R. tomentosa* is extensively found in all the midland and low mountainous regions, sometimes in higher mountains. Recently, it has been grown in medicinal plant gardens at hospitals and village health-care stations for convenience of use. The young buds and leaves are collected in spring, they are then sun-dried. The buds and young leaves of *R. tomentosa* are effective for treatment of abdominal pain, diarrhoea, and dysentery. They are used in the form of oral decoction. In external use, the decoction of the leaves is used for lavage of wound [1].

Triterpenoids, steroids [2, 3], malvidin-3-glucoside [4], tannins [5, 6], and flavone glycosides [7] were isolated from the leaves, stems, and flowers of *R. tomentosa*. Our study is the first report dealing with the buds of the plant. We investigated the *n*-BuOH-soluble fraction of the MeOH extract from the buds of *R. tomentosa* [8] and isolated a flavonol glycoside [9]. The ^{1}H- and ^{13}C-NMR spectra [10] showed the presence of a kaempferol glycoside [11]. The glycoside moiety was determined to be 3-*O*-β-D-xylopyranosyl-(1→2)-*O*-β-D-glucopyranoside (sambubioside) on the basis of comparison of the ^{1}H- and ^{13}C-NMR spectroscopic data of **1** with those reported in the literature [12]. The downfield ^{13}C shift at C-2 and upfield ^{13}C shift at C-3 when compared with those of kaempferol were reliable for the placement of the sambubioside moiety at C-3. Therefore the structure of **1** was determined to be kaempferol 3-*O*-β-D-xylopyranosyl-(1→2)-*O*-β-D-glucopyranoside (kaempferol 3-*O*-β-sambubioside), which was isolated from *Aesculus hippocastanum* L. (Hippocastanaceae) [13] and *Astragalus camplanatus* R. Br. (Leguminosae) [14]. It is worthwhile to mention the complexity in the isolation of the other polar minor constituents in the *n*-BuOH-soluble fraction. The analysis of the fractions obtained after column separation by TLC and HPLC showed the isolation and purification of the others remain an arduous task with the conditions used in this study.



1

*Kaempferol 3-O-β-sambubioside (**1**):*

Yellow amorphous powder. $[\alpha]^{25}_{D}$ -69.5 (c = 3.36, MeOH).

^{1}H-NMR (DMSO-d_6): δ 4.58 (1H, d, *J* = 7.2 Hz, Xyl-1), 5.69 (1H, d, *J* = 7.6 Hz, Glc-1), 6.17 (1H, d, *J* = 2.0 Hz, H-8), 6.41 (1H, d, *J* = 2.0 Hz, H-6), 6.88 (2H, d, *J* = 8.8 Hz, H-3′, H-5′), 8.06 (2H, d, *J* = 8.8 Hz, H-2′, H-6′). ^{13}C-NMR (DMSO-d_6): δ 60.5 (Glc-6), 65.7 (Xyl-5), 69.4 (Xyl-4), 69.6 (Glc-4), 73.8 (Xyl-2), 76.1 (Glc-5), 76.8 (Xyl-3), 77.5 (Glc-3), 81.7 (Glc-2), 93.5 (C-8), 97.9 (Glc-1), 98.6 (C-6), 103.9 (C-10), 104.4 (Xyl-1), 115.1 (C-3′, C-5′), 120.9 (C-1′), 130.8 (C-2′, C-6′), 132.8 (C-3), 155.2 (C-9), 156.3 (C-2), 159.9 (C-4′), 161.2 (C-5), 164.2 (C-7), 177.4 (C-4).

^{1}H-NMR (CD_3OD): δ 4.77 (1H, overlapped with HOD signal, Xyl-1), 5.47 (1H, d, *J* = 7.3 Hz, Glc-1), 6.19 (1H, d, *J* = 2.0 Hz, H-8), 6.38 (1H, d, *J* = 2.0 Hz, H-6), 6.89 (2H, d, *J* = 8.8 Hz, H-3′, H-5′), 8.05 (2H, d, *J* = 8.8 Hz, H-2′, H-6′). ^{13}C-NMR (CD_3OD): δ 62.8 (Glc-6), 67.0 (Xyl-5), 71.4 (Xyl-4), 71.5 (Glc-4), 75.2 (Xyl-2), 77.4 (Glc-5), 78.5 (Xyl-3), 78.6 (Glc-3), 82.7 (Glc-2), 95.1 (C-8), 100.2 (Glc-1), 101.2 (C-6), 105.7 (C-10), 106.2 (Xyl-1), 116.6 (C-3′, C-5′), 123.3 (C-1′), 132.6 (C-2′, C-6′), 135.4 (C-3), 158.8 (C-9), 158.9 (C-2), 161.8 (C-4′), 163.5 (C-5), 166.2 (C-7), 179.9 (C-4).

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8. The flower buds of *R. tomentosa* were collected in June 2004 in province Thai Nguyen and identified by Dr Tran Ngoc Ninh, Institute of Ecology and Biological Resources, Vietnamese Academy of Science and Technology, Hanoi, Vietnam.
9. The dried flower buds of *R. tomentosa* was extracted with MeOH at room temperature. The MeOH extract (100 g) was fractionated successively between H_2O and *n*-hexane, CH_2Cl_2, EtOAc, and *n*-BuOH. The *n*-BuOH-soluble fraction (11.0 g) was subjected to column chromatography (CC) on Diaion to give five fractions on eluting with H_2O, 20%, 40%, 60% MeOH in H_2O, and MeOH. The fifth fraction was separated by silica gel CC, eluted with $CHCl_3$-MeOH-H_2O (9:1:0, 7:3:0, and 15:6:1), and preparative octadecyl silica gel HPLC (17% MeCN in H_2O and 50% MeOH in H_2O) to afford compound **1** (33.6 mg).
10. ^{1}H-NMR (400 MHz) and ^{13}C-NMR (100 MHz) spectra were obtained on a JEOL JNM-α 400 NMR spectrometer.
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