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STILBENOID COMPOUNDS FROM RHIZOMES OF *SMILAX MYOSOTIFLORA*

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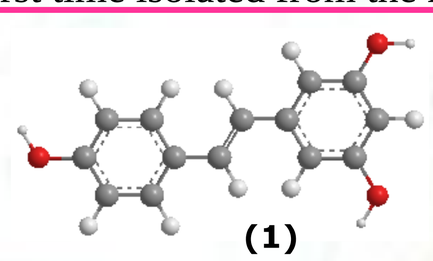
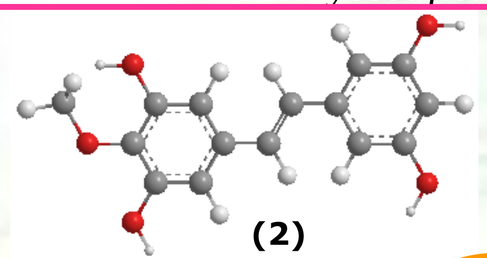
ABSTRACT

Two stilbenoid compounds, trans-3,4',5-trihydroxystilbene **(1)**, and trans-3,3',5,5'-tetrahydroxy-4'-methoxystilbene **(2)** were isolated from ethyl acetate extract of the rhizomes of *Smilax myosotiflora* by using silica gel and sephadex LH-20 column chromatography. Their structures were characterized on the basis of MS, 1D and 2D NMR spectroscopic analysis and confirmed by spectral data available in the literatures. These compounds were the first time isolated from the rhizomes of *Smilax myosotiflora*.





INTRODUCTION

The genus Smilax belongs to family (Smilacaceae) is a climbing shrub, which contains 350 species,which are native to tropical and warm parts around the world, and especially in East Asia , South and North America. Many of them have been used as medicinal herbs and known to be rich in steroid saponins. *Smilax myosotiflora* is a climbing plant usually grow in low altitude and foothills of Peninsular (Malaysia), Jawa (Indonesia) and Southern (Thailand). It is commonly known as "Ubi Jaga" in Malaysia, people have been used its leaves and fruit for treating, rheumatism and syphilis. Externally, *Smilax myosotiflora* is used as skin ailments such as wounds, inflammations, boils and ulcers. Moreover, its rhizomes are used as a sexual tonic to improve the male libido. Recently, pharmacological studies showed that smilax species have anti-bacterial antioxidant, anti-tumor anti-inflammatory, cytotoxic activities and etc….

OBJECTIVES:

To study the chemical constituents and biological activities of the rhizomes of *Smilax myosotiflora*.

Extraction: The rhizomes of *S. myosotiflora* (4.20 kg) was collected from Kedah (Peninsular Malaysia) in March 2011. After that, it was washed with distilled water, then air-dried under shadow at room temperature and finally ground to fine powder. The ground rhizomes were then extracted in a Soxhlet apparatus with n-hexane, dichloromethane and methanol to get three extracts respectively.

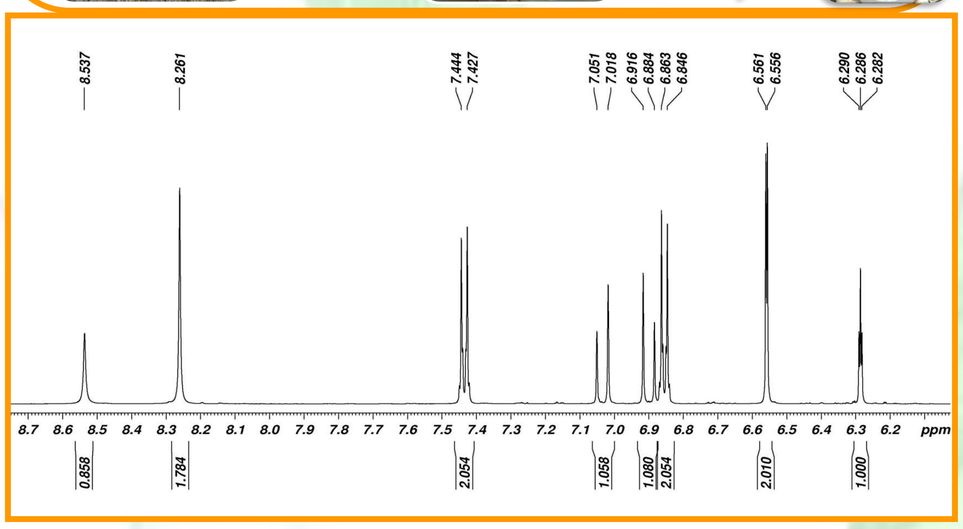
Isolation

The methanol extract was suspended in water and partitioned with, chloroform, ethyl acetate and n-butanol sequentially. The acetate extract (was subjected to column chromatography (CC) on silica gel and eluted with n-Hex: EtOAc to give 9 fractions (Fr.1–9) based on TLC analysis . Fr. 2 was applied to CC on a silica gel and fractionated using n-Hex:EtOAc (2:8) gradient system. All fractions obtained were pooled based on TLC analysis to afford 4 sub-fractions. Fr. 2.2 was subjected to silica gel CC and eluted with gradient solvent system n-Hex:EtOAc to yield 9 sub-fractions. Fr. 2.2.5 was further purified by using Sephadex LH-20, eluting with acetone, to give **Comp (1).** Fr. 2.3 was chromatographed on a silica gel CC with n-Hex: EtOAc (2:8) as the eluting solvent to give 6 sub-fractions. Fr. 2.3.3 was rechromatographed on a silica gel CC and eluted with n-Hex: EtOAc (3:7) to yield 4 sub-fractions. Fr. 2.3.3.1 was further purified by Sephadex LH-20 by using methanol to give **Comp (2).**

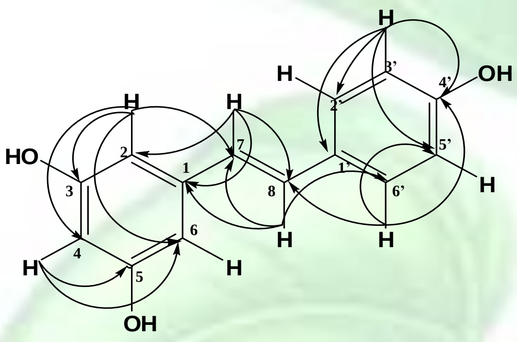
RESUTS

Compound (1) was colorless needles (MeOH), $C_{14}H_{12}O_3$. ESI-MS m/z 226.9 $[M-1]^-$,^{1}H NMR (aceton-d_6, 500 MHz) : 6.30 (1H, t, J = 2.0 Hz, H-4), 6.57 (2H, d, J = 2.0 Hz, H-2, 6), 6.86 (2H, d, J = 8.5 Hz, H-3', 5'), 6.91 (1H, d, J = 16.5 Hz, H-7), 7.03 (1H, d, J = 16.5 Hz, H-8), 7.43 (2H, d, J = 8.5 Hz, H-2', 6'), 8.21(2-OH), 8.48(1-OH). ^{13}C NMR spectroscopic 139.99 (C-1), 104.78(C-2, 6), 158.68 (C-3, 5), 101.76 (C-4), 125.94(C-7), 128.21 (C-8), 129.08 (C-1'), 127.84 (C-2', 6'), 115.51 (C-3',5'), 157.25 (C-4')

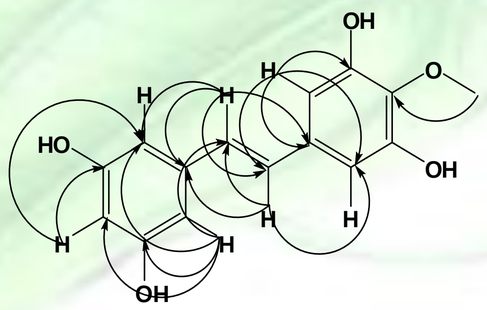
Compound (2) was pale yellow, $C_{15}H_{14}O_5$. ESI-MS m/z 272.9 $[M-1]^-$,^{1}H NMR (aceton-d_6, 500 MHz) : 6.29 (1H, t, J = 2.0 Hz, H-4), 6.55 (2H, d, J = 2.0 Hz, H-2, 6), 6.86 (1H, d, J = 16.5 Hz, H-7), 6.91 (1H, d, J = 16.5 Hz, H-8), 6.64 (2H, s, H-2', 6'), 3.82(3H,s,-OCH_3), 8.13(2-OH), 8.38(2-OH). ^{13}C NMR spectroscopic 140.47 (C-1), 105.89 (C-2, 6), 159.62 (C-3, 5), 103.03 (C-4), 128.72(C-7), 129.24 (C-8), 134.23 (C-1'), 106.77 (C-2', 6'), 151.49 (C-3',5'), 136.30 (C-4'),60.73(OCH_3,q). The obtained results in conclusion, were conformed in comparison with spectral data available in some other previous studies .



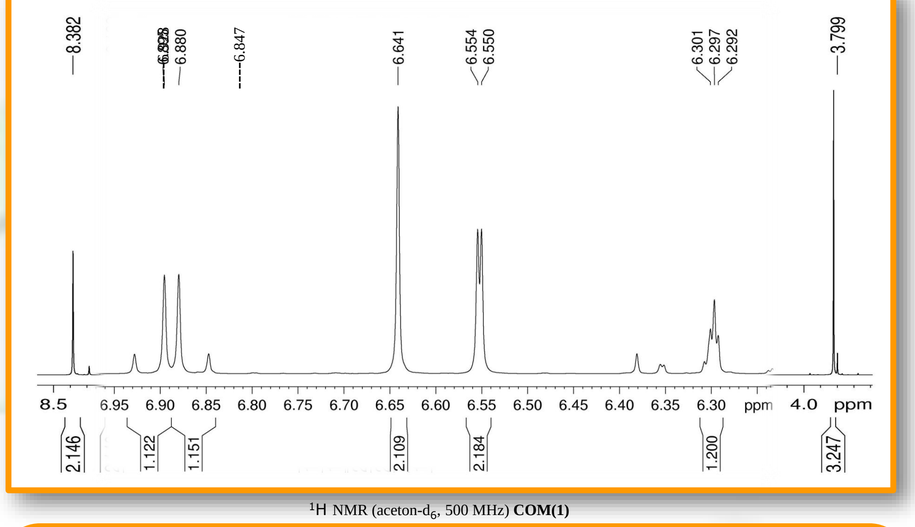
^{1}H NMR (aceton-d_6, 500 MHz) **COM(1)**



HMBC- NMR (aceton-d_6, 500 MHz) **COM(1)**



HMBC- NMR (aceton-d_6, 500 MHz) **COM(2)**



^{1}H NMR (aceton-d_6, 500 MHz) **COM(1)**

References

1] Damayanthi D, Azman MAB, Aminuddin AHK, Hamid A, Nwe KHH;, *Biomedical Research*, 22(2) (2011)188-193.
[2] Bo Shao. Hongzhu Guo, Yajun Cui, Min Ye, Jian Han, Dean Guo;, *Phytochemistry*, 68(2007) 623-630.
[3]Yao-Haur Kuo, Ya-Wen Hsu,Chia-Ching Liaw,Jiun Kuan Lee,Hui-Chi Huang, Li-Ming Yang Kuo;, *J.Nat.Prod*, 68 (2005)1475-1478.
[4] Zhari bin Ismail, Norhayati bent Ismail, Jaafar bin Lassa;, *Malayisan Herbal MonoGraph*, Malaysian monograph committee,Kuala Lumpur, (1999) P.67.
[5] Lijie Zhang, Yao-Haur Kuo, Chia-Ching Liao, Hui-Chi Huang, Ya-Chi Hng Shen, Li-Ming Yang;, *Phytochemistry*, 69(2008)1398-1404.
[6]Marc Sautour, Tomofumi Miyamoto, Marie-Aleth Lacaille-Dubois;, *J.Nat.Prod*,68(2005)1489-1493.
[7]Robson Roney Bernardo, Antonio Ventura Pinto, Jose Paz Parente;, ;, *Phytochemistry*, 43(2)(1996)465-469.
[8]Shu Yuau Li, Hiroyuki Fuchino, Nobuo Kawahara, Setsuko Sekita, Motoyoshi Satake;, *J.Nat.Prod*,65(2002)262-266.
[9] N. Ozsoy, A. Can, R. Yanardag, N. Akev;, *Food Chemistry*, 110 (2008)571-583.
[10] Li-Sheng Wu, Xiao-Jing Wang, Hong Wang, Hi-Wei yang, Ai-Qun Jia,Qiang Ding;, *Journal of Ethnopharmacology*, 130(2010) 460-464.
[11] Wieslaw Oleszek, Magdalena Sitek, Anna Stochmal, Sonia Piacennte,Cosimo Pizza, Peter Cheeke;, *J.Agric.Food Chem*;, 49 (2001) 747-752.

Acknowledgments

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Conclusion:

To stilbenoid compounds (1) & (2) were isolated from ethyl acetate extract of the rhizomes of *Smilax myosotiflora*. Their structures were established by NMR and ESI-MS spectroscopic analysis.