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A novel polyphenolic compound isolated from *Acacia sieberiana*

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ABSTRACT

Chromatographic separation of 80% MeOH extract of the leaves of *Acacia sieberiana* was performed. Chemical structures of the isolated compounds were established by spectral techniques (UV, ^{1}H, and ^{13}C NMR, MS) resulted in identification of a novel polyphenolic compound 6,7,8-trihydroxy-3,4'-dimethoxy dihydroflavone with seven known polyphenolic compounds isolated for the first time from this species.

KEYWORDS

Fabaceae;
Acacia sieberiana;
Tannins and flavonoids.

INTRODUCTION

The family Leguminoseae (Fabaceae) is particularly rich in flavonoids and related compounds[9]. Among the Fabaceae is the genus *Acacia* which comprises about 1350 species and distributed in tropical and subtropical regions[21]. *Acacia* species contain variety of bioactive components such as phenolic acid, alkaloids, tannins and flavonoids which have numerous biological and pharmacological properties as hypoglycemic, analgesic, anti-inflammatory, antihypertensive, antiatherosclerotic, anthelmintic, antibacterial and anticancer[3,5,11,17,20,22]. No studies report on the constitutive polyphenols of *Acacia sieberiana*. Thus, it was deemed necessary to carry out this phytochemical and biological study to throw light on this important species native to Central America and Mexico and naturalized in over 150 countries including Egypt and KSA[24]. The present study deals with the isolation and identification of some polyphenolic constituents of *Acacia sieberiana* species growing in KSA.

MATERIALS AND METHODS

Instruments and materials

^{1}H- NMR spectra were recorded at 300 MHz on a Varian Mercury 300. While ^{13}C- NMR spectra were recorded at 75 MHz. The δ-values are reported as ppm relative to TMS in DMSOd_6 and *J*-values are in Hz. ESI-MS spectra were measured on mass spectrometer connected to an ESI-II ion source (Finnigan, Lc-MSLCQdeca. Advantage MAX, Finnigan Surveyor LC pump) (Department of Biological Genetics, National Research Center, Cairo, Egypt). The UV analysis for pure samples was recorded on a Shimadzu UV 240 spectrophotometer, separately as solutions in methanol and with different diagnostic UV shift reagents. Fractionation of the extracts was done by column chromatography using polyamide 6S (Riedel-De Han Ag, Seelze Hannover, Germany), isolation and purification of compounds was done on either cellulose (Pharmacia, Uppsala, Sweden) or Sephadex LH-20 columns (Fluka, Switzerland) of different dimensions and eluted with

different solvent systems. Separation processes were followed up by 2D-PC and CoPC using Whatmann No. 1 paper with (S1) *n*-BuOH-AcOH-H_2O (BAW) (4:1:5, top layer) and (S2) 15% aqueous acetic acid as solvent systems.

Plant material

Fresh leaves of *Acacia sieberiana* were collected from a mature tree growing in KSA during April 2011. Identification of the plant was confirmed by Dr. Kamal zayed, Department of Flora and Taxonomy Beni-Suif University, Egypt.

EXTRACTION AND ISOLATION

Powdered, air-dried leaves of *Acacia sieberiana* (1.5 kg) were exhaustively extracted with hot 80% MeOH (4 × 3 L), under reflux. The dry residue obtained was suspended in absolute methanol. The methanol soluble part was concentrated under vacuum then was fractionated on a polyamide column and was eluted with water followed by water/methanol mixtures of decreasing polarities to afford five collective fractions. These fractions were concentrated under vacuum and fractionated on Sephadex LH-20 column and or cellulose columns using BIW/ MeOH or absolute MeOH for elution.

RESULTS AND DISCUSSION

The methanol extract of leaves of *Acacia sieberiana* resulted in isolation of 7 known polyphenolic compounds which identified as Ellagic acid, gallic acid, isoferulic acid, Quercetin, kaempferol, Quercetin 3-O-β-D-glucoside and Kaempferol 3-alpha-L-arabinoside. The known isolated compounds identified on the basis of acid hydrolysis, comparative PC, UV, ESI-MS, ^{1}H-, ^{13}C-NMR and in some cases 2D-NMR spectroscopic analyses and comparing with previous reported data[1,2,4-6,8-10,13-17,27]. In addition of isolation of new natural polyphenolic compound this described as the following:

The compound is dull yellow amorphous powder R_f values: 0.64 (S1) and 0.53 (S2); it was appeared on PC as a faint brown spot. The UV spectrum in methanol showed an intense band II at 264 nm with low intensity band I at 360 nm, which are ideal with those of dihydroflavonol. UV λ_{max} nm (MeOH): 256, 264, 360; +NaOEt: 255, 303, 370; +NaOAc: 255, 320, 369; +NaOAc/H_3BO_3: 261, 272, 359; + $AlCl_3$: 272, 296, 370; + $AlCl_3$/HCl: 252, 303, 361; ^{1}H-NMR (DMSO-d_6): δ (ppm) 7.4 (s, *J*=8 Hz, H-5); 7.62 (2H, d, *J*=9 Hz, H-2' and H-6'); 7.1 (2H, d, *J*=8 Hz, H-3' and H-5'); 5.3 (dd, *J*=10, 9 Hz, H-2); 4.2 (d, *J*=10 Hz, H-3); 3.85&3.78 (s, 2 methoxyl groups); ESI-MS: m/z 334.1 (100%), 319 [M-1 methoxyl group], 304.1 [M-2 methoxyl groups]. The hypthochromic shift of 11 nm in band II of its methanol curve and the bathochromic shift of 40 nm in band II on the addition of NaOAc indicated the lack of 5-hydroxyl group and or substitution of 3-hydroxyl group[19,26], while hydroxylation in the A-ring was proved from both the bathochromic shift (12 nm) of the major band II on the addition of NaOAc/H_3BO_3 and the bathochromic shift in the $AlCl_3$/HCl spectra relative to that of $AlCl_3$. The ^{1}H-NMR spectrum exhibited the typical AB system due to the presence of a double of doublet signal at δ 5.3. (*J*=10 Hz) and a doublet at δ 4.2 (*J*=10 Hz) of H-2 and H-3 respectively, the appearance of a singlet signal at δ 7.61 ppm was assigned to a 5-deoxy[7,19]. The downfield shift of (H-3' and 5') at δ 7.1 ppm and (H-2' and 6') at δ 7.62 ppm signals indicated methylation at 4'-position[12], this was ensured by the presence of singlet signal at δ 3.78 ppm of the methoxyl groups. R_f- values together with UV spectral data with $AlCl_3$ and $AlCl_3$/HCl showed methylation at position 3, this was ensured by the presence of singlet signal at δ 3.85 ppm of the methoxyl group. This also was confirmed by ESI-MS and ^{1}H-NMR. This were finally confirmed by MS, which gave M+1 at m/z 334.1 (100%) constitute with a molecular formula $C_{17}H_{15}O_7$ with molecular weight of 333 while the peak fragment of 304.1 [M-2 methoxyl groups] indicated the presence of two methyl group. Thus the compound was identified as 6,7,8- trihydroxy-3,4'-dimethoxy dihydroflavone.

CONCLUSIONS

In conclusion, the fractionation of methanol extract of the leaves of *Acacia sieberiana* resulted in isolation of eight polyphenolic compounds, one of them considered as a new natural product (6,7,8- trihydroxy-3,4'-dimethoxy dihydroflavone)

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