CONSTITUENTS OF *Lychnophora pinaster* HYDROALCOHOLIC EXTRACT

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The medicinal plant *Lychnophora pinaster* (*Asteraceae, Vernonieae*) belongs to a genus restricted to the Brazilian "cerrado." A previous phytochemical analysis of apolar extracts yielded α- and β-amyrin, lupeol, friedeline, and one cariofilene derivative, namely, lychnophoic acid [1]. In this work, we focused on the polar fractions of the plant. In this way, the aerial parts of *L. pinaster* (voucher No. 60265) collected at Ingai, Minas Gerais, were powdered (233 g) and extracted with EtOH–H_2O (65:35). Dry hydroalcoholic extract (53 g) was partitioned on a triphasic mixture of hexane–acetonitrile–$CHCl_3$–H_2O (2:3.4:1:1). The aqueous layer (25 g) was fractionated on silica gel CC using EtOAc–MeOH (10:0 up to 9:1) to give seven pooled fractions after TLC analysis. Pool 1 was submitted to preparative HPLC on 250 mm × 20 mm C18 Shim-pack column (Shimadzu) with acetronitrile:H_2O gradient at 6 mL/min to give compounds **1–3**. Compound **4** was identified as the major phenolic in pool 2 by comparing UV spectra and retention times against authentic standard compound [2]. To the best of our knowledge, all the four compounds identified are reported for *L. pinaster* for the first time.

Compound 1. Yellow powder; UV spectrum (acetonitrile: H_2O, λ_{max}, nm): 204, 215, 275 (log ε 2.98, 2.97, 3.16). PMR (200 MHz, CD_3OD, δ, ppm, J/Hz): 6.46 (1H, d, J_{87} = 16, H-8), 7.38–7.43 (3H, m, H-3, H-4, H-5) 7.52–7.57 (2H, m, H-2, H-6) 7.89 (1H, d, J_{78} = 16, H-7), 11.25 (1H, COOH). ^{13}C NMR (100 MHz, CD_3OD): 117.47 (CH, C-8), 128.59 (2CH, C-2, C-6), 129.18 (2CH, C-3, C-5), 130.98 (C, C-4), 134.23 (C, C-1), 147.33 (CH, C-7), 172.49 (C, C-9). Identified as cinnamic acid according to previously published spectral data [3].

Compound 2. White powder; UV spectrum (acetonitrile: H_2O, λ_{max}, nm): 218, 235, 296 (sh), 322 (log ε 2.46, 2.35, 2.50, 2.57). PMR (400 MHz, CD_3OD, δ, ppm, J/Hz): 6.22 (1H, d, J_{87} = 15.9, H-8), 6.78 (1H, d, J_{56} = 8.2, H-5), 6.94 (1H, dd, J_{62} = 2.0, J_{65} = 8.2, H-6), 7.04 (1H, d, J_{26} = 2.0, H-2), 7.53 (1H, d, J_{78} = 15.9, H-7). ^{13}C NMR (100 MHz, CD_3OD): 115.3 (CH, C-8), 115.6 (CH, C-2), 116.7 (CH, C-5), 123.0 (CH, C-6), 127.9 (C, C-1), 146.9 (C, C-3), 147.2 (CH, C-7), 149.6 (C, C-4), 171.2 (C, C-9). Identified as caffeic acid [4].

Compound 3. Yellow powder; UV spectrum (acetonitrile: H_2O, λ_{max}, nm): 209, 290 (log ε 2.78, 2.60). PMR (400 MHz, CD_3OD, δ, ppm, J/Hz): 4.60 (1H, d, J_{32} = 11.5, H-3), 5.08 (1H, d, J_{23} = 11.5, H-2), 5.92 (1H, d, J_{86} = 2.1, H-8), 5.95 (1H, d, J_{68} = 2.1, H-6), 7.38–7.45 (3H, m, H-3′, H-4′, H-5′), 7.52–7.55 (2H, m, H-2′, H-6′). ^{13}C NMR (100 MHz, CD_3OD): 72.3 (CH, C-3), 83.6 (CH, C-2), 95.0 (CH, C-8), 96.1 (CH, C-6), 100.4 (C, C-10), 127.5 (2CH, C-2′, C-6′), 128.0 (2CH, C-3′, C-5′), 128.5 (CH, C-4′), 137.1 (C, C-1′), 163.0 (C, C-9), 163.7 (C, C-5), 167.3 (C, C-7), 196.8 (C, C-4). Identified as pinobanksin according to COSY, HSQC, HMBC, and previously published spectral data [5].

Compound 4. R_t 31.26 min; UV spectrum (acetonitrile: H_2O, λ_{max}, nm): 218, 240, 298 (sh), 326 (log ε 2.76, 2.67, 2.81, 2.91). Identified as chlorogenic acid by HPLC analysis.

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