METHOD FOR OBTAINING TOTAL FLAVONOIDS FROM Climacoptera subcrassa AND THEIR BIOLOGICAL ACTIVITY

A. K. Kipchakbaeva,¹ R. A.-A. Khamid,^{1,2} B. K. Eskalieva,^{1*} G. Sh. Burasheva,¹ Zh. A. Abilov,¹ S. R. Numonov,³ and H. A. Aisa³

Isolation of biologically active compounds (BAC) from plants is an important step in drug manufacturing. Therefore, the analysis of and search for modern and effective methods for isolating valuable BAC raw materials for the drug industry have definite theoretical and practical interest [1, 2].

Column chromatography over macroporous AV-8 resin was used to separate total flavonoids. This method was used for the first time to separate total flavonoids from the aerial part of *Climacoptera subcrassa* (Chenopodiaceae).

Total flavonoids from *C. subcrassa* were purified over the macroporous adsorption resin. Their contents were determined using UV spectrophotometry. The conditions for purifying them using AV-8 macroporous resin were optimized. EtOH (70%) gave the highest yield (3.4 g) of flavonoids from the plant (100 g); 50% EtOH, 2.2 g; 80% EtOH, 2.75 g; and 95% EtOH, 1.60 g. The maximum static adsorption capacity was 23.06 mg/g of resin; adsorption rate, 89.22 mL/min; absorption time, 2 h at 80°C. Under these conditions, up to 90.8% of total flavonoids were isolated. Total flavonoids from the 70% EtOH extract showed pronounced antioxidant activity with $IC_{50} = 49.66 \pm 4.57 \mu g/mL$. The standard was vitamin C with $IC_{50} = 5.34 \pm 0.42 \mu g/mL$ [3, 4].

Compounds 1–5 were isolated using AV-8 macroporous resin and HPLC and were identified using physicochemical data and comparisons with the literature.

Quercetin (1). $C_{15}H_{10}O_7$, light-yellow crystals, mp 315–316°C. ESI-MS, *m/z* 301.03 [M – H][–]. ¹H NMR spectrum (400 MHz, MeOH-d₄, δ , ppm, J/Hz): 7.73 (1H, d, J = 2.0, H-2'), 7.63 (1H, dd, J = 2.0, 8.5, H-6'), 6.89 (1H, d, J = 8.5, H-5'), 6.39 (1H, d, J = 1.9, H-8), 6.19 (1H, d, J = 1.9, H-6) [5].

Quercetin 3-*O*-β**-D**-Glucopyranoside (2). $C_{21}H_{20}O_{12}$, light-yellow crystals, mp 269–271°C. ESI-MS *m/z*: 477 [M – H]⁻, 315 [M – H – 162]⁻. ¹H NMR spectrum (400 MHz, Py-d₅, δ, ppm, J/Hz): 7.89 (1H, d, J = 2.4, H-2'), 7.55 (1H, dd, J = 8.8, 2.4, H-6'), 7.06 (1H, d, J = 1.6, H-8), 7.04 (1H, d, J = 8.8, H-5'), 7.03 (1H, s, H-3), 6.86 (1H, d, J = 1.6, H-6), 5.56 (1H, d, J = 7.2, H-1''), 4.57 (1H, d, J = 12.4, H-6''), 4.43 (1H, d, J = 8.8, H-3''), 4.40 (1H, dd, J = 12.4, 5.2, H-6''), 4.37 (1H, d, J = 8.8, H-2''), 4.37 (1H, d, J = 8.8, H-4''), 4.25 (1H, d, J = 8.8, H-5''), 3.80 (3H, s, OCH₃). ¹³C NMR spectrum (100 MHz, Py-d₅, δ, ppm): 165.2 (C-2), 105.2 (C-3), 183.3 (C-4), 163.6 (C-5), 101.1 (C-6), 164.5 (C-7), 95.8 (C-8), 158.3 (C-9), 107.0 (C-10), 124.7 (C-1'), 114.9 (C-2'), 149.5 (C-3'), 152.5 (C-4'), 112.5 (C-5'), 119.3 (C-6'), 56.3 (OCH₃), 102.2 (C-1''), 75.2 (C-2''), 78.9 (C-3''), 71.5 (C-4''), 79.7 (C-5''), 62.7 (C-6'') [6].

Rutin (3). $C_{27}H_{30}O_{16}$, light-yellow crystals, mp 242°C. ESI-MS *m/z*: 609 [M-H]⁻, 301 [M-H-162-146]⁻. ¹H NMR spectrum (400 MHz, Py-d₅, δ , ppm, J/Hz): 7.66 (d, J = 1.7, H-2'), 7.63 (dd, J = 8.4, 1.7, H-6'), 6.8 (d, J = 8.4, H-5'), 6.4 (d, J = 1.7, H-8), 6.2 (d, J = 1.6, H-6), 5.19 (d, J = 7.5, H-1''), 4.5 (s, H-1''), 0.8 (3H, s, CH₃) [6].

3-O-Methylquercetin (4). $C_{16}H_{12}O_7$, light-yellow crystals, mp 269–271°C. ESI-MS *m/z* 315 [M – H][–]. ¹H NMR spectrum (400 MHz, Py-d₅, δ , ppm, J/Hz): 8.64 (1H, d, J = 2.2, H-2'), 8.14 (1H, dd, J = 8.4, 2.2, H-6'), 7.59 (1H, s), 7.41 (1H, d, J = 8.5, H-5'), 7.22 (1H, d, J = 1.5), 6.78 (1H, d, J = 2.0, H-8), 6.74 (1H, d, J = 2.0, H-6), 3.91 (3H, s, OCH₃). ¹³C NMR spectrum (100 MHz, Py-d₅, δ , ppm): 145.3 (C-2), 137.9 (C-3), 177.3 (C-4), 162.4 (C-5), 99.2 (C-6), 165.5 (C-7), 94.3 (C-8), 157.5 (C-9), 104.5 (C-10), 123.8 (C-1'), 116.7 (C-2', 5'), 147.1 (C-3'), 147.7 (C-4'), 121.0 (C-6'), 55.4 (OCH₃) [7].

¹⁾ Al-Farabi Kazakh National University, Department of Chemistry and Chemical Technology, 71 Al-Farabi Prosp., Almaty, 050040 Kazakhstan, fax: (+7 727) 292 37 31, e-mail: balakyz@mail.ru; 2) Al Azhar University, Faculty of Pharmacy, Assiut, Egypt; 3) Xinjiang Technical Institute of Physics and Chemistry, CAS, 830011, Urumqi, P. R. China. Translated from *Khimiya Prirodnykh Soedinenii*, No. 2, March–April, 2016, pp. 281–282. Original article submitted May 8, 2015.

Kaempferol (5). $C_{15}H_{10}O_6$, light-yellow crystals, mp 276–278°C. ESI-MS *m/z* 285 [M – H]⁻. ¹H NMR spectrum (400 MHz, Py-d₅, δ , ppm, J/Hz): 8.09 (2H, d, J = 8.8, H-2', 6'), 6.91 (2H, d, J = 8.8, H-3', 5'), 6.40 (1H, d, J = 2.0, H-8), 6.19 (1H, d, J = 2.0, H-6). ¹³C NMR spectrum (100 MHz, MeOH-d₄, δ , ppm): 148.0 (C-2), 137.1 (C-3), 173.3 (C-4), 162.5 (C-5), 99.3 (C-6), 165.7 (C-7), 94.4 (C-8), 158.2 (C-9), 104.5 (C-10), 123.7 (C-1'), 116.3 (C-2', 6'), 130.6 (C-3', 5'), 160.5 (C-4') [8].

Total flavonoids were isolated from plants of the genus *Climacoptera* (*C. subcrassa*, Chenopodiaceae) for the first time using macroporous AV-8 resin [9, 10].

REFERENCES

- 1. V. P. Goloskokova, Illustrated Guide to Plants of Kazakhstan [in Russian], Vol. 1, Nauka, Alma-Ata, 1969, 644 pp.
- 2. K. R. Markham, in: *Methods in Plant Biochemistry*, Vol. 1, Academic Press, London, 1989, pp. 203–210.
- 3. C. A. Rice-Evans, N. J. Miller, G. P. Bolwell, P. M. Bramley, and J. B. Pridham, Free Radical Res., 22, 375 (1995).
- 4. N. J. Miller, J. Sampson, L. P. Candeias, P. M. Bramley, and C. A. Rice-Evans, FEBS Lett., 384, 240 (1996).
- 5. X. C. Su, L. Chen, and H. A. Aisa, Chem. Nat. Compd., 44, 365 (2008).
- 6. A. Malik, M. P. Yuldashev, A. Obid, T. Ismoil, and L. Ya. Ping, Chem. Nat. Compd., 38, 612 (2002).
- 7. M. G. Campos, R. F. Webby, and K. R. Markham, Z. Naturforsch., C: J. Biosci., 57, 944 (2002).
- 8. A. Sultan, Bahang, H. A. Aisa, and K. A. Eshbakova, Chem. Nat. Compd., 44, 366 (2008).
- 9. H. Sasaki, M. Takei, M. Kobayashi, R. B. Pollard, and F. Suzuki, *Pathobiology*, **70**, 229 (2002).
- A. Sofowara, *Medicinal Plants and Traditional Medicines in Africa*, Spectrum Books Ltd., Ibadan, Nigeria, 1993, 125 pp.