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CHEMICAL COMPOSITION OF *Callisia fragrans* JUICE
1. PHENOLIC COMPOUNDS

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Callisia fragrans Wood. (Commelinaceae) is a cultivated perennial succulent herbaceous plant, the juice of which is widely used at present as a drug. The chemical composition of this species is insufficiently studied. Information is available on the composition of neutral, glyco-, and phospholipids of the leaves, runners, and stems. Carotinoids, chlorophylls, ascorbic acid, and anthocyans have been found [1]. The goal of our work was to investigate the composition of the phenolic compounds from *C. fragrans* juice.

C. fragrans raw material (runners) was supplied by the experimental farm of N. I. Vavilov Saratov State Agricultural University (Saratov, Russia). Juice was extracted by grinding fresh raw material (27 kg) in a blender, squeezing the resulting pulp through cloth, and filtering the juice through filter paper under vacuum. The resulting juice (21.5 L) was concentrated to 3 L. The residue was extracted successively with hexane, $CHCl_3$, and ethylacetate. Solvents were removed to produce hexane (63 mg), $CHCl_3$ (1.21 g), and ethylacetate (4.30 g) extracts.

The $CHCl_3$ extract was chromatographed over a column of silica gel (Chemapol, Silicagel, L 100/400) using a gradient of hexane→hexane:ethylacetate (90:10). The resulting fractions were purified by preparative TLC (PTSKh-AF-V Sorbfil plates, 160 μm, Sorbpolimer, solvent system ethylacetate:MeOH:H_2O, 100:13.5:10). The ethylacetate fraction was separated by column chromatography over polyamide (Woelm for CC) using H_2O→5-95% ethanol followed by preparative TLC (solvent system ethylacetate:toluene:HCOOH:H_2O, 100:5:10:10). Bands of pure compounds were eluted with methanol, concentrated, and recrystallized.

Seven compounds were isolated by chromatography. The $CHCl_3$ extract gave aloe-emodin (**1**), umbelliferone (**2**), and scopoletin (**3**); ethylacetate, quercetin (**4**), gallic (**5**), caffeic (**6**), and chicoric acids (**7**).

Aloe-emodin (1), $C_{15}H_{10}O_5$, mp 218°C (MeOH), UV (MeOH, λ_{max}, nm): 253, 286, 431; +NaOH: 237, 286, 527 [2].

Umbelliferone (2), $C_9H_6O_3$, mp 232°C (MeOH), UV (MeOH, λ_{max}, nm): 245, 255, 300, 324 [3].

Scopoletin (3), $C_{10}H_8O_4$, mp 202°C (MeOH), UV (MeOH, λ_{max}, nm): 230, 255, 299, 345 [3].

Quercetin (4), $C_{15}H_{10}O_7$, mp 316°C (EtOH), UV (MeOH, λ_{max}, nm): 255, 268, 370; +CH_3COONa: 270, 405; +C_2H_5ONa: 274, 440 [4].

Gallic acid (5), $C_7H_6O_5$, mp 238-240°C (EtOH), UV (EtOH, λ_{max}, nm): 211, 274 [5].

Caffeic acid (6), $C_9H_8O_4$, mp 220°C (EtOH), UV (EtOH, λ_{max}, nm): 240, 300, 327; +CH_3COONa: 281, 310; +C_2H_5ONa: 250, 362 [6, 7].

Chicoric acid (7), $C_{22}H_{18}O_{12}$, mp 202°C (H_2O), UV (EtOH, λ_{max}, nm): 245, 300 sh, 330 [6]. Chromatography of the products from basic hydrolysis detected caffeic and quinic acids (PC, FN-12, solvent system AcOH, 15%).

The ethylacetate fraction of *C. fragrans* was also analyzed by HPLC [Gilston 305 with Rheodyne 7125 manual injector, Kromasil C18 column (4.6 × 250 mm, 5 μm particle size), mobile phase MeOH:H_2O:H_3PO_4 (400:600:5), flow rate 0.8 mL/min, UV detector (Gilston UV/VIS 151, λ = 254 nm)].

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The fraction (0.1 g) was transferred to a 25-mL volumetric flask, dissolved in ethanol (70%, 20 mL) in an ultrasonic bath at 50°C, adjusted to the mark with the same solvent, and chromatographed. A series of solutions (0.05%) of standard reference compounds in ethanol (70%) (rutin, quercetin, luteolin, hesperedin, apigenin, kaempferol, and hyperoside and gallic, caffeic, chlorogenic, chicoric, cinnamic, and ferulic acids) was prepared in parallel.

HPLC detected 12 compounds, of which we identified gallic acid (38.12% of the mass of the ethylacetate fraction), caffeic acid (2.97), chicoric acid (7.16), ferulic acid (0.50), quercetin (6.51), and kaempferol (0.22).

The total content of phenolic compounds in *C. fragrans* juice that was determined by the Folin method [8] was 7.12 μg/mL calculated for gallic acid or 3.21 mg/g of dry juice compounds.

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