

PHYTOCHEMICAL INVESTIGATION OF *CASSIA ABSUS* (ROOTS AND LEAVES)

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All plant parts of *Cassia absus* Linn. are used in folk medicine. According to Chopra *et al.* (1) the leaves are used for treatment of tumors and asthma. The root is used for treatment of constipation. Siddiqui and Ahmed (2, 3) isolated the alkaloids chaksine and isochaksine from the seeds, but there is no report of a chemical examination of the roots and leaves of this plant. The present paper reports the isolation of chrysophanol, aloe-emodin, chaksine and isochaksine from the roots and quercetin, rutin, and the above two alkaloids from the leaves. The reported medicinal uses of the roots are consistent with the presence of chrysophanol and aloe-emodin.

EXPERIMENTAL

PLANT MATERIAL.—The plants, which grow abundantly around the Andhra University Campus, were collected, the roots and leaves were separated. The identity of the plant material was established by Dr. P. N. Rao, Department of Botany, Andhra University.

EXAMINATION OF ROOTS.—Dried, powdered roots (1 kg) were extracted with ethanol (8 liters), and the resulting extractive was concentrated under reduced pressure. The concentrate was suspended in water (500 ml), adjusted to pH 6 with dilute sulfuric acid and extracted successively with petroleum ether, benzene, and ethyl acetate. The residual aqueous suspension was neutralized with ammonia (pH 8.0) and extracted with ethyl acetate-methanol (9:1).

ISOLATION OF CHRYSOPHANOL AND ALOE-EMODIN.—The residue remaining after evaporation of the solvent from the benzene extract gave a pink color with alkali and ammonia, thus indicating the presence of anthraquinones. The residue (2 g) was taken up in benzene and chromatographed on silica gel (60 g) by elution with benzene-ethyl acetate mixtures. Crystallization of the product from the benzene-ethyl acetate (3:1) eluate gave pale yellow needles (120 mg), mp 194°, uv λ max (EtOH) 227, 257.5, 277, 289 nm.

Anal. Calcd for $C_{15}H_{10}O_4$: C, 70.86; H, 3.96. Found: C, 71.04; H, 3.92.

The methyl ether was prepared by using diazomethane, mp 190°.

Anal. Calcd for $C_{17}H_{14}O_4$: C, 72.33; H, 5.00; OCH_3 , 22.00.

Found: C, 72.52; H, 4.99; OCH_3 , 21.86.

These properties corresponded with those of chrysophanol and its dimethyl ether. Further confirmation of the identity was provided by mixture mp, tlc and ir comparison with reference chrysophanol. The product from the benzene-ethyl acetate (1:3) eluate on crystallization gave orange yellow needles (150 mg), mp 221°, uv λ max (EtOH) 225, 255, 274, 285 nm.

Anal. Calcd for $C_{13}H_{10}O_5$: C, 66.65; H, 3.72. Found: C, 66.18; H, 4.02.

The acetate was prepared by using acetic anhydride and pyridine, mp 172°.

Anal. Calcd for $C_{21}H_{16}O_5$: C, 63.63; H, 4.06; $COCH_3$, 30.05.

Found: C, 63.80; H, 3.90; $COCH_3$, 30.30.

These properties were identical with those of aloe-emodin and its triacetate. The identity was confirmed by direct comparison with reference aloe-emodin.

ISOLATION OF CHAKSINE AND ISOCHAKSINE.

—The residue (1.6 g) from evaporation of the solvent from the ethyl acetate-methanol (9:1) extract was chromatographed on silica gel (45 g) using ethyl acetate-methanol mixtures. Two amorphous fractions (I and II) were obtained from ethyl acetate-methanol (199:1) and ethyl acetate-methanol (49:1) eluates, respectively. These substances gave positive tests for alkaloids. Fraction I was obtained as a powder (120 mg) and found to be a single component by tlc (solvent system: ethyl acetate-methanol, 98:2).

Anal. Calcd for $C_{11}H_{12}O_3N_3$: C, 54.30; H, 8.69; N, 17.28.

Found: C, 54.60; H, 8.55; N, 17.06.

The chloride was prepared (3) by adding barium chloride to a hot solution of the substance (20 mg) in aqueous methanol and crystallizing the precipitate from methanol, mp 175°. The picrate was prepared by adding a saturated solution of picric acid to the substance (20 mg) in aqueous methanol and crystallizing the precipitate from methanol, mp 236°. Fraction II was also obtained as a powder (100 mg) and was found to be a single component by tlc (solvent system: chloroform-methanol, 95:5).

Anal. Calcd for $C_{11}H_{12}O_3N_3$: C, 54.30; H, 8.69; N, 17.28.

Found: C, 54.18; H, 8.76; N, 17.08.

The chloride and picrate were prepared as described for fraction I. The chloride melted at 248° and the picrate at 182-4°. The properties of fractions I and II compared well with those reported for chaksine and isochaksine, respectively (4, 5). Their identity was confirmed by direct comparison with authentic samples of chaksine and isochaksine isolated from the seeds of *C. absus*.

EXAMINATION OF LEAVES.—Dried powdered leaves (1 kg) were extracted with ethanol (10 liters). The concentrate of the extract was fractionated as described above for roots.

The residue (2.2 g) remaining after evaporation of the solvent from the ethyl acetate extract was dissolved in benzene and chromatographed on silica gel (60 g) by elution with solvents of increasing polarity. The benzene-ethyl acetate (1:1) eluate gave a flavonoid compound (160 mg) mp 312°.

Anal. Calcd for $C_{15}H_{10}O_7$: C, 59.60; H, 3.33.

Found: C, 59.88; H, 3.50.

The acetate was prepared by refluxing with acetic anhydride and sodium acetate, mp 192-4°.

Anal. Calcd for $C_{25}H_{20}O_{12}$: C, 58.59; H, 3.93; $COCH_3$, 42.00.

Found: C, 58.65; H, 3.80; $COCH_3$, 42.35.

The compound was found to be identical with quercetin by mixture mp, uv and pc comparison with authentic quercetin. The ethyl acetate-methanol (99:1) eluate gave another flavonoid compound (120 mg) mp 186°.

Anal. Calcd for $C_{27}H_{20}O_{16}$: C, 53.10; H, 4.95.

Found: C, 53.60; H, 4.82.

Acetate mp 116-8°.

Anal. Calcd for $C_{41}H_{44}O_{23}$: C, 54.44; H, 4.90; $COCH_3$, 33.30.

Found: C, 54.20; H, 5.01; $COCH_3$, 33.60.

Hydrolysis of this compound with 7% sulfuric acid gave quercetin and the sugars rhamnose and glucose. This compound was identified as rutin by direct comparison with authentic rutin.

ISOLATION OF CHAKSINE AND ISOCHAKSINE.—The residue (1.25 g) obtained from the ethyl acetate-methanol (9:1) extract was chromatographed on silica gel (40 g) using ethyl acetate-methanol mixtures. Two alkaloids, chaksine (80 mg) and isochaksine (55 mg), were obtained from the eluates. The properties of these compounds were identical with fractions I and II obtained from the roots. Their identity was confirmed by direct comparison with authentic samples.

ACKNOWLEDGMENTS

The authors thank the Head of the Department of Chemistry for the elemental and spectral data, the University authorities for facilities, and the University Grants Commission for the Fellowship (JVLNS).

Received 5 May 1978.

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