ISOLATION OF CAFFEINE AND THEOPHYLLINE FROM RIPE TEA SEEDS

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At the present time, the seeds of the tea plant are being studied intensively for their saponin content. There are statements that the theasaponins exhibit antiexudative and anti-inflammatory activity [1-4]. It was known from previous information in the literature that the skin of the unripe fruit of the tea plant contains caffeine, but this base had not been detected in the seeds [5].

The hull of tea seeds is a waste of the production of theasaponin and amounts to 30% of their weight.

We have investigated this waste material for its content of purine compounds, including theophylline. In order to isolate the purine bases, the comminuted raw material was first wetted with an 8% aqueous solution of ammonia, and the alkaloids were extracted with chloroform [6], giving the total purine bases. When this total material was chromatographed in a thin layer of silica gel LS 5/40 in the chloroform–ethanol (9:1) system followed by visualization with a modified Dragendorff reagent (the Munier reagent) [7], one spot was detected, with R_f 0.58 (I), which was present at the level of a caffeine marker. When the same material was chromatographed on plates coated with active alumina (L 5/40 neutral) in the chloroform–ethanol (9:1) system and the zones were revealed with a reagent consisting of a solution of potassium iodide and crystalline iodine in concentrated acetic acid [7], a second spot with R_f 0.35 (II) was detected. The isolation of this substance preparatively gave a yellow crystalline base, optically inactive, with mp 268-270°C (water). A comparison with literature information and the performance of the chromatography of a mixed sample of (II) with an authentic sample of theophylline permitted us to consider that base (II) was the alkaloid theophylline, this being the first time that it has been detected in tea seed hulls.

The gas-liquid analysis of the fractions obtained from the tea seed hulls confirmed the presence of caffeine in them. The analysis was performed on a chromatograph of the Tsvet-100 series with a flame-ionization detector. The column (3 m × 3 mm) was filled with Chromaton NAW (0.12-0.16 mm fraction) impregnated with polyphenylmethylsiloxane (PMS-4). The mixture to be analyzed was dissolved in acetone. For quantitative identification, standard caffeine was added to the material under investigation. As a result, it was established that the peak recorded on the chromatogram at 36 min 32 sec did correspond to caffeine. The caffeine that we isolated consisted of optically inactive white silky crystals with mp 235-237°C (water). The UV spectrum taken in chloroform showed an absorption maximum at 273 nm (log ε 3.99), which is characteristic for purine compounds. The IR spectrum showed absorption bands at 3120-3130 (>NH), 1700 (C=O), 980, 761, and 750 cm^{-1} — deformation vibrations of a purine ring. The determination of the melting point of a mixture of the substance that we had isolated with an authentic sample of caffeine gave no depression.

Thus, by the investigation performed we have shown for the first time the presence of caffeine and theophylline in ripe tea seed hulls.

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