EXTRACTION AND FRACTIONATION:

EXPERIMENTAL

EXTRACTION AND FRACTIONATION:

Fresh flowers of J. mimosifolia collected from Kolli hills of Namakal district during March were extracted with 80% MeOH (4x500 ml)under reflux. The alc. extract was concentrated in-vacuo and the aq. concentrate was successively fractionated with benzene (3x250 ml), peroxide free EtOAc (4x250 ml). The benzene and EtOAc fractions did not yield any isolable material.

The EtOAc fraction was concentrated in-vacuo and left in an ice chest for 2 days. A yellow solid that separated was filtered and studied. It was recrystallized from MeOH when it afforded yellow crystals, m.p.229-30°C (yield 0.1%). It was freely soluble in EtOAc and MeOH and sparingly soluble in water. It gave an olive green colour with alc.FeCl₃, deep pink colour with Mg-HCl, yellow colour with NaOH and appeared deep purple under UV that turned yellow on exposure to NH₃. It did not answer the Horhammer-Hansel test but responded to Wilson’s boric acid, Gibb’s and Molisch’s test. The pigment had λ values as indicated in Table-I and had λ max 257, 269sh, 299sh, 362; (+NaOMe) 272, 327, 409sh; (+AlCl₃) 275, 303sh, 430; (+NaOAc) 271, 320sh, 372; (+NaOAc/H₃BO₃) 265, 300 sh, 372. The 1H- and 13C-NMR of the glycoside were appended.

Hydrolysis of the glycoside:

To a solution of the glycoside (0.1g, 0.2 m mole) in hot aq. MeOH (10 ml, 50%) and an equal volume of H₂SO₄ (10%) was added and the mixture was refluxed at 100°C for 2 h and the hydrolytic products were identified as described below.

Identification of the aglycone:
The EtOAc fraction from the hydrolysate was concentrated in vacuo and left in an ice chest for about a week. A yellow solid that separated was filtered and studied. It came out as pale yellow needles m.p.316-18°C on recrystallisation from MeOH.
It was soluble in organic solvents and sparingly in hot water. It gave a red colour with Mg-HCl, olive green with NH₃ and NaOH, yellow solution with a pale green fluorescence with conc. H₂SO₄ and appeared yellow under UV and UV/NH₃. It answered Wilson’s boric acid, Horhammer-Hansel and Gibb’s tests but did not respond to Molisch’s test. It had λₒ(HCl) nm 255, 269 sh, 301 sh, 370; (+NaOMe) 247 sh, 321 (dec); (+AlCl₃) 272, 304 sh, 333, 458; (+AlCl₃/HCl) 265, 301 sh, 359, 428; (+NaOAc) 257 sh, 274, 329, 390; (+NaOAc/H₁BO₃) 262, 304 sh; 388 and had RF values as depicted in Table I-1. The ‘H and 13C-NMR of the flavonol were appended. It was identified as quercetin and the same was confirmed by co-PC and mixed-PC and m.m.p with an authentic sample of quercetin from Physalis minima.

Identification of the sugar:(glucose)
The aq.solution from the above hydrolysate was neutralized with BaCO₃ and filtered. The concentrated filtrate on chromatographic examination (PC) gave RF values corresponding to those of glucose. The running properties of the glycoside were in favour of a monoside. The identity of the sugar was also confirmed by direct comparison with an authentic sample of glucose.

### Table I-2

<table>
<thead>
<tr>
<th>Compound</th>
<th>C₁</th>
<th>C₂</th>
<th>C₃</th>
<th>C₄</th>
<th>C₅</th>
<th>C₆</th>
<th>C₇</th>
<th>C₈</th>
<th>C₉</th>
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<th>C₂₃</th>
<th>C₂₄</th>
<th>C₂₅</th>
<th>C₂₆</th>
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</thead>
<tbody>
<tr>
<td>Isoquerctrin (from literature) (gppm)</td>
<td>156.5</td>
<td>133.7</td>
<td>176.7</td>
<td>161.3</td>
<td>98.8</td>
<td>164.2</td>
<td>93.6</td>
<td>156.5</td>
<td>104.2</td>
<td>121.4</td>
<td>115.3</td>
<td>144.8</td>
<td>148.5</td>
<td>116.5</td>
<td>121.6</td>
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<tr>
<td>Glycoside (gppm)</td>
<td>156.385</td>
<td>133.383</td>
<td>177.518</td>
<td>161.303</td>
<td>98.724</td>
<td>164.164</td>
<td>93.568</td>
<td>156.385</td>
<td>104.055</td>
<td>121.666</td>
<td>115.274</td>
<td>144.868</td>
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</table>

### RESULTS AND DISCUSSION

The fresh flowers of J.mimosifolia have been found to contain isoquerctin. The UV spectrum of the glycoside showed two major absorption peaks at 362 nm (band-I) and 257 nm (band-II) showing a flavonol skeleton. A bathochromic shift of 39 nm was noticed in the glycoside and +18 nm noticed in case of the aglycone on the addition of HCl. The presence of an ortho di hydroxyl group in both. A bathochromic shift of 39 nm was noticed in the glycoside as well as its aglycone did not exhibit any in tense UV fluorescence, ascertaining the presence of a free 5-OH group in both. The absorption peaks and a shoulder indicating a free 5-OH group in both. The presence of a free 4′-OH group. The AlCl₃ (band-II) showing a flavonol skeleton. A bathochromic shift of 39 nm was noticed in the glycoside as well as its aglycone showed 3 absorption peaks and a shoulder indicating a free 5-OH group in both. The presence of a free 4′-OH group.

**REFERENCES**