Click www.quattroprep.com for more information

Separation of Phenylpropanoid and Iridoid Glycosides from roots of Stachytarpheta cayennensis (Rich.) Vahl, by High Speed Countercurrent Chromatography

Gilda G. Leitão[1], Pierre André de Souza[2], Anselmo A. Moraes[2] and Les Brown[3]

[1] Núcleo de Pesquisas de Produtos Naturais (Natural Products Research Centre), Federal University of Rio de Janeiro, Rio de Janeiro, Brazil

[2] Chemistry Department, Federal Rural University of Rio de Janeiro, RJ, Brazil

[3] AECS, PO Box 80, Bridgend, Mid Glamorgan, , South Wales, UK, CF314XZ

In the beggining there was the Craig.....

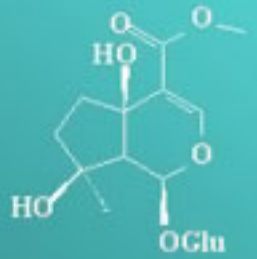
...and then ...the HSCCC era..

Stachytarpheta cayennensis

- The roots of this plant are used in Brazilian Folk Medicine for rheumatism, and back pain.
- *Topical application of the macerated leaves and roots – also recommended to treat sore skin wounds*

The Family Verbenaceae and the Genus Stachytarpheta

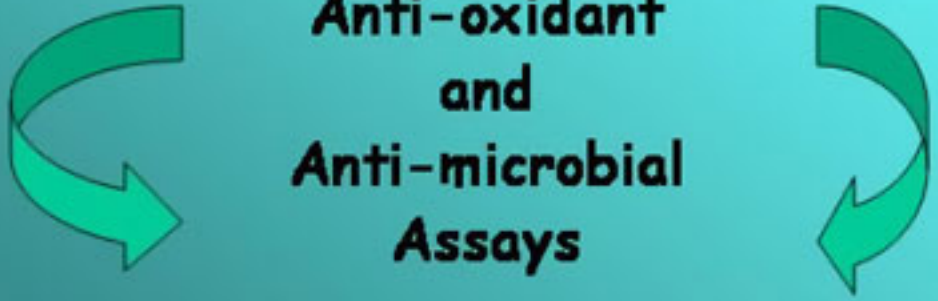
- Verbenaceae are known to be rich in both phenylpropnoid and iridoid glycosydes
- The Genus *Stachytarpheta* – rich in iridoids mainly of the ipolamiide type



Ipolamiide

Stachytarpheta cayennensis

Crude Ethanolic Extract from the Roots



the Ethyl Acetate extract afforded the best results in the two assays!!

Gradient Elution in CCC...

...can be very useful when samples have a wide range of polarities to be covered...

...*which is obtained when* the gradient progressively increases the elution strength of the mobile phase.

The choice of a proper solvent system for a gradient elution in CCC can be a difficult task!!!

Gradient Elution in CCC

Systems consisting of

EtOAc/1-BuOH/H_2O and EtOAc/2-BuOH/H_2O are very useful for both isocratic and gradient separation of rather polar compounds

A. Foucault, 1995

compounds that prefer water to ethyl acetate, but prefer butanol to water can be purified by using these two systems.

Optimization of the BuOH Ratio in the Solvent System $EtOAc:BuOH:H_2O$

Foucault 1995

A: $EtOAc:H_2O$ 1:1

B: $BuOH:H_2O$ 1:1

$EtOAc:BuOH:H_2O$ 1:X:1

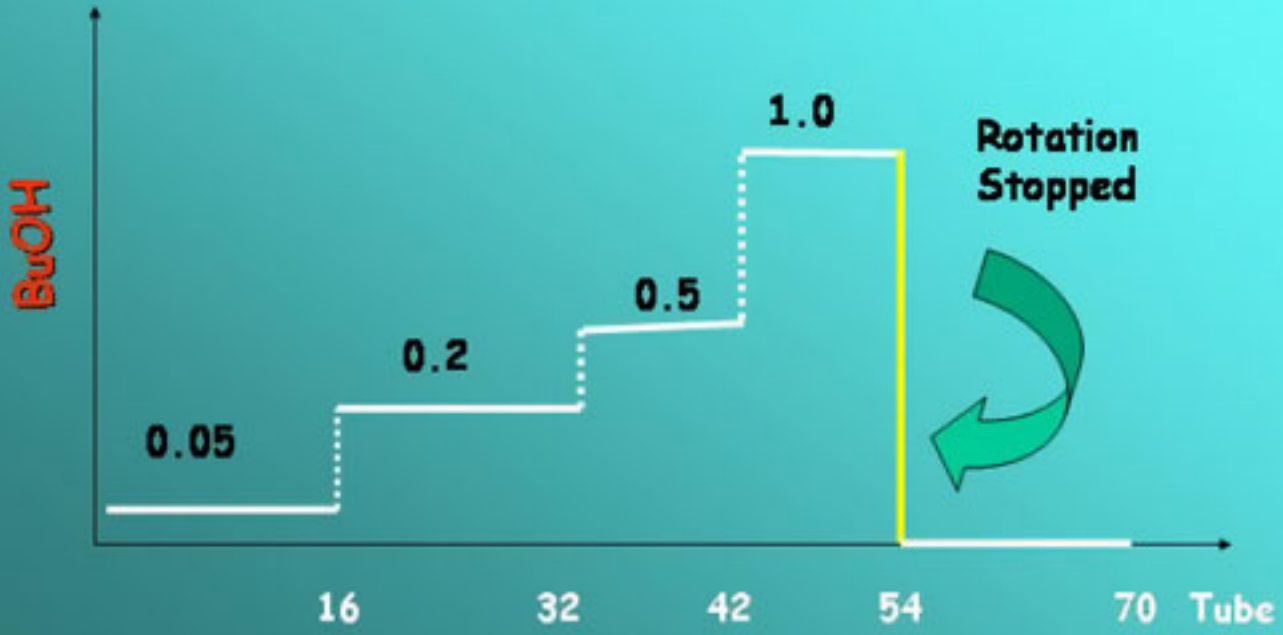
A: X= 0.2

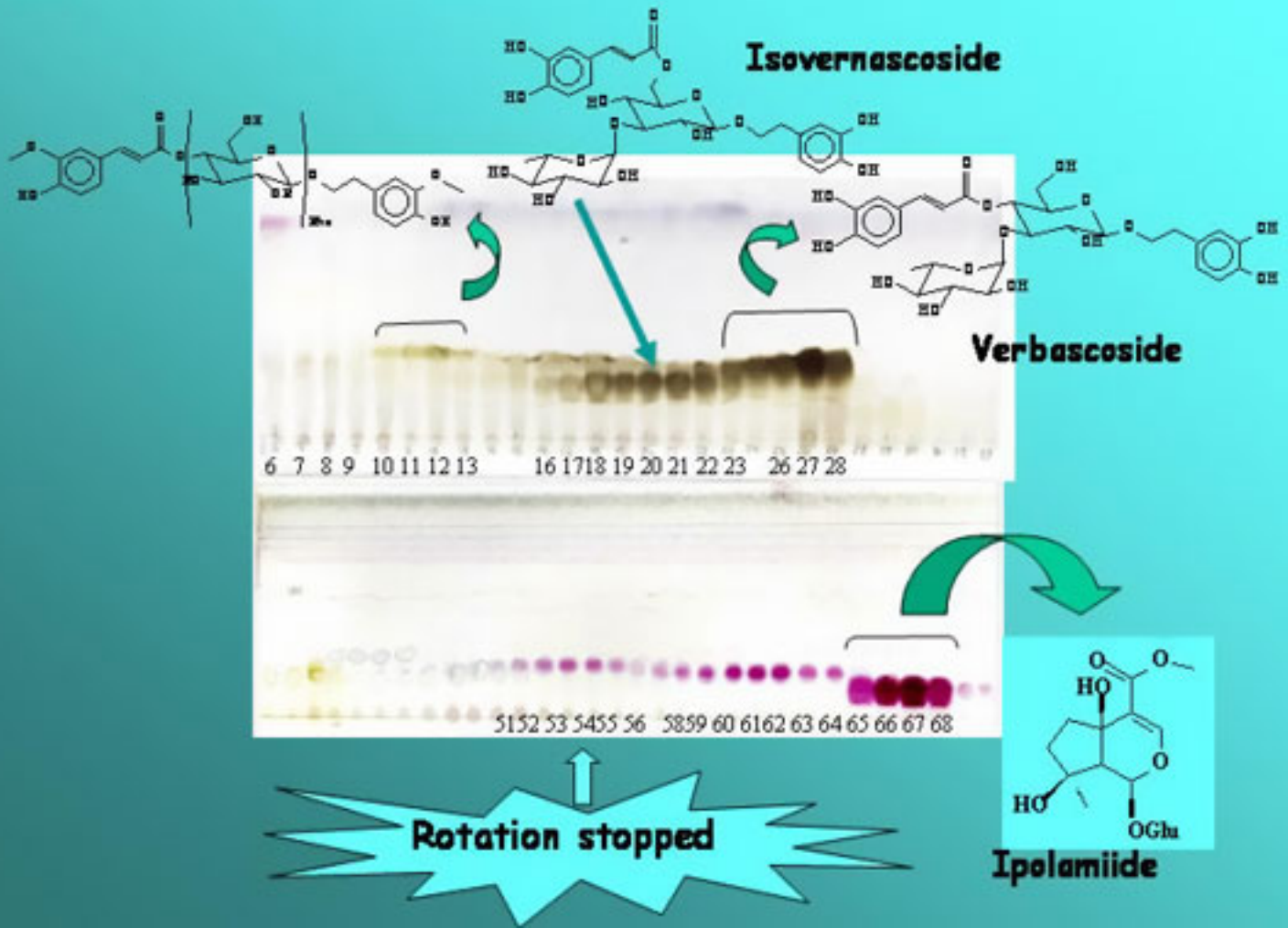
B: X=0.5

C: X=1.0

The Step-gradient was set as....

EtOAc:BuOH:H_2O 1:X:1, normal phase, 2ml/min., 850RPM, S_F = 78,7, 5ml fractions




Isovernascoside
Verbascoside
6 7 8 9 10 11 12 13 16 1718 19 20 21 22 23 26 27 28
5152 53 5455 56 5859 60 6162 63 64 65 66 67 68
Rotation stopped
Ipolamiide

Conclusions

- The sequential increase of BuOH in the organic phase of an EtOAc : BuOH : H_2O normal phase *CCC* solvent system, and wash-off of stationary phase allowed the isolation of the bio-active compounds from the ethyl acetate extract of the roots of *Stachytarpheta cayennensis*.
- These compounds covered a far wider span of polarities than could have been achieved by more conventional isocratic *CCC*.

Acknowledgements

- CNPq and FAPERJ
- CAPES