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Chemical constituents isolated from Mandevilla dardonoi (Apocynaceae)

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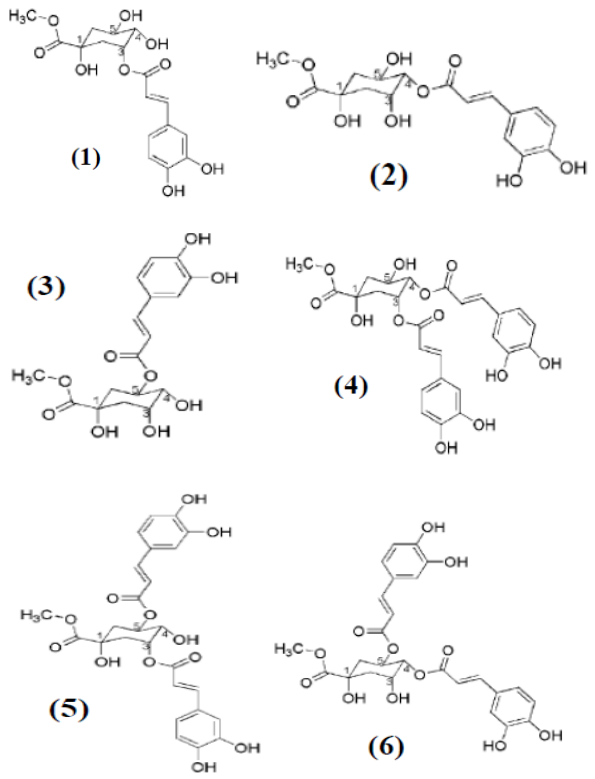
Figure 1: Isolated ethyl acetate phase compounds of the species *Mandevilla dardonoi*:



Abstract.
The aim of this study was to obtain bioactive compounds from *Mandevilla dardonoi* (Apocynaceae), in order to contribute positively to the discovery of new sources of active natural substances. Based on this, the compounds obtained from the ethyl acetate phase were isolated by medium pressure column chromatography and high performance liquid chromatography and identified by nuclear magnetic resonance (^{1}H) and electrospray mass spectrometry. It was possible to isolate 6 chlorogenic acids: 3-caffeoylquinic (3-ACQ), 4-caffeoylquinic (4-ACQ), 5-caffeoylquinic (5-ACQ), 3,4-dicaffeoylquinic 4-diACQ), 3,5-dicaffeoylquinic (3,5-diACQ) and 4,5-dicaffeoylquinic (4,5-diACQ) acids. The research contributed positively to the expansion of the knowledge about the genus *Mandevilla* and especially of the species *M. dardonoi*, which did not exist reports of phytochemical studies in the literature.
Keywords: *Mandevilla dardonoi*, chemical constituents, chlorogenic acids.

Introduction

Mandevilla Lindl. is among the 366 genera belonging to the family Apocynaceae and has about 190 species, of which 66 are reported in Brazil. Phytochemical studies performed with this genus showed different classes of compounds, such as steroids, terpenoids and pregnanes glycosides of interesting pharmacological profile [1,2]. Based on these data and considering that the genus above includes species present in the Northeast region, such as *Mandevilla dardonoi* (located in the states of Pernambuco and Paraíba), as well as the absence of phytochemical and pharmacological data on this

species, the interest arises to carry out a research that allows the obtaining of bioactive compounds, in order to contribute positively to the discovery of new sources of active natural substances.

Materials and Methods

The botanical material was collected in Serra do Jatobá (Serra Branca-Paraíba, 07◦29'00 "S, 36◦39'54 "), identified by Prof. Dr. José Iranildo Miranda de Melo, Department of Biological Sciences, State University of Paraíba and deposited in the Herbarium Manuel de Arruda Câmara (exsicata number 1663). The roots were dried in an air circulation oven at an average temperature of 40 ° C, ground in a mechanical mill and subjected to maceration with 95% EtOH, giving the ethanolic extract of *M. dardonoi* (EEBMd-303g). The extract was partitioned and 2.8 g of the AcOEt phase were chromatographed on a silica gel 60 column, giving 32 fractions. The fractions 22-30 (35mg) were pooled and subjected to semi-preparative High Performance Liquid Chromatography (mobile phase water acidified with 0.1% formic acid / methanol). Subsequently, the data were analyzed using the following methods: ^{1}H NMR (Brucker, 400 MHz, CD3OD) and Electrospray Mass Spectrometry, operating in negative mode (Bruker, microTOF II-Ion-Trap Amazon).

Results and Discussion

The analysis allowed the isolation and identification of 6 chlorogenic acids: (1) 3-caffeoylquinic (3-ACQ), (2) 4-caffeoylquinic (4-ACQ), (3) 5-caffeoylquinic) 3,4-dideofeoylquinic (3,4-diACQ), (5) 3,5-dicapheoylquinic (3,5-diACQ) and (6) 4,5-dicapheoylquinic (4,5-diACQ) (Figure 1). Table 1 shows the retention times (Rt) as well as the ^{1}H NMR chemical shift values of the isolated compounds.

Table 1: Retention time (Rt) and chemical shifts (δ_H in ppm) of ^{1}H NMR (400 MHz, CD_3OD) of 3-ACQ, 4-ACQ, 5-ACQ, 3.4 diACQ, 3,5 diACQ and 4,5 diACQ.

^{1}H	**3-ACQ (Rt = 30,5 min)**	**4-ACQ (Rt = 53,9min)**	**5-ACQ (Rt = 63,0min)**	**3,4diACQ (Rt = 90,8 min)**	**3,5diACQ (Rt = 96,5min)**	**4,5diACQ (Rt = 107 min)**
1	-	-	-	-	-	-
2	1,92-2,22 (m)	2,0-2,20 (m)	1,93-1,97 (m)	2,29-2,34 (m)	2,28-2,33 (m)	2,10-2,14 (m)
3	5,34 (ddd, *J*= 4,12, 9,28 e 8,56Hz)	4,31 (m)	4,07 (*sl*)	4,30 (ddd, *J*= 4,12; 8,0 e 8,2Hz)	5,40 (m)	4,39 (m)
4	3,73 (dd, *J* = 3,12 e 8,56Hz)	4,82 (m)	3,70 (m)	5,06 (dd, *J*=3,2 e 8,36Hz)	3,96 (dd, *J*=3,36 e 8,0Hz)	4,13 (dd, *J*= 3,04 e 9,0Hz)
5	4,20 (m)	4,31 (m)	5,37 (*sl*)	5,65 (m)	5,46 (m)	5,63 (ddd, *J*=5,0; 9,04 e 9,08 Hz)
6	1,91-2,22 (m)	2,0-2,20 (m)	2,09-2,13 (m)	2,06-2,08 (m)	2,11-2,20 (m)	2,22-2,34 (m)
7	-	-	-	-	-	-
1'	-	-	-	-	-	-
2'	7,07 (d, *J*=2,04Hz)	7,08 (d, *J* = 1,8 Hz)	7,04 (*sl*)	6,88 (d, *J*=2,5Hz)	7,05(d, *J*=2,8Hz)	4,02 (d, *J*=2,04 Hz)
3'	-	-	-	-	-	-
4'	-	-	-	-	-	-
5'	6,76 (d, *J*=8Hz)	6,81 (d, *J* = 8,12Hz)	6,78 (d, *J*=8,2Hz)	6,76 (d, *J*=8,24Hz)	6,77 (sl)	6,75 (d, *J*=2,3 e 8,2Hz)
6'	6,97 (dd, *J*= 2,04 e 8,0Hz)	6,99 (dd, *J* = 1,92 e 8,2Hz)	6,93 (d, *J*= 8,2Hz)	6,92 (d, J=2,0 e 8,48Hz)	6,97 (m)	6,91 (m)
7'	7,58 (d, *J*=16Hz)	7,63 (d, *J* = 15,92Hz)	7,56 (d, *J* = 15,8Hz)	7,59 (d, *J*=15,8Hz)	7,56 (d, *J*=14,8Hz)	7,51 (d, *J*=15,9 Hz)
8'	6,30 (d, *J*=16Hz)	6,36 (d, *J* = 15,88Hz)	6,28 (d, J= 15,2Hz)	6,30 (d, *J*=15,84Hz)	6,25 (d, *J*=6,25Hz)	6,19 (d, *J*=16Hz)
9'	-	-	-	-	-	-
1''	-	-	-	-	-	-
2''				6,81 (d,	7,07 (d, *J*=2,04	7,04 (d,

	J=2,6Hz)	Hz)	J=2,04Hz)
3''	-	-	-
4''	-	-	-
5''	6,74 (d, *J*=8,24Hz)	6,79 (sl)	6,77 (d, *J*=2,3 e 8,16Hz)
6''	6,90 (dd, *J*= 2,0 e 8,4Hz)	6,95 (m)	6,92 (m)
7''	7,55 (d, J=15,8Hz)	7,59 (d, *J*=14,88Hz)	7,63 (d, *J*= 16Hz)
8''	6,26 (d, *J*=15,85Hz)	6,34 (d, *J*=6,34Hz)	6,28 (d, *J*=16Hz)
9''	-	-	-

References: [3,4,5]

Mass spectrometry analysis was of great relevance for analyzing the intensities of the isomer fragments and the values were compared with studies by De Maria and Moreira (2004) and Jaiswal and Kuhnert [6, 7].

Conclusions

The research contributed positively to the expansion of the knowledge about the genus *Mandevilla* and especially of the species *M. dardonoi*, which did not exist reports of phytochemical studies in the literature.

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