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Supporting Information

Inhibition of stromelysin-1 by caffeic acid derivatives from a propolis sample from Algeria

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Table 1S NMR data of compounds **1-5** (500/125MHz; δ in ppm, J in Hz).

	1^a		2^a		3^b		4^a		5^a	
	δ_{H}	δ_{C}	δ_{H}	δ_{C}	δ_{H}	δ_{C}	δ_{H}	δ_{C}	δ_{H}	δ_{C}
1	-	168.1	-	169.8			-	169	-	172.0
2	7.15 s	72.0	7.15 d (2.9)	74.0			6.85 d (2.0)	73.0	6.80 d (2.0)	75.0
3	7.15 s	72.0	7.10 d (2.9)	73.7			6.10 d (2.0)	72.0	6.15 d (2.0)	72.0
4	-	168.1	-	169.8			-	173.0	-	nd
1'	-	127.6	-	129.3	-	127.0	-	126.5	-	127.0
2'	8.50 d (1.9)	115.8	8.55 m	117.7	8.23 d (2.0)	112.5	8.50 d (2.0)	115.0	8.40 d (2.0)	115.0
3'	-	146.7	-	148.5	-	146.0	-	146.0	-	146.5
4'	-	149.6	-	151.5	-	149.0	-	149.5	-	149.5
5'	8.20 d (8.2)	116.8	8.15 d (8.2)	118.6	7.98 d (8.5)	115.9	8.10 d (8.0)	116.0	8.10 d (8.0)	115.5
6'	8.45 dd (8.2, 1.9)	123.3	8.40 dd (8.2, 1.9)	125.1	8.10 dd (8.4, 2.0)	122.3	8.35 dd (8.0, 2.0)	123.0	8.30 dd (8.0, 2.0)	123.5
7'	8.90 d (15.9)	148.0	8.94 d (15.9)	149.9	8.71 d (16.0)	145.8	8.95 d (16.0)	148.0	8.90 d (16.0)	147.0
8'	7.65 d (15.8)	114.2	7.69 d (15.9)	115.8	7.39 d (16.0)	115.3	7.55 d (16.0)	114.0	7.55 d (16.0)	114.0
9'	-	166.7	-	168.5	-	169.8	-	167.0	-	167.0
1''	-	127.6	-	129.4						
2''	8.50 d (1.9)	115.8	8.55 m	117.7						
3''	-	146.7	-	148.5						
4''	-	149.6	-	151.5						
5''	8.20 d (8.2)	116.8	8.15 d (8.2)	118.6						
6''	8.45 dd (8.2, 1.9)	123.3	8.40 dd (8.2, 1.9)	125.2						
7''	8.90 d (15.9)	148.0	8.93 d (15.9)	150.1						
8''	7.65 d (15.8)	114.2	7.66 d (15.9)	115.9						
9''	-	166.7	-	168.6						
OCH ₃			5.05 s	55.3					5.00 s	53.3

^a in CD₃OD + CF₃COOD ; ^b in CD₃OD.

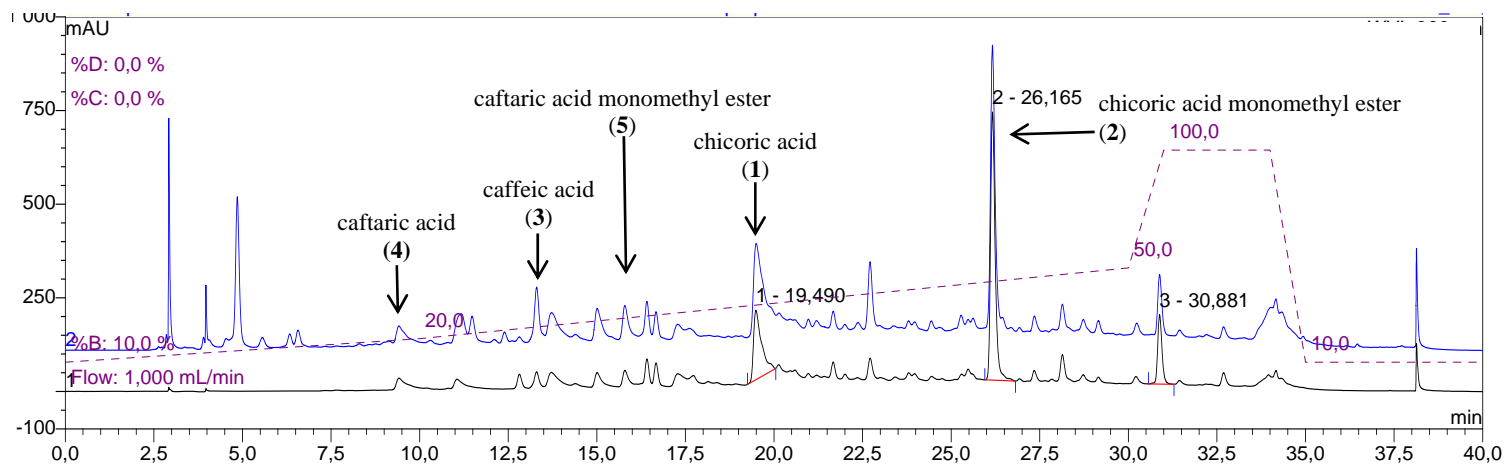


Fig. 1S The analytical HPLC chromatogram of fraction B₃. Conditions: RP-C₁₈ column (201 SPTM, 4.6 x 250 mm, 10µm, 90 A° Dionex, Vydac, France) using H₂O/0.0025% TFA as eluent A and CH₃CN as eluent B at a flow rate of 1 mL/min; an increasing linear gradient of solvent B starting at 10% B up to 20% B in 10 min, then to 50% B in 20 min; detection: 280 (in blue) and 325 (in black) nm. 5 mg were dissolved in 1 ml of H₂O-CH₃CN (90:10) and 25 µL were injected.

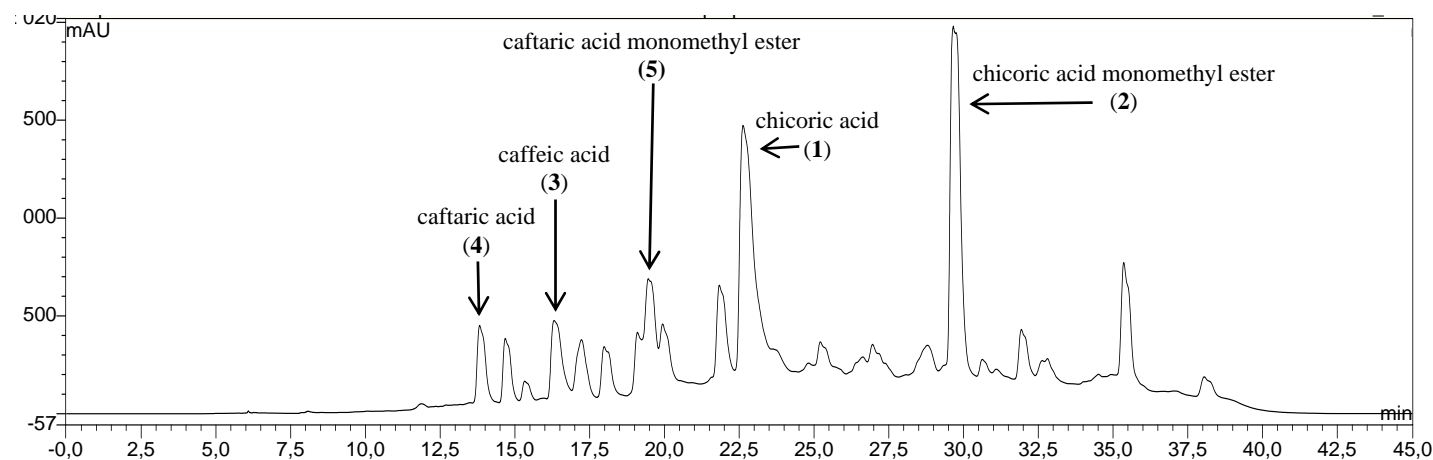


Fig. 2S Semi-preparative HPLC chromatogram of fraction B₃. Conditions: RP- C₁₈ column (Thermo Electron Corporation Hyperprep HS, 21.2 x 250 mm, 10 µm) using H₂O/0.0025% TFA as eluent A and CH₃CN as eluent B at a flow rate of 5 mL/min; an increasing linear gradient of solvent B, from 10% of B up to 50% in 45 min; detection: 325 nm; injected amount: 10 mg in 240 µL of H₂O-CH₃CN (90:10).