Electronic Supplementary Material (ESI) for Analytical Methods.

Electronic Supplementary Material

Development of a low pressure chromatographic flow system for monitoring biodegradation of ofloxacin and ciprofloxacin

Inês C. Santos[a], Raquel B. R. Mesquita[a,b], Catarina L. Amorim[a], Paula M. L. Castro[a], António O. S. S. Rangel[*a]

[a] Universidade Católica Portuguesa, CBQF - Centro de Biotecnologia e Química Fina – Laboratório Associado, Escola Superior de Biotecnologia, Rua Arquiteto Lobão Vital, Apartado 2511, 4202-401 Porto, Portugal

[b] Laboratory of Hydrobiology, Institute of Biomedical Sciences Abel Salazar (ICBAS), Universidade do Porto, Rua de Jorge Viterbo Ferreira n.º 228, 4050-313 Porto, Portugal

[*] Tel.: +351 225580064; Fax: +351 225090351; e-mail address: arangel@porto.ucp.pt

Table S1 Flow injection low pressure chromatography methods using a monolithic column; LOD, limit of detection; RSD, relative deviation

Analyte(s)	Column	Mobile phase	Sample pre-treatment	Detection	Dynamic range	LOD	RSD, %	Determination Rate (h^{-1})	Sample	Reference
Vitamin A	C-18 Sep-Pak/ C18	65:35 MeOH:0.075 M acetate buffer pH 5.0	-	Electrochemical	6 - 180 ng	300 pg	3.8	n. g.	Multi-vitamin preparation	11
Immunoglobulin	Protein A immobilized to a activated Fractogel	Buffer	-	Compared to radial immune-diffusion	4 - 8 mg mL^{-1}		<3	6	Biological samples	12
Inorganic Arsenic	Anion exchange cartridge and C18 cartridges	20 mM phosphate buffer pH 7	SPE	Hydride generation atomic fluorescence	1 - 10 ng mL^{-1}	0.2, 0.4 ng mL^{-1}	-	40	Water	13
Hemoglobin	DEAE-Sephadex A-50 anion exchange resin	0.05 M Tris-HCl buffer pH 6.5, 7.5, 8.5	Dilution	Spectrophotometry (Vis)	1.2 - 20.7%	-	<4.6	1	Blood	14
Cadmium, lead, zinc	Cation exchange column	3 mM Tartaric acid and 1 mM oxalic acid	Digestion and Preconcentration	Conductometry	6 - 60; 18 - 180; 60 - 600 µg	5, 3, 4 µg	3.1 - 5.1	1	Zinc ore	15
Antioxidants, preservatives, sweetener additives	C18 monolithic column	4% ACN: 10 mM phosphate buffer pH 6.0 for 200s changed to 30% MeOH	SPE	Spectrophotometry (UV)	0.03 - 80 µg mL^{-1}	0.01 - 1.60 µg mL^{-1}	0.93-2.82	9	Food and cosmetics	16
Opiate alkaloids and biogenic amines	C18 monolithic column	100 mM acetone	-	Chemiluminescence	-	$3x10^{-9}$ - $1x10^{-8}$ M; $2x10^{-8}$ - $5x10^{-7}$ M	<4.6	-	Human urine	17

Analyte(s)	Column	Mobile phase	Sample pre-treatment	Detection	Dynamic range	LOD	RSD, %	Determination Rate (h^{-1})	Sample	Reference
Aspartame, acesulfame-K, and saccharin	Quaternary amine ion exchange monolithic disc	0.03 M pH 9.0 Tris buffer, 0.4 M NaCl and 0.005 M NaClO4	SPE	Spectrophotometry (UV)	9.5 - 130; 2.2 - 600; 3.0 - 600 µg mL^{-1}	2.87; 1.0; 0.9 µg mL^{-1}	1.46; 0.08; 0.09	7	Food and soft drinks	18
Parabens	C18 monolithic column	12% ACN for 75s changed to 27% ACN	-	Chemiluminescence	$8.3x10^{-7}$ - $3.3x10^{-4}$ M	$1.9x10^{-8}$ - $9.9x10^{-5}$ M	3.5 - 6.2	24	Cosmetic samples	19
Methylparaben, ethylparaben, propylparaben, butylparaben	C18 monolithic column	12% ACN for 75s changed to 27% ACN	Extraction, preconcentration, filtration	Spectrophotometry (UV)	$1.6x10^{-5}$-$1.1x10^{-3}$; $3.7x10^{-5}$-$2.0x10^{-3}$; $3.9x10^{-5}$-$2.0x10^{-3}$; $6.0x10^{-5}$-$2.0x10^{-3}$ M	$4.8x10^{-4}$; $1.2x10^{-5}$; $1.2x10^{-5}$; $1.8x10^{-5}$ M	0.65; 1.2; 1.2; 1.8	n.g.	Commercial cosmetics	20
Chloride, bromide, iodide	Ion-exchange column	4.0 mM sodium carbonate	-	Spectrophotometry (Vis)	0.15 - 35; 0.03 - 5.0; 0.10 - 25.0 mg L^{-1}	0.011, 0.002, 0.023 mg L^{-1}	<2.9	10, 8, 4	Aquatic samples	21
Phenolic compounds	C18 monolithic column	Acetate buffer pH 3, 5% ACN	SPE	Chemiluminescence	0.06 - 230 µM	0.1, 4 µM	2.8 - 6.8	13	Healthcare products	22
Methylxanthines	C18 monolithic column	2:98 ACN:water	-	Spectrophotometry (UV)	$5x10^{-6}$ - $5x10^{-5}$; $2x10^{-5}$ - $1x10^{-3}$ M	$2x10^{-6}$, $2.9x10^{-6}$, $1.7x10^{-5}$ M	<6	10	Coffee brewed samples	23
Sugars and ethanol	Micro-guard cation H^+ cartridge	5 mM H_2SO_4	-	Spectrophotometry (UV)	up to 12 g L^{-1} and up to 2% (v/v)	2.3 g L^{-1}	<4	36	Vinification process and port wine	24
Niacin	C18 monolithic column	2.5 mM HCl	-	Amperometry	-	$7.9x10^{-7}$ M	<5	10	Coffee	25

DEAE, diethylaminoethyl; SPE, solid phase extraction.

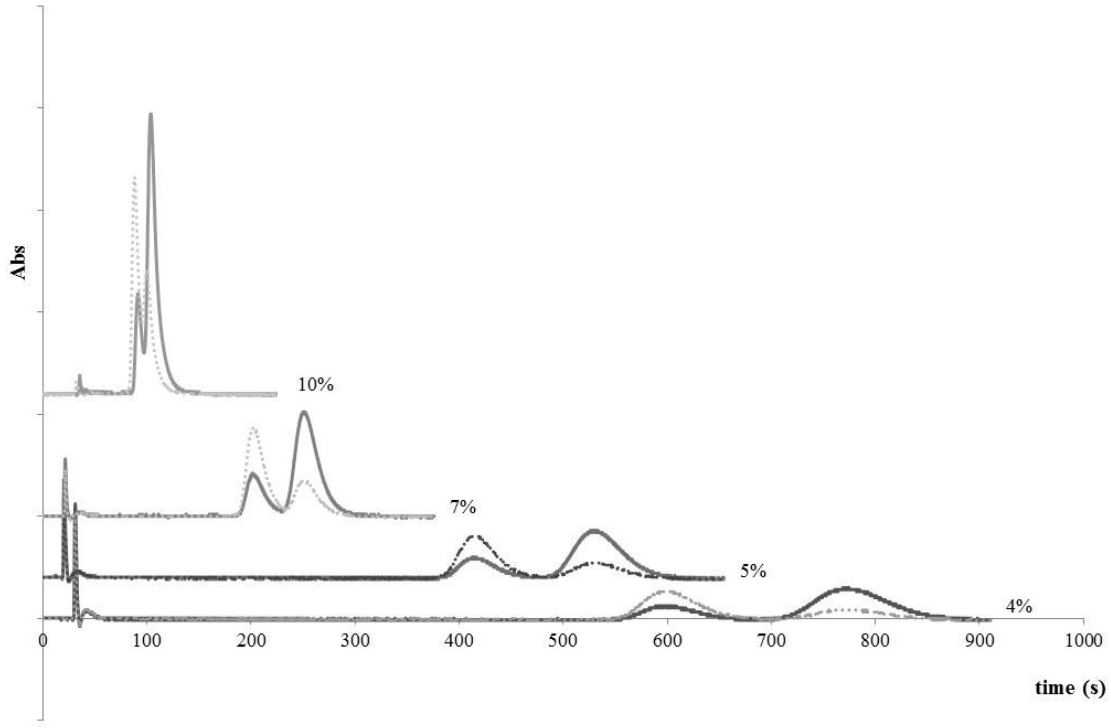

Fig. S1 Effect of mobile phase composition (ACN percentage) on peak elution using a 1 cm monolithic column at a flow rate of 0.8 mL min^{-1} and pH 3.0. Dashed line, ofloxacin; full line, ciprofloxacin.

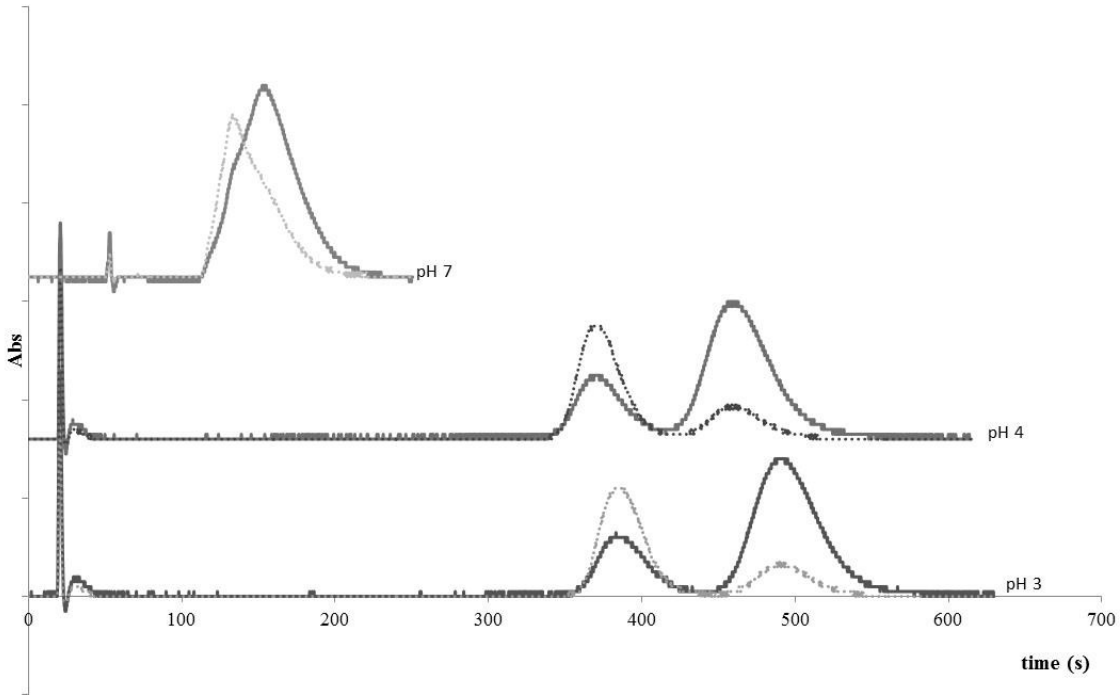

Fig. S2 Effect of mobile phase pH on peak elution using a 1 cm monolithic column at a flow rate of 0.8 mL min^{-1} and a carrier of 5:95 ACN:Phosphate buffer. Dashed line, ofloxacin; full line, ciprofloxacin.

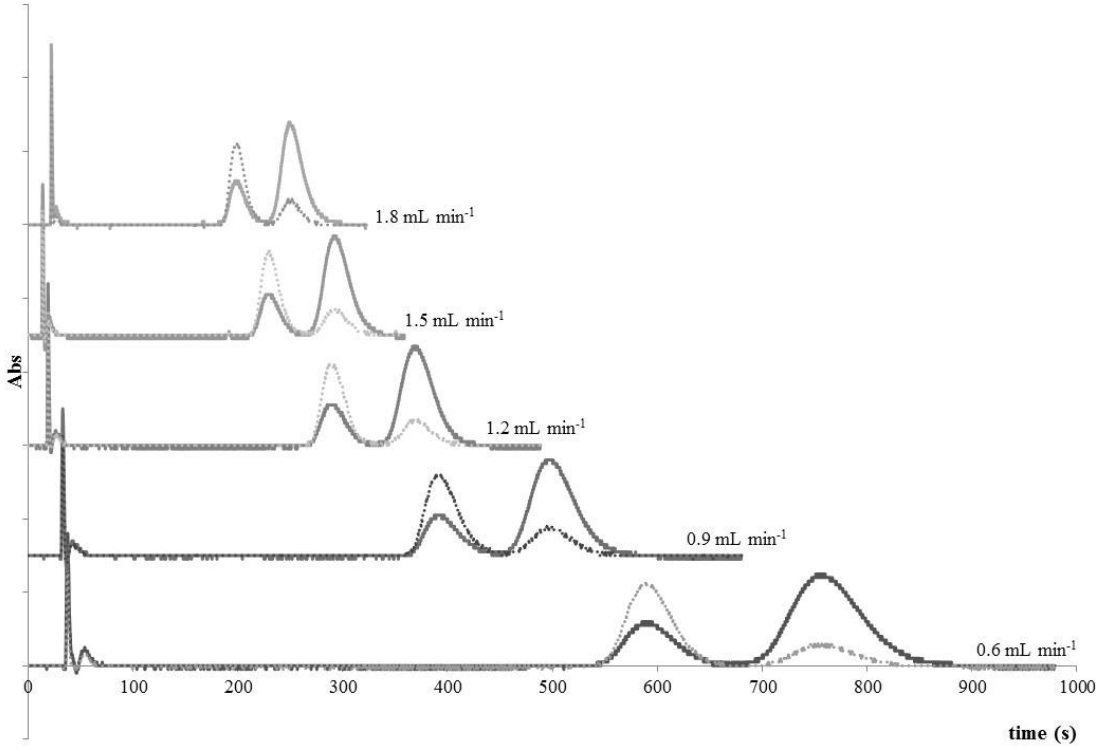

Fig. S3 Effect of flow rate on peak elution using a 1 cm monolithic column and a carrier of 5:95 ACN:Phosphate buffer pH 3.0. Dashed line, ofloxacin; full line, ciprofloxacin.

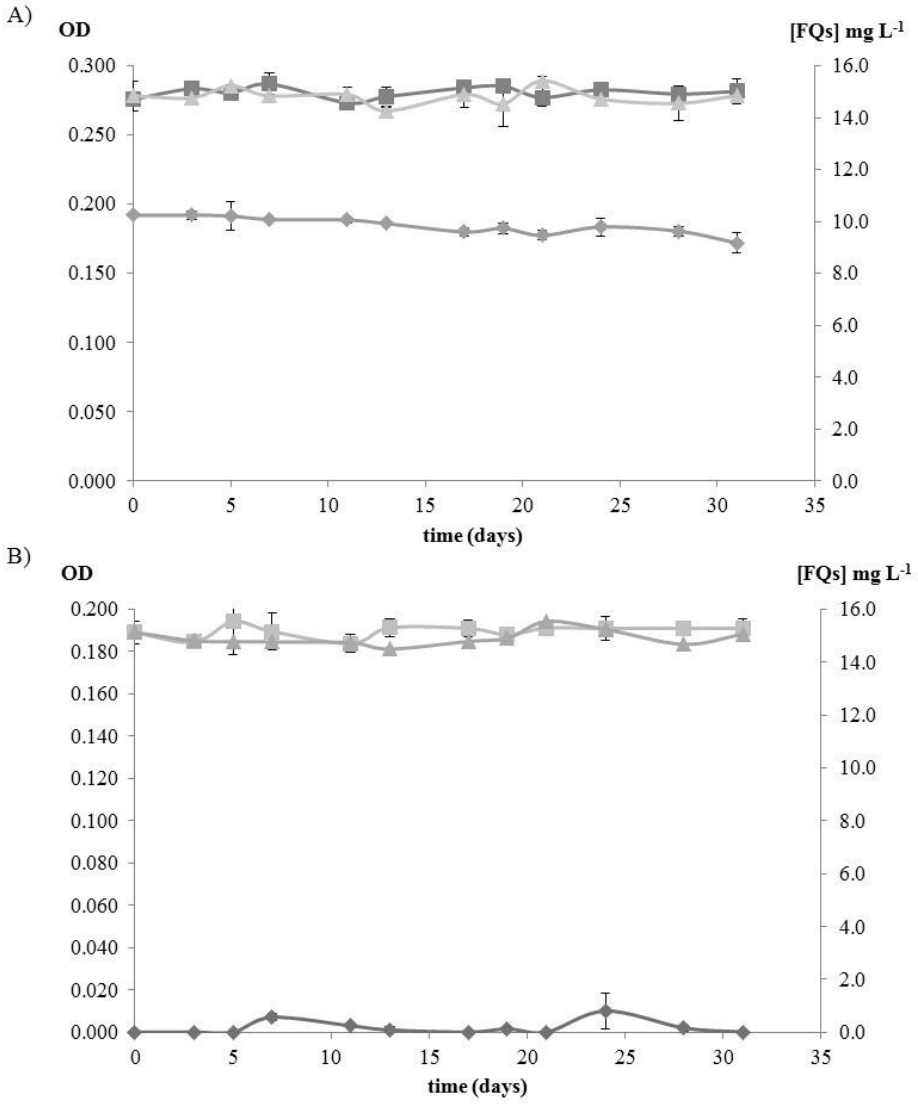

Fig. S4 Degradation profile of A) FQs inoculated with dead cells of *L. portucalensis* F11 and B) FQs in un-inoculated assay during 30 days: ♦ represents OD values, ■ represents OFLO values; ▲ represents CIPRO values. Data points represent the mean of duplicates and the error bars the standard deviation of the duplicates. OD measured at 600 nm.