

# SIMULTANEOUS DETERMINATION OF FIVE CLASSES OF ANTIBIOTICS IN VEGETABLE MATRICES BY SONICATION, SPE CLEANUP AND UPLC-MS/MS DETERMINATION

D. Tadic, V. Matamoros and J. M. Bayona

Department of Environmental Chemistry, Institute of Environmental Assessment and Water Research, Spanish Council for Scientific Research (IDAEA-CSIC), Jordi

Girona 18-26, E-08034, Barcelona, Spain

e-mail: dorde.tadic@idaea.csic.es



## Objective:

To develop an analytical method based on ultrasound-assisted extraction and solid-phase extraction followed by ultraperformance liquid chromatography-tandem mass spectrometry determination for the analysis of **12 antibiotics** in two different vegetable matrices, **lettuce leaves and tomato fruits**.

## Background:

Most of the **antibiotics** administered to livestock end up in farmland soil resulting in antibiotic contamination of soil and plants [1]. Quantification of antibiotics in crops can be difficult due to the complexity of vegetable matrices and the presence at relatively low concentrations in samples [2]. The method developed in this study was evaluated for the determination of 12 antibiotics from five classes such as: **fluoroquinolones** (i.e. ofloxacin, enrofloxacin, ciprofloxacin), **sulfonamides** (i.e. sulfamethaxine, sulfamethizole, sulfathiazole, sulfadiazine, sulfadimetaxine, sulfamethoxazole), **macrolides** (erythromycin), **lincosamides** (clindamycin) and **dihydrofolate reductase inhibitors** (trimethoprim).

## Method



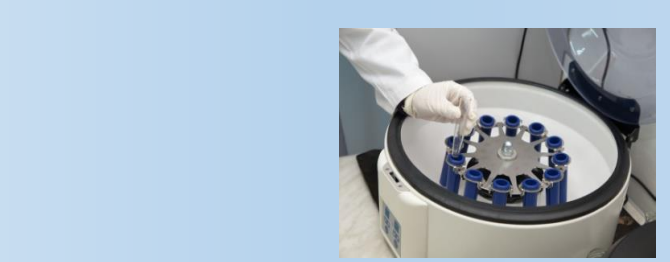
Sample homogenization – Spiking at 10 and 100 ng /g fresh weight



Freeze and thaw lysis



Ultrasound extraction – 2 x 10 min



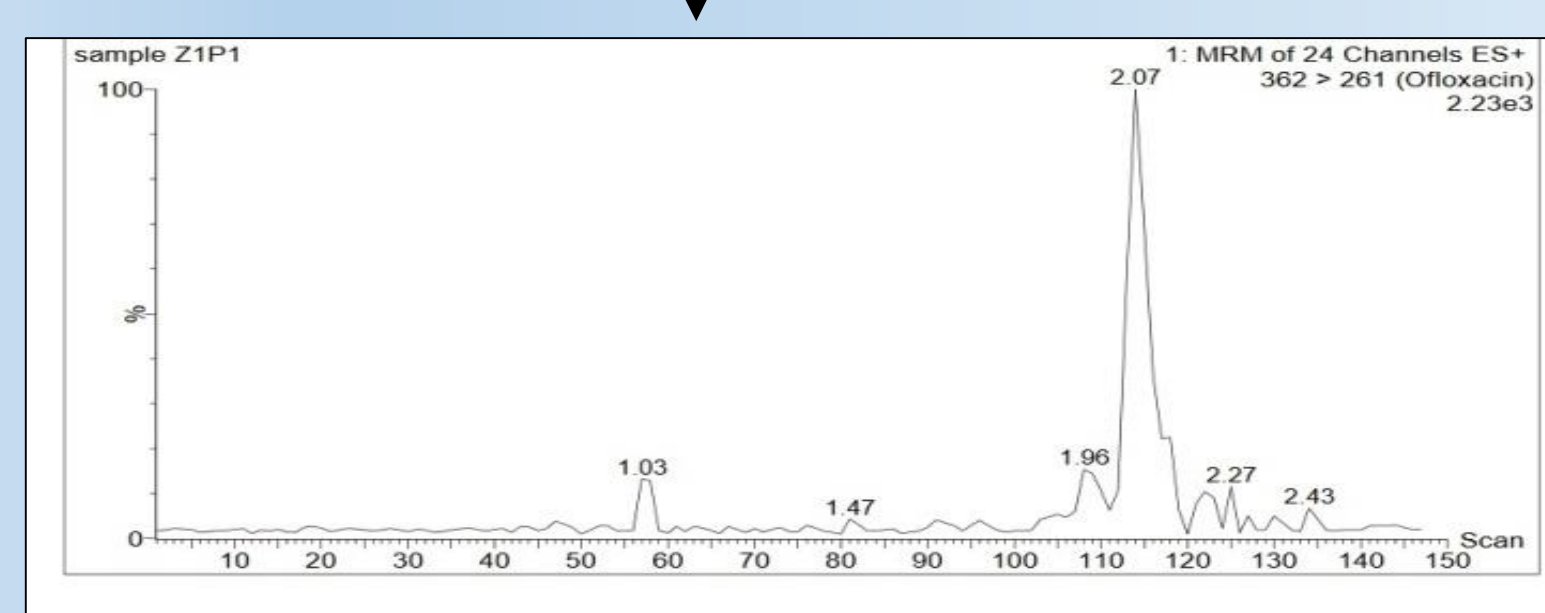
Centrifugation



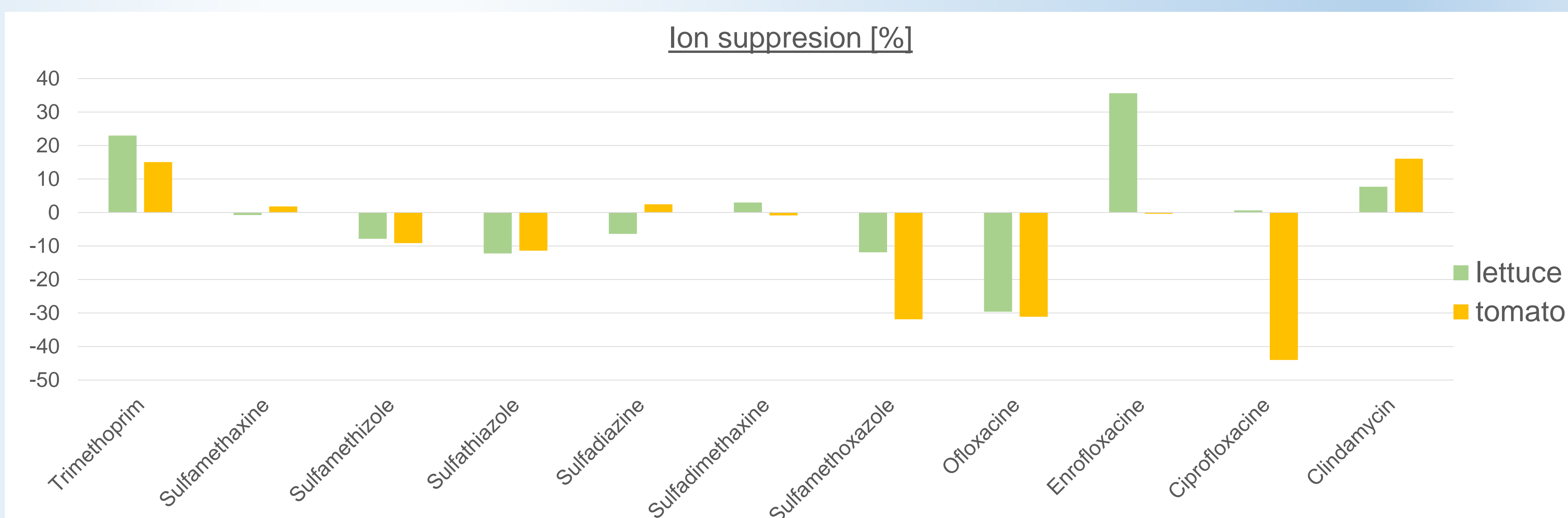
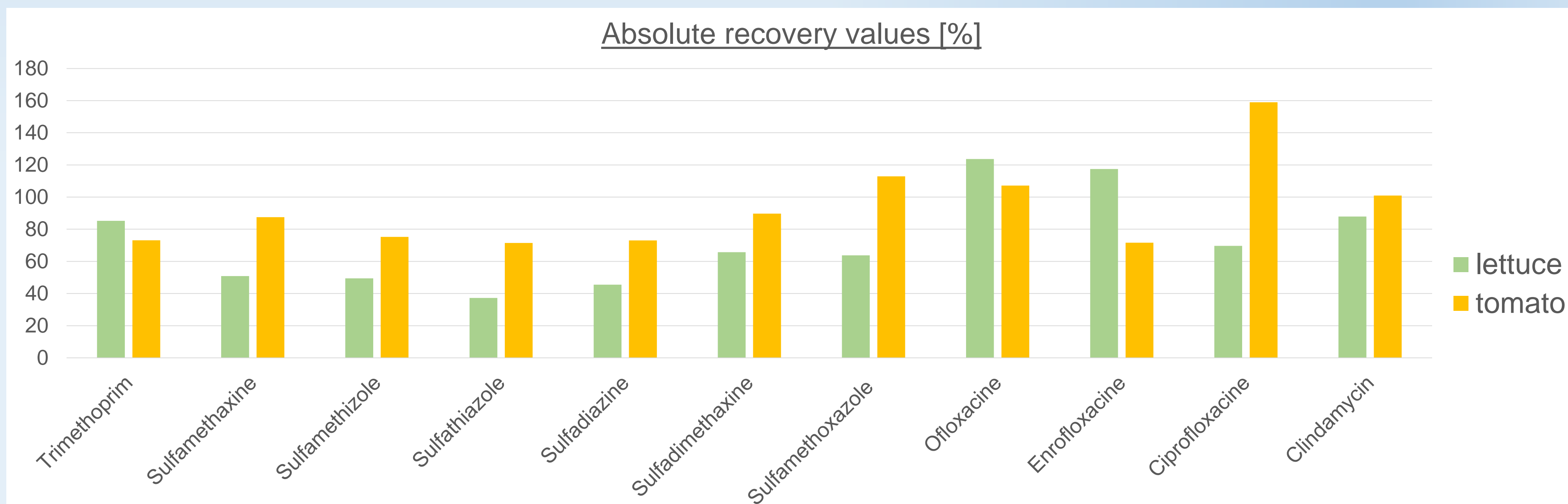
SPE cleanup – HLB cartridge



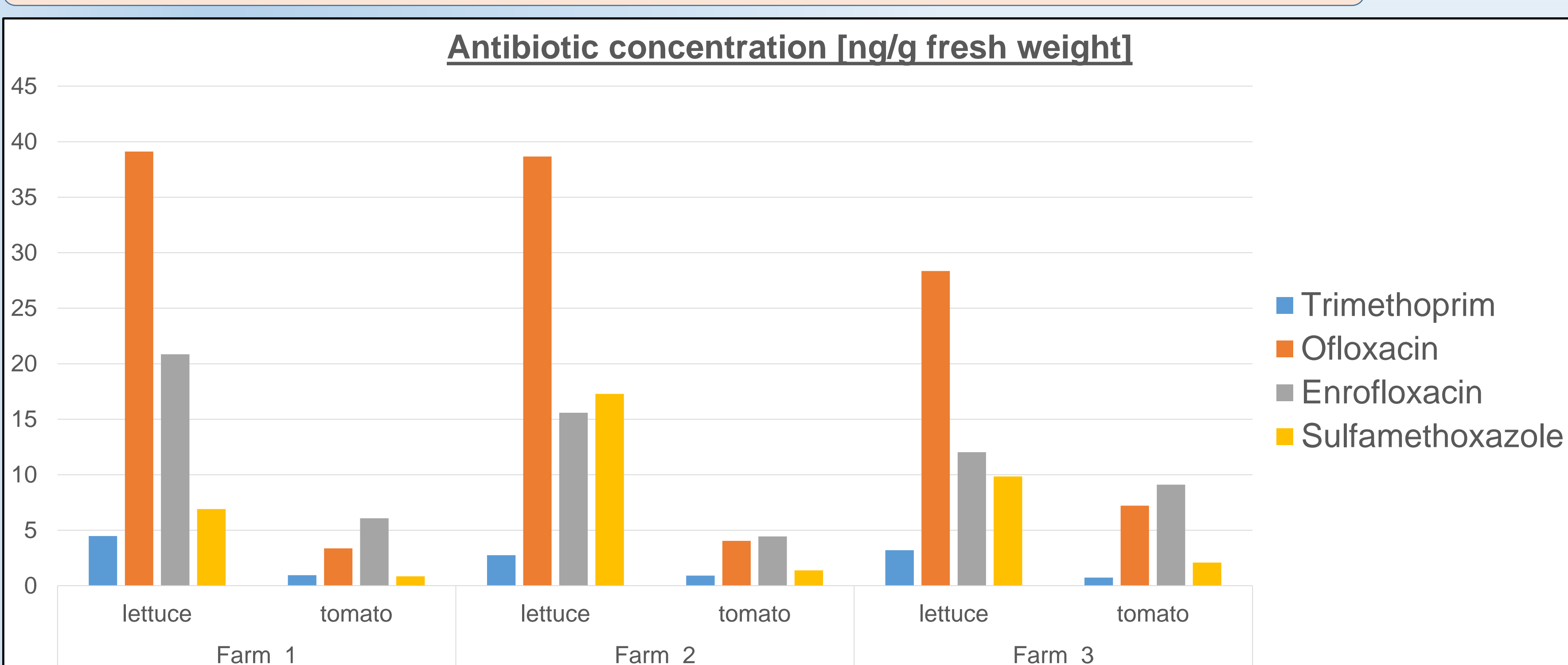
UPLC (ESI+) MS/MS – MRM mode



## Quality assurance & Quality control



## Occurrence of antibiotics in lettuce and tomato samples



Antibiotics	MLOD [ng/g fresh weight]		ILOD [ng/ml]
	lettuce	tomato	
Trimethoprim	0.06	0.08	0.01
Sulfamethaxine	0.24	0.60	0.02
Sulfamethizole	0.20	0.60	0.01
Sulfathiazole	10.00	0.60	0.02
Sulfadiazine	5.00	1.84	0.05
Sulfadimetaxine	0.06	0.30	0.01
Sulfamethoxazole	0.06	0.12	0.04
Ofloxacin	0.06	0.07	0.02
Enrofloxacin	0.07	0.69	0.05
Ciprofloxacin	0.83	1.60	0.71
Clindamycin	0.60	0.14	0.02

## Discussion and conclusion

Among the tested solvents (acetonitrile, acetonitrile + EDTA, aqueous phosphate buffer pH 7.5 and aqueous phosphate buffer pH 3) aqueous phosphate buffer pH 7.5 was selected as solvent for ultrasound extraction, because it provides the best selectivity and the lowest ion suppression. Strata-X solid-phase extraction (SPE) cartridges were applied for separation of antibiotics from complex matrices. Recoveries of SPE extracts eluted with methanol and methanol/ethyl acetate (1:1) were compared. The best quality parameters were obtained using 2 mL of methanol:ethyl acetate (1:1) for the cartridge elution and 1 mL of 5% methanol as washing eluent. Four antibiotics (trimethoprim, ofloxacin, enrofloxacin and sulfamethoxazole) were detected, at levels above MDLs in commercially available vegetable samples of lettuce leaves and tomato fruits grown in a periurban area irrigated with reclaimed water.

## Acknowledgement:



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## References:

- [1] M. Yea, M. Sunb, Y. Fengc, J. Wand, S. Xieb, D. Tianb, Y. Zhaoe, J. Wub, F. Hub, H. Lib and X. Jianga, J. Hazard. Mater., 309, 219-227 (2016).
- [2] I. Tlili, G. Caria, B. Ouddane, I. Ghorbel-Abid, R. Ternane, M. Trabelsi-Ayadi and S. Net, Sci. Total Environ., 563-564, 424-433 (2016).