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Development of a novel UHPLC-MS/MS method for the determination of ochratoxin A in tea

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Abstract

Tea is the most frequently consumed beverage worldwide. It is projected that by 2023 at around 7 million tons will be produced. Tea contains several compounds associated to promote wellbeing. Also, numerous chemical contaminants and natural toxins can be found in raw tea. Several studies have detected mycotoxins in the different types of tea (black, red, green), among them, *ochratoxin A* (OTA). OTA is considered within Group 2B (possibly carcinogenic to humans) by the International Agency for Research on Cancer. Although available for foods and feed products, there is no European Regulation for any mycotoxin content in tea.

Regarding OTA determination and quantification, the tea infusion is considered a real complex, but interesting matrix to be analyzed. The presence of concomitant compounds (polyphenols, caffeine, amino acids, and colored substances, etc.) might interfere during analysis. The development of sensitive, accurate, low-cost, quick, and eco-friendly methods has become a major challenge in the Analytical Chemistry field, a growing trend related to environmental awareness has emerged in recent years. In this context, dispersive liquid-liquid microextraction based on the solidification of a floating organic drop (DLLME-SFO) technique satisfies the general characteristics of green strategies. Nowadays, to establish an analytical methodology, not only the overall performance should be considered, but also the greenness assessment through a variety of metrics. Several green metrics can assess the sustainability of any analytical procedure, among them, the Analytical Eco-Scale, the Green certificate (GC), and the recently introduced Analytical GREEnness calculator (AGREE), are the most reported. An overall evaluation of these aspects will be revised in this presentation.

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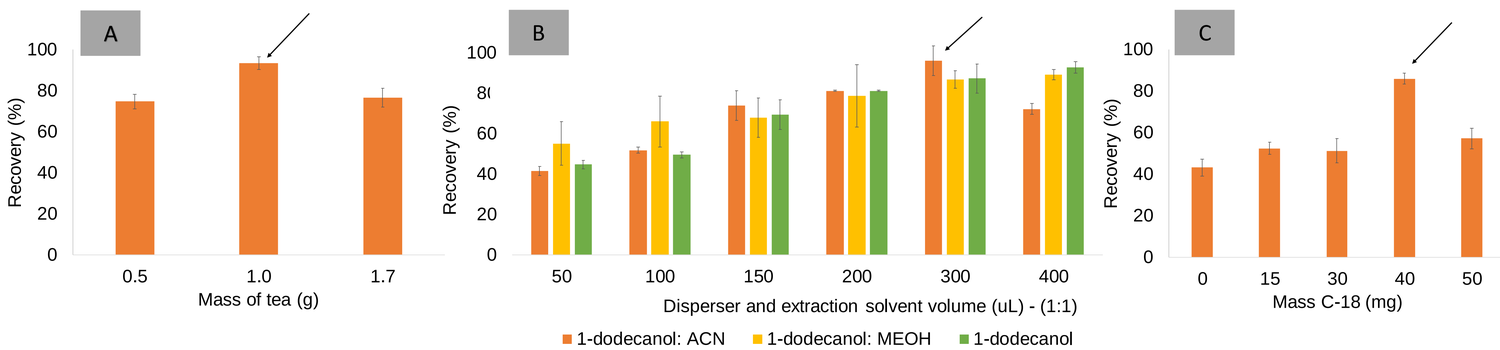
Results and discussion



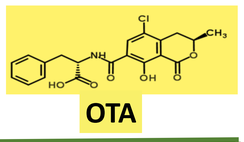


Table 1. Analytical figures of merit and recovery study

Figures of merit	
[1]LR (ng mL^{-1})	0,5 - 74
[2]LOD (ng mL^{-1})	0,5
[3]LOQ (ng mL^{-1})	1,4
Intra-day precision [4]RSD%, (n = 3)	6,5
Inter-day precision [4]RSD%, (n = 3)	7,8
r^2	0,9985

Recovery study for the analysis of spiked infusion tea samples after applying the proposed methodology

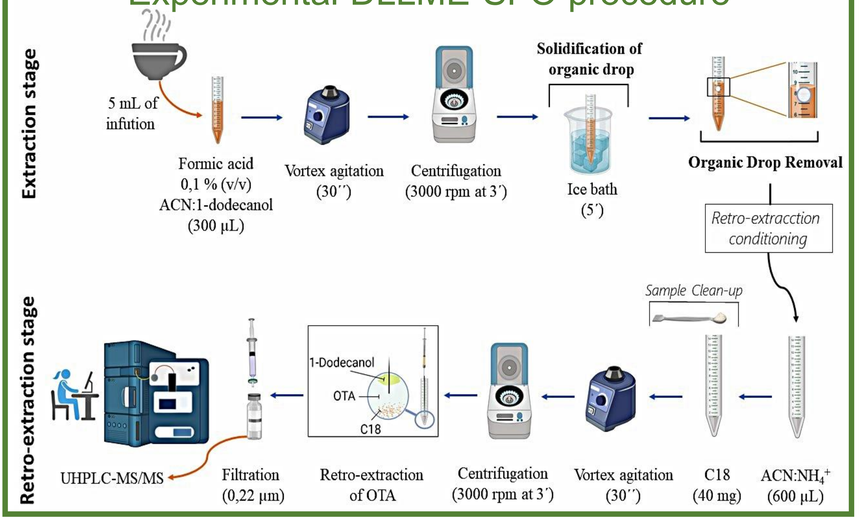
Sample concentration (ng mL^{-1})	Concentration Added (ng mL^{-1})	Concentration found (ng mL^{-1})	[5]RR (%)	RSD (%) n=3	[6]EF
N/D*	0	---	---	---	7
*	1	5,1	76,1	3,8	
*	5	31,9	95,6	5	
*	8	49,5	92,8	2,8	
*	10	64,5	96,7	4,2	

[1]LR: linear range, [2]LOD: Limit of Detection, [3]LOQ: Limit of Quantitation, [4]RSD: Relative Standard Deviation, [5]RR: Relative Recovery, [6]EF: Enrichment Factor, N.D.: not detected

Table 2. Application of the proposed methodology to different varieties of tea samples

Sample	Tea variety	Concentration Added (ng mL^{-1})	[1]EF	Concentration found (ng mL^{-1})	[2]RR (%)	[3]RSD (%)
Sample 1	Black	5	7	32,9	98,6	3,4
Sample 2	Green	5		33,9	101,7	0,8
Sample 3	Boldo	5		34,4	103,3	2,5
Sample 4	Herbal mix	5		30,0	90,0	3,7
Sample 5	Linden	5		29,8	89,3	3,9

Experimental DLLME-SFO procedure



Greenness profile procedure

Conclusions

In this work, the evaluation of the concentration levels of OTA in tea beverage samples was accomplished. Extraction and preconcentration steps through the DLLME-SFO strategy were developed. The obtained extract was analyzed by UHPLC-MS/MS. Interferences from the matrix were effectively reduced and, consequently, recovery increased from 43.18% ± 4.1% to 96.02% ± 2.54%. The validation assays were carried out by external and spiked samples calibration, with satisfactory recoveries. An adequate dynamic calibration range was obtained over a concentration interval between 0.5 and 70 µg mL^{-1} OTA. The capabilities of detection and quantification were 0.5 µg mL^{-1} and 1.4 µg mL^{-1}; respectively. Finally, the greenness evaluation of the developed methodology was assessed through the green certificate, the A classification was achieved. **Published by Cina, M., et *al.*, *Development of a novel UHPLC-MS/MS method for the determination of ochratoxin A in tea. Heliyon, 2021. 7(4): p. e06663.***

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