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Optimisation of an analytical method to determine phthalates acid esters in wastewater samples

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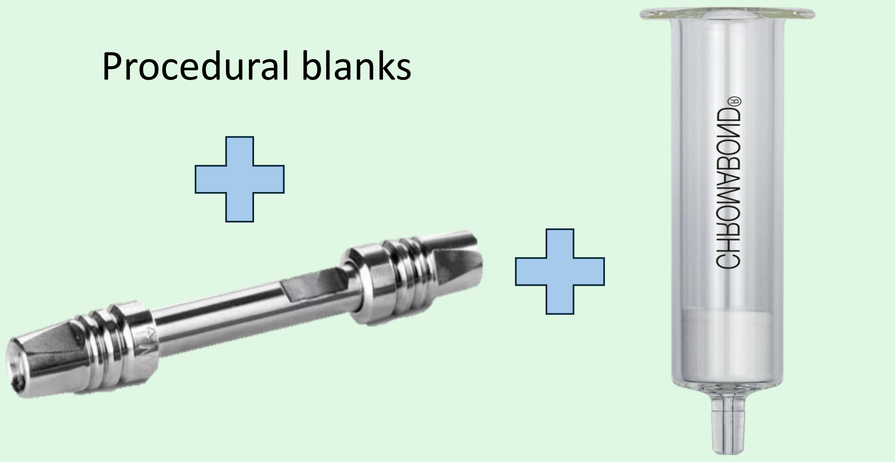
Introduction

Phthalates acid esters (PAEs) are synthetic plasticizers widely used in the industrial sector and consumer products. Their presence in several environments has already been confirmed and their potential endocrine-disrupting effects affecting growth or reproduction in biota (Net *et al*., 2015; Zhang *et al*., 2018). In this work, we developed and validated a methodology based on solid-phase extraction (SPE) coupled to high-performance liquid chromatography–tandem mass spectrometry (UHPLC-MS/MS) for the determination of six priority PAEs (BBP, DBP, DEHP, DNOP, DINP and DIDP) in wastewater samples. The developed method was then applied to samples from a conventional treatment plant (Arinaga) and a natural one (Tafira).

To avoid PAEs interferences

Procedural blanks



Isolator column Glass c18 cartridge

Experimental designs SPE

1st design 2^3

Vol. sample = 100 and 250 mL
pH sample = 4 and 9
Vol. extractant = 2,5 and 5 mL MeOH

+

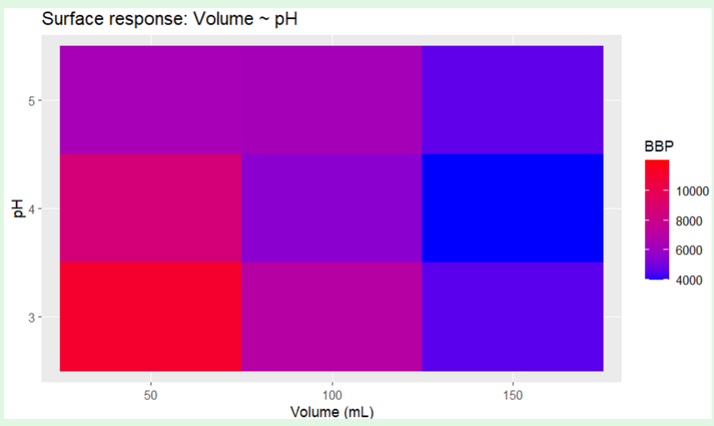
% recovery
ANOVA test p-value < 0,05

2nd design 3^2

Vol. sample = 50, 100 and 150 mL
pH sample = 3, 4 and 5
Vol. extractant = 5 mL MeOH

+

% recovery
ANOVA test p-value < 0,05



Optimal conditions: **50 mL of sample at pH 3 and 5 mL MeOH as extractant**

Analytical parameters

	LOD	LOQ	Absolute recoveries (%)			Relative recoveries (%)			Matrix effect (%)			Intra-day precisión (%)			Inter-day precisión (%)		
	µg·L^{-1}	µg·L^{-1}	10 µg·L^{-1}	50 µg·L^{-1}	200 µg·L^{-1}	10 µg·L^{-1}	50 µg·L^{-1}	200 µg·L^{-1}	10 µg·L^{-1}	50 µg·L^{-1}	200 µg·L^{-1}	10 µg·L^{-1}	50 µg·L^{-1}	200 µg·L^{-1}	10 µg·L^{-1}	50 µg·L^{-1}	200 µg·L^{-1}
BBP	**0,018**	**0,063**	**63,20**	**56,46**	**57,36**	**165,7**	**87,22**	**88,99**	**38,13**	**64,72**	**64,45**	**9,282**	**4,689**	**2,390**	**8,602**	**7,425**	**7,585**
DBP	**0,005**	**0,018**	**67,91**	**50,69**	**56,29**	**117,1**	**93,51**	**89,04**	**58,84**	**54,20**	**63,22**	**5,373**	**3,863**	**2,791**	**7,374**	**8,476**	**7,628**
DEHP	**0,010**	**0,034**	**98,86**	**69,54**	**79,08**	**123,1**	**78,89**	**84,58**	**86,04**	**88,67**	**92,34**	**6,801**	**4,509**	**2,613**	**18,56**	**2,424**	**3,199**
DNOP	**0,026**	**0,088**	**67,24**	**68,23**	**78,10**	**82,09**	**72,87**	**83,26**	**81,90**	**93,63**	**93,80**	**14,36**	**7,587**	**5,277**	**9,106**	**4,363**	**1,362**
DINP	**0,012**	**0,041**	**47,26**	**61,23**	**67,64**	**73,63**	**70,53**	**81,75**	**64,18**	**86,82**	**82,73**	**14,85**	**5,450**	**6,424**	**9,584**	**3,026**	**3,623**
DIDP	**0,008**	**0,027**	**42,30**	**69,74**	**83,32**	**83,40**	**72,21**	**80,28**	**50,71**	**96,58**	**103,7**	**14,99**	**8,454**	**4,327**	**5,394**	**5,109**	**2,950**

Samples results

			DBP	DEHP
Arinaga	May	**Inlet**	<LOQ	**5,018 µg·L^{-1}**
		Oulet	<LOQ	<LOQ
	June	Inlet	<LOQ	<LOQ
		Oulet	<LOQ	<LOQ
	July	Inlet	<LOQ	<LOQ
		Oulet	<LOQ	<LOQ
Tafira	May	Inlet	<LOQ	<LOQ
		Oulet	<LOQ	<LOQ
	June	Inlet	<LOQ	<LOQ
		Oulet	<LOQ	<LOQ
	July	Inlet	<LOQ	<LOQ
		Oulet	<LOQ	<LOQ

References

Net *et al*. Occurrence, fate, behavior and ecotoxicological state of phthalates in different environmental matrices. Environmental Science & Technology, 2015, 49(7), 4019-4035.

Zhang *et al*. Occurrence, distribution, and ecological risks of phthalate esters in the seawater and sediment of Changjiang River Estuary and its adjacent area. The Science Of The Total Environment, 2018, 619-620, 93-102.

Acknowledgments

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