

Supplementary material

Table S1. *Plackett-Burman Experimental Factorial Design*

Run	Factors under study							
	Type of dispersant solvent (A)	Volume of dispersant solvent mL (B)	Type of extractant solvent (C)	Volume of extractant solvent mL (D)	Use of US bath (E)	Salting out effect (F)	Sample volume mL (G)	pH modification (H)
1	ACN	3	CF	0.6	YES	NO	6	YES
2	ACN	3	DCM	0.3	YES	YES	6	NO
3	MeOH	1	DCM	0.6	YES	NO	3	NO
4	MeOH	3	CF	0.3	NO	YES	3	YES
5	MeOH	1	DCM	0.6	NO	YES	6	YES
6	MeOH	3	CF	0.6	YES	YES	3	NO
7	MeOH	1	CF	0.3	YES	NO	6	YES
8	ACN	1	CF	0.3	NO	NO	3	NO
9	MeOH	3	DCM	0.3	NO	NO	6	NO
10	ACN	3	DCM	0.6	NO	NO	3	YES
11	ACN	1	CF	0.6	NO	YES	6	NO
12	ACN	1	DCM	0.3	YES	YES	3	YES

Table S2. Factors influencing the extracting process of ITCs through a DLLME and list of experiments using a multivariate experimental model One-Factor.

Variables		Factors levels and its codification	
F1(A)	Volume extractant solvent (mL)	0.4	0.7
F2(B)	Type of extractant solvent	CF (-1)	DCM (1)
Factors to optimize			
Run	Volume extractant solvent (μ L)	Type of extractant solvent	
1	550	DCM	
2	550	DCM	
3	700	CF	
4	550	CF	
5	550	DCM	
6	475	CF	
7	400	DCM	
8	625	DCM	
9	475	DCM	
10	550	CF	
11	700	DCM	
12	400	CF	
13	400	CF	
14	400	DCM	
15	625	CF	
16	550	CF	
17	550	DCM	
18	700	DCM	
19	700	CF	
20	550	CF	

Table S3. *Summary of the influence of each factor on the response variable.*

FACTORS								
	A	B	C	D	E	F	G	H
Analytes	Type of dispersant solvent	Volume of dispersant solvent (mL)	Type of extractant solvent	Volume of extractant solvent (mL)	Use of US bath	Salting out effect	Sample volume (mL)	pH modification
Allyl ITC	-	1	-	0.7	-	-	-	-
Phenyl ITC	-	-	DCM	0.3	-	-	-	-
Sulforaphane	ACN	-	CF	0.7	-	-	-	NO
Indole-3-carbinol	-	-	-	0.7	-	-	-	-
Erucin	-	-	CF	0.7	-	-	-	NO

(-) It does not influence the extraction process for that analyte.

Table S4. Accuracy of the standard compounds determinates by the DLLME-HPLC-UV in the different Brassicaceae vegetables.

Standard Compound	Concentration ($\mu\text{g mL}^{-1}$)	% ACCURACY*								
		Broccoli	White cabbage	Red cabbage	Brussels sprouts	Radishes	Watercress	Rocket seeds	Cauliflower	Green mustard
Sulforaphane	20	93 (3.2)	87 (2.9)	95 (3.8)	82 (2.1)	113 (0.4)	74 (1.4)	91 (0.2)	89 (2.5)	78 (3.0)
	30	113 (2.7)	114 (0.4)	99 (3.7)	92 (3.7)	107 (2.7)	91 (2.9)	86 (1.3)	107 (2.5)	111 (0.1)
	50	95 (2.8)	98 (1.2)	96 (1.4)	102 (4.2)	99 (2.2)	95 (3.9)	91 (1.3)	107 (0.6)	97 (1.6)
Indole-3-Carbinol	20	109 (1.0)	112 (4.3)	107 (0.2)	113 (2.8)	95 (0.7)	107 (1.2)	93 (1.6)	102 (2.4)	108 (1.5)
	30	110 (0.5)	101 (1.5)	110 (1.1)	102 (1.1)	105 (0.2)	94 (5.8)	81 (4.8)	95 (5.5)	108 (4.1)
	50	87 (5.3)	91 (2.6)	85 (3.1)	92 (2.1)	88 (3.7)	96 (3.9)	84 (4.7)	86 (1.9)	97 (2.4)
Allyl ITC	20	77 (3.3)	70 (0.8)	87 (0.3)	79 (1.3)	71 (0.9)	82 (1.4)	82 (7.7)	75 (0.8)	87 (1.4)
	30	78 (2.0)	81 (1.5)	88 (1.5)	77 (2.8)	95 (2.7)	88 (1.8)	88 (3.3)	95 (0.1)	93 (0.4)
	50	97 (1.1)	94 (1.1)	91 (2.4)	102 (1.1)	89 (0.2)	88 (0.6)	78 (5.4)	87 (2.1)	103 (2.8)
Erucin	20	105 (2.3)	111 (2.1)	111 (3.4)	85 (2.3)	104 (3.9)	104 (0.8)	90 (1.6)	109 (0.4)	78 (2.5)
	30	108 (2.9)	105 (0.1)	111 (2.6)	84 (10.6)	101 (0.5)	104 (2.2)	104 (2.0)	101 (1.3)	104 (2.6)
	50	109 (1.7)	109 (1.5)	109 (2.0)	87 (0.9)	106 (3.0)	112 (0.6)	101 (1.7)	108 (0.4)	96 (1.4)
Phenyl ITC	20	104 (1.0)	106 (0.5)	104 (1.0)	85 (2.6)	106 (3.2)	80 (2.1)	74 (3.2)	101 (2.0)	81 (2.6)
	30	88 (2.1)	82 (2.7)	83 (3.8)	71 (1.3)	91 (1.3)	78 (4.7)	74 (4.8)	79 (1.4)	80 (1.6)
	50	103 (1.0)	90 (3.0)	82 (2.4)	93 (0.3)	96 (1.0)	101 (2.8)	89 (2.1)	94 (4.0)	102 (1.8)

* Values expressed as the mean \pm SD (n = 3).

