

Supplementary material

Table S1. Samples analysed in this work.

Code	Name	Scientific name	Type	Format	Type of farming	Additional Information
Bu-F1O	Buckwheat	<i>Fagopyrum Esculentum</i>	Pseudo-cereal	Flour	Organic	-
Bu-F2O	Buckwheat	<i>Fagopyrum Esculentum</i>	Pseudo-cereal	Flour	Organic	-
Bu-GC	Buckwheat	<i>Fagopyrum Esculentum</i>	Pseudo-cereal	Grain	Conventional	-
Qu-GO	Quinoa	<i>Chenopodium quinoa</i>	Pseudo-cereal	Grain	Organic	-
Am-GO	Amaranth	<i>Amaranthus spp</i>	Pseudo-cereal	Grain	Organic	-
Te-FO	Teff	<i>Eragrostis tef</i>	Cereal	Flour	Organic	Whole flour
Co-RFC	Corn	<i>Zea mays</i>	Cereal	Flour	Conventional	Refined
Co-FO	Corn	<i>Zea mays</i>	Cereal	Flour	Organic	-
bCo-FC	Blue corn	<i>Zea mays</i>	Cereal	Flour	Conventional	-
So-FO	Sorghum	<i>Sorghum bicolor</i>	Cereal	Flour	Organic	-
Mi-GO	Millet	<i>Panicum miliaceum</i>	Cereal	Grain	Organic	Peeled
rLe-FO	Red lentil	<i>Lens culinaris</i>	Legume	Flour	Organic	-
gLe-FO	Green lentil	<i>Lens culinaris</i>	Legume	Flour	Organic	-
Ch-FC	Chickpea	<i>Cicer arietinum</i>	Legume	Flour	Conventional	-
Pe-FC	Pea	<i>Pisum sativum</i>	Legume	Flour	Conventional	-

Table S2. Information declared in the nutrition facts label of the samples analysed in this work.

Sample	Per 100 g						
	Fat (g)	Of which saturated (g)	Carbohydrates (g)	Of which sugars (g)	Fibre (g)	Protein (g)	Sodium (g)
Buckwheat *	3.2	0.66	65.9	1.16	3	12.9	0.001
Quinoa	5.94	0.53	62.44	1.6	-	12.19	-
Amaranth	7	1.5	65.2	1.7	6.7	13.6	0
Teff	3.3	0.8	60.1	3.2	5.9	8.8	0.08
Refined Corn	<0.5	<0.1	86	0	1	<0.5	<0.01
Corn	3	1	66	2	-	9	0.002
Blue Corn	2	0.4	80.3	0	-	6.6	0
Sorghum	2.6	0.4	72	1.3	4.3	8.1	0.01
Millet	4.2	0.72	64.3	8.2	8.5	11.2	0.01
Red Lentil	2.6	0.2	54.9	2.3	5.2	27.2	0.003
Green Lentil	2.2	0.4	59.2	0.4	10.8	25	<0.02
Chickpea	6.5	0.8	55.7	7.4	9.67	21.33	<0.1
Pea	1	0	64	0	6	22	0

* Buckwheat flour of organic farming sample (Bu-F1O)

Table S3. Instrumental validation parameters of the HPLC/MS-MS separation.

Parameters	Concentrations	Atropine	Scopolamine
Linear range ($\mu\text{g/mL}$)		0.005- 5	0.005 - 5
Linearity		$7.3 \cdot 10^8 x + 1.5 \cdot 10^7$	$3.3 \cdot 10^8 x + 1.8 \cdot 10^7$
R ²		0.998	0.996
Slope RSD%		7	2
LOD ^a ($\mu\text{g/L}$)		0.004	0.014
LOQ ^b ($\mu\text{g/L}$)		0.013	0.05
Repeatability, RSD%	0.05 $\mu\text{g/L}$	2	4
	5 $\mu\text{g/L}$	1	2
	200 $\mu\text{g/L}$	1	1
	5000 $\mu\text{g/L}$	2	1
Whithin-laboratory reproducibility, RSD%	0.05 $\mu\text{g/L}$	5	4
	5 $\mu\text{g/L}$	5	8
	200 $\mu\text{g/L}$	5	3
	5000 $\mu\text{g/L}$	7	7

^a Limit of detection.

^b Limit of quantification.

Table-S4. Evaluation of the matrix effect comparing the spiked sample (0.1 mg/kg) with a standard solution of atropine and scopolamine with the same concentration*.

Sample	Atropine		
	Group 1	Group 2	Group 3
Buckwheat	Bu-F1O		
Quinoa	Qu-GO		
Corn	Co-RFC		
Blue corn	bCo-FC		
Corn	Co-FO		
Teff	Te-FO		
Millet	Mi-GO		
Sorghum	So-FO		
Amaranth		Am-GO	
Chickpea		Ch-FC	
Red lentil		rLe-FO	
Pea			Pe-FC
Green lentil			gLe-FO
Sample	Scopolamine		
	Group 1	Group 2	Group 3
Corn	Co-RFC		
Buckwheat	Bu-F1O		
Quinoa	Qu-GO		
Sorghum	So-FO		
Corn	Co-RFC		
Millet	Mi-GO		
Teff	Te-FO		
Blue corn	bCo-FC		
Red lentil		rLe-FO	
Chickpea		Ch-FC	
Amaranth		Am-GO	
Green lentil			Pe-FC
Pea			gLe-FO

* Data were statistically compared by Analysis of Variance (ANOVA) and Duncan's Multiple range test.

Table S5. Recovery, intra-day and inter-day repeatability of the method calculated on a sorghum flour sample (So-FO) spiked with atropine and scopolamine.

Analyte level	Recovery (% ± SD)	Intra-day precision (%RSD)	Inter-day precisión (%RSD)
Atropine (µg/kg)			
1.5	105 ± 9	-	-
5	93 ± 7	4	8
100	94 ± 12	11	13
Scopolamine (µg/kg)			
2.4	96 ± 8	-	-
5	96 ± 17	6	18
100	93 ± 17	8	19

Table S6. Textural properties and functionalization degree of five different batches of prepared M-SBA-15-SO₃⁻ sorbent.

M-SBA-15-SO ₃ ⁻	S _{BET} (m ² /g) ^a	Pore volume (cm ³ /g) ^b	Pore size (Å) ^c	L ₀ (mmol S/g) ^d
Batch 1	475.1	0.56	48.5	0.944
Batch 2	461.8	0.54	49.4	0.981
Batch 3	533.6	0.61	49.5	1.049
Batch 4	507.4	0.58	49.1	1.151
Batch 5	459.4	0.52	50.0	0.928

^a S_{BET}: Specific surface area calculated by Brunauer-Emmett-Teller (BET) method. ^b Pore volume: Total pore volume measured at relative P/P₀=0.97. ^c Pore size: Pore diameter estimated by Barret-Joyner-Halenda (BJH) model applied in the desorption branch. ^d L₀: Functionalization degree estimated through the %S determined by elemental analysis.

Table S7. RASFF notifications of the last 5 years (2015-2020) on atropine and scopolamine in cereal and bakery products.

Date	Country	Subject	Action taken	Decision
04/06/2015	Germany	Atropine (198.5 µg/kg - ppb) and scopolamine (45 µg/kg - ppb) in organic polenta cornmeal from Germany	Recall from consumers	Serious
18/09/2015	Germany	Atropine (29 µg/kg - ppb) and scopolamine (6 µg/kg - ppb) in microwave popcorn from Spain	Recall from consumers	Serious
26/11/2015	Czech Republic	Atropine (1200; 1500 µg/kg - ppb) and scopolamine (360; 460 µg/kg - ppb) in sorghum flour from the Czech Republic with raw material from the Czech Republic and Slovakia	Withdrawal from the market	Serious
01/02/2016	Czech Republic	Atropine (180; 130 µg/kg - ppb) and scopolamine (36;27 µg/kg - ppb) in gluten-free baking mix and related products based on sorghum from the Czech Republic	Destruction	Serious
13/04/2016	Czech Republic	Atropine (7.9 µg/kg - ppb) in baby porridge without milk from Spain	Withdrawal from the market	Undecided
22/07/2016	Austria	Atropine (10.5; 12.2; 12.3 µg/kg - ppb) and scopolamine (2.0; 3.8; 3.1 µg/kg - ppb) in corn from Germany	Withdrawal from the market	Serious
20/09/2016	Austria	Atropine (23.5 µg/kg - ppb) and scopolamine (9.5 µg/kg - ppb) in millet flour from Hungary	Public warning - press release	Serious
24/05/2018	Germany	Atropine (6.60 µg/kg - ppb) and scopolamine (1.77 µg/kg - ppb) in popcorn from France	Withdrawal from the market	Serious
05/06/2018	Germany	Scopolamine (10.3 µg/kg - ppb) in popcorn in grain from Argentina	Recall from consumers	Serious
26/09/2018	Germany	Atropine (60.7 µg/kg - ppb) and scopolamine (38.8 µg/kg - ppb) in organic muesli from Austria	Recall from consumers	Serious
20/12/2018	France	Atropine (53 µg/kg - ppb) and scopolamine (20 µg/kg - ppb) in organic buckwheat flour from France		Serious
01/04/2019	Croatia	Atropine (4.5 µg/kg - ppb) and scopolamine (4.3 µg/kg - ppb) in corn grits from Serbia	Public warning - press release	Serious
06/08/2019	Netherlands	Atropine (2.2 µg/kg - ppb) and scopolamine (2.7 µg/kg - ppb) in breakfast porridge 8 cereals with honey from Spain	Withdrawal from the market	Undecided
26/08/2019	France	Atropine (47 µg/kg - ppb) and scopolamine (30 µg/kg - ppb) in organic buckwheat flour from France	Recall from consumers	Serious
24/01/2020	Germany	Atropine (19 µg/kg - ppb) and scopolamine (6.4 µg/kg - ppb) in organic soy flakes from Austria, with raw material from the Czech Republic	Withdrawal from the market	Serious

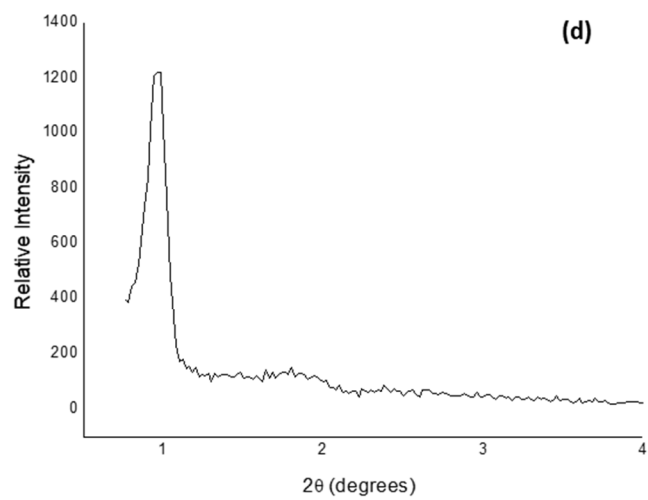
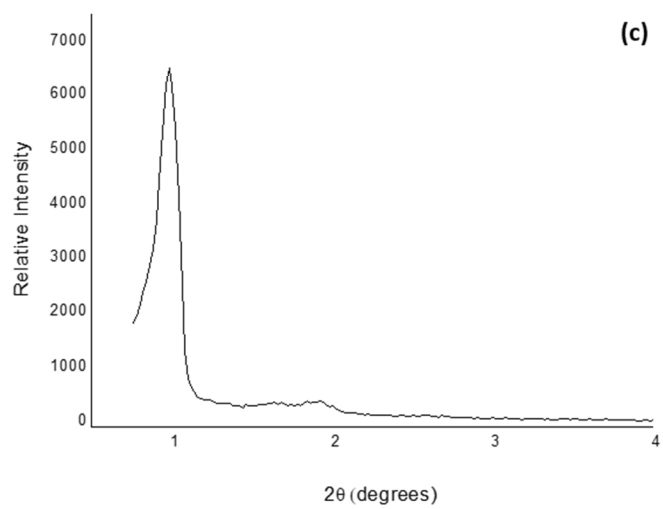
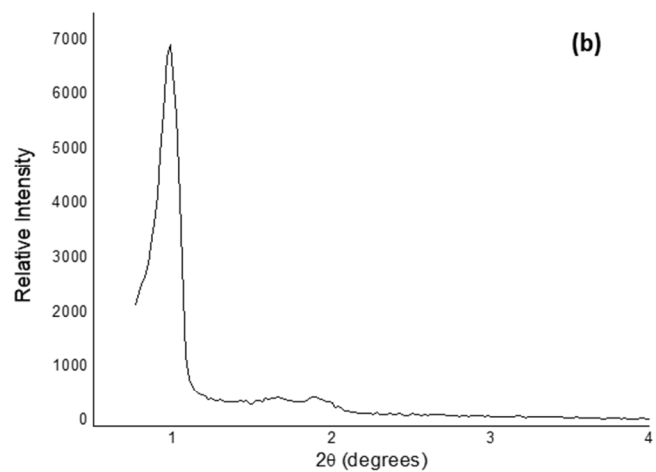
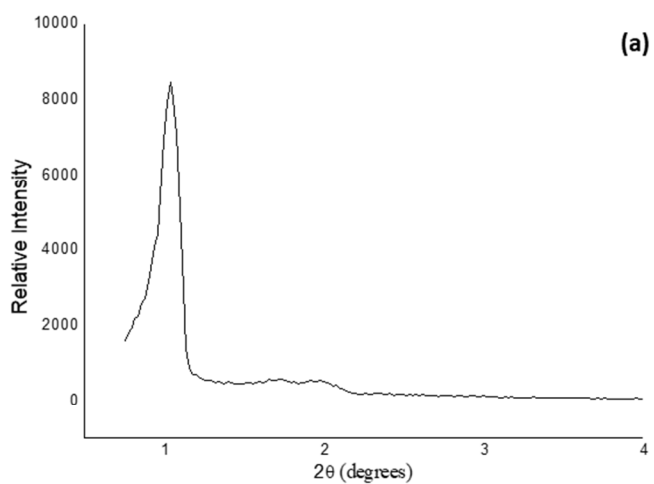


Figure S1. XRD patterns of: (a) SBA-15; (b) L-SBA-15-SO₃⁻; (c) M-SBA-15-SO₃⁻ and (d) H-SBA-15-SO₃⁻.

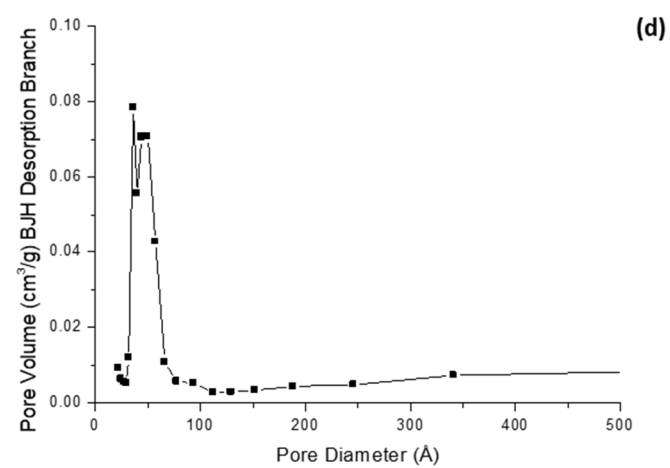
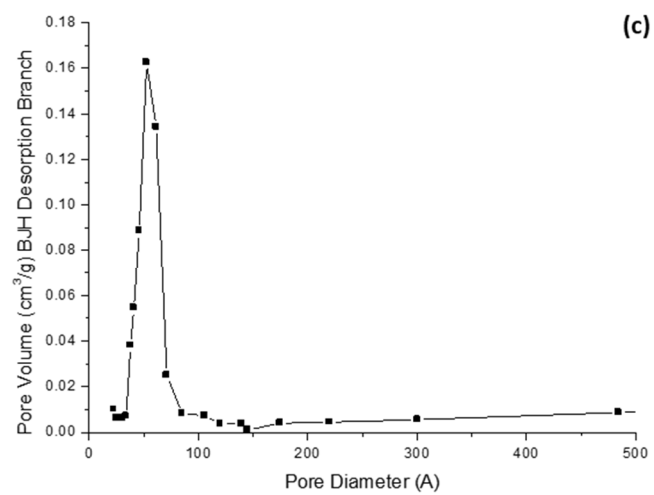
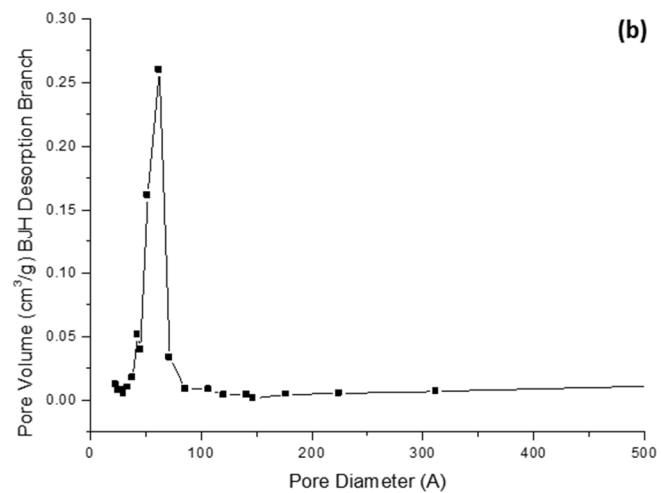
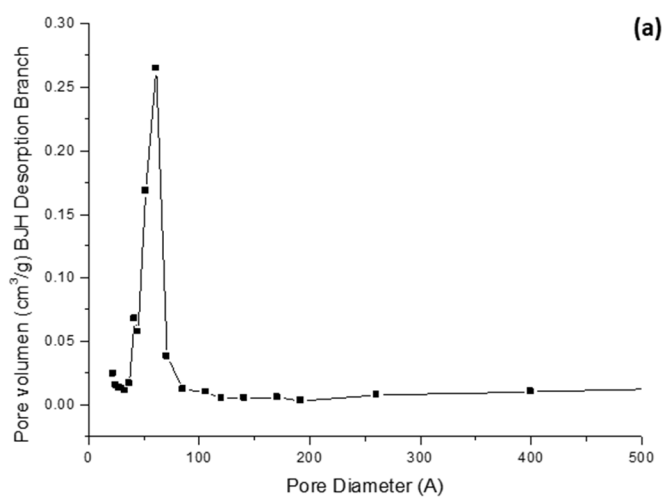


Figure S2. Pore size distribution of: (a) SBA-15, (b) L-SBA-15-SO₃⁻, (c) M-SBA-15-SO₃⁻ and (d) H-SBA-15-SO₃⁻.