

Supplementary Table 1. Plackett-Burman factorial design for determination of significant factors.

Run	Factors									Recovery (%)		
	a	b	c	d	e	f	g	h	j	Dinoseb	Bromoxynil	Ioxynil
1	1	-1	-1	-1	1	-1	1	1	-1	67.6	94.5	73.9
2	-1	-1	-1	1	-1	1	1	-1	1	29.9	49.4	38.7
3	-1	1	1	1	-1	-1	-1	1	-1	40.9	65.1	51.9
4	1	-1	1	1	1	-1	-1	-1	1	41.7	66.1	51.6
5	-1	-1	-1	-1	-1	-1	-1	-1	-1	44.6	73.4	64.9
6	-1	1	1	-1	1	1	1	-1	-1	24.8	42.3	36.1
7	1	1	1	-1	-1	-1	1	-1	1	10.1	20.9	14.1
8	1	1	-1	1	1	1	-1	-1	-1	24.8	44.9	33.7
9	-1	-1	1	-1	1	1	-1	1	1	29.1	49.6	33.4
10	1	-1	1	1	-1	1	1	1	-1	68.9	90.3	78.7
11	1	1	-1	-1	-1	1	-1	1	1	73.2	102.1	84.8
12	-1	1	-1	1	1	-1	1	1	1	37.8	63.7	60.1

Supplementary Table 2. Factors, symbols and levels for the CCD

Factor	Symbol	Level				
		$-\alpha$ (low)	-1	0	1	$+\alpha$ (high)
Amount of Na_2CO_3 (mg)	A	50	70	100	130	150
Amount of AIL (mg)	B	200	280	400	520	600
Amount of Fe_3O_4 (mg)	C	15	21	30	39	45

Supplementary Table 3. Design matrix and responses for the CCD

Run	Block	A: Amount of Na ₂ CO ₃ (mg)	B: Amount of AIL (mg)	C: Amount of Fe ₃ O ₄	Recovery (%)
1	1	100	400	30	102.3
2	1	100	400	30	98.6
3	1	70	520	21	48.9
4	1	130	280	21	61.9
5	1	130	520	21	73.1
6	1	70	280	39	57.9
7	1	100	400	30	105.8
8	1	70	520	39	70.9
9	1	100	400	30	97.8
10	1	70	280	21	48.2
11	1	130	520	39	93.2
12	1	130	280	39	71.7
13	2	100	600	30	80.9
14	2	100	400	30	104.6
15	2	100	400	15	60.8
16	2	150	400	30	83.7
17	2	100	400	45	85.8
18	2	50	400	30	59.5
19	2	100	400	30	101.8
20	2	100	200	30	63.3

Supplementary Table 4. Precision and accuracy data for the determination of PPs in wheat, rice, corn and soybean flour samples (6 days with 6 replicates).

Analytes	Compound	Concentration ($\mu\text{g kg}^{-1}$)			RSD (%)	Concentration ($\mu\text{g kg}^{-1}$)			RSD (%)
		Spiked	Detected	ER (%)	Intra-day	Spiked	Detected	ER (%)	Inter-day
Wheat flour	Dinoseb	10	9.3±0.6	93.0	3.2	10	9.4±0.7	94.0	3.7
		50	49.1±1.3	98.2	1.3	50	46.2±2.1	92.4	2.3
		100	96.5±2.2	96.5	1.1	100	97.8±4.6	97.8	2.4
		500	486.8±5.6	97.4	0.6	500	487.6±6.2	97.5	0.6
	Bromoxynil	10	9.2±0.5	92.0	2.7	10	9.3±0.8	93.0	4.3
		50	47.8±2.4	95.6	2.5	50	45.2±3.2	90.4	3.5
		100	93.6±4.1	93.6	2.2	100	94.7±5.4	94.7	2.9
		500	506.3±4.4	101.3	0.4	500	492.8±7.2	98.6	0.7
	Ioxynil	10	9.1±1.2	91.0	6.6	10	8.9±1.3	89.0	7.3
		50	46.6±1.8	93.2	1.9	50	46.2±2.6	92.4	2.8
		100	102.6±2.5	102.6	1.2	100	98.6±3.6	98.6	1.8
		500	510.5±4.8	102.1	0.5	500	489.2±6.2	97.8	0.6
Rice flour	Dinoseb	10	9.1±0.8	91.0	4.4	10	8.9±1.1	89.0	6.2
		50	47.8±2.3	95.6	2.4	50	46.1±2.6	92.2	2.8
		100	96.2±3.3	96.2	1.7	100	95.8±4.6	95.8	2.4
		500	499.2±6.4	99.8	0.6	500	504.7±8.7	100.9	0.9
	Bromoxynil	10	9.1±1.1	91.0	6.0	10	9.2±1.6	92.0	8.7
		50	47.8±3.2	95.6	3.3	50	48.2±3.8	96.4	3.9
		100	92.7±4.6	92.7	2.5	100	93.5±6.6	93.5	3.5
		500	478.6±6.3	95.7	0.7	500	479.6±10.2	95.9	1.1
	Ioxynil	10	8.7±0.9	87.0	5.2	10	9.1±1.4	91.0	7.7
		50	45.6±2.6	91.2	2.9	50	46.1±4.8	92.2	5.2
		100	96.8±6.4	96.8	3.3	100	97.6±8.2	97.6	4.2
		500	457.9±11.2	91.6	1.2	500	482.7±15.2	96.5	1.6

		10	8.8±1.1	88.0	6.3	10	9.1±1.2	91.0	6.6
		50	46.2±3.7	92.4	4.0	50	47.6±4.3	95.2	4.5
	Dinoseb	100	92.7±3.9	92.7	2.1	100	90.6±5.2	90.6	2.9
		500	462.5±6.2	92.5	0.7	500	478.6±7.8	95.7	0.8
		10	9.2±0.8	92.0	4.3	10	8.9±1.2	89.0	6.7
		50	51.3±2.2	102.6	2.1	50	53.6±2.7	107.2	2.5
Corn flour	Bromoxynil	100	104.5±4.2	104.5	2.0	100	106.6±5.4	106.6	2.5
		500	517.2±9.4	103.4	0.9	500	509.7±11.3	101.9	1.1
		10	8.8±0.9	88.0	5.1	10	9.6±1.3	96.0	6.8
		50	50.8±4.7	101.6	4.6	50	49.7±4.6	99.4	4.6
	Ioxynil	100	105.5±5.1	105.5	2.4	100	100.6±5.3	100.6	2.6
		500	509.3±7.8	101.9	0.8	500	491.8±8.5	98.4	0.9
		10	8.6±1.4	86.0	8.1	10	8.7±1.3	87.0	7.5
		50	46.6±3.1	93.2	3.3	50	48.3±3.6	96.6	3.7
	Dinoseb	100	92.2±4.3	92.2	2.3	100	91.5±5.2	92.7	2.8
		500	478.3±8.2	95.7	0.9	500	482.2±8.8	96.4	0.9
		10	9.1±1.1	91.0	6.0	10	9.2±1.3	92.0	7.1
		50	48.3±2.6	96.6	2.7	50	48.7±3.3	97.4	3.4
Soybean flour	Bromoxynil	100	104.2±4.6	104.2	2.2	100	99.5±4.5	99.5	2.3
		500	514.6±6.8	102.9	0.7	500	498.3±10.6	99.7	1.1
		10	8.5±0.9	85.0	5.3	10	9.3±1.1	93.0	5.9
		50	53.6±2.7	107.2	2.5	50	49.6±2.7	99.2	2.7
	Ioxynil	100	96.7±3.6	96.7	1.9	100	99.9±4.6	99.9	2.3
		500	487.9±13.2	97.6	1.4	500	492.2±16.8	98.4	1.7

Note: (1) ER indicates extraction recovery; (2) RSD indicates relative standard deviations.