

Soil

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Response to the second reviewer's comments

First of all, we would like to thank you for your valuable comments and suggestions which help us to improve our manuscript. Below we try to address all the points which you have indicated in your assessment opinions.

General comment:

Comment: This study provide results on herbicide EPM behaviour in paddy soils. I think that these experimental data bay help understand this compound in risk assessment.

Response: Thank you very much for your support of our manuscript. We further revised our manuscript according to your comments.

Specific comments:

Comment 1: Suggestions:

For degradation study, did the author test the degradation products by MASS or other detection means?

Response 1: This suggestion has been adopted. We apologize for not analyzing and testing the degradation products. Thus, this experiment has been included in our work this year. Thank you for your valuable suggestions to improve our research.

Comment 2: It's recommended to provide the analytical method performance in validation, and typical chromatograms.

Response 2: This suggestion has been adopted. We supplemented typical chromatograms of the analytical method performance. The selective ion chromatograms of EPM in acetonitrile, paddy water, paddy soil, paddy straw, brown rice and rice hulls samples spiked at 0 and 0.1 mg kg⁻¹ were shown in Figure 1 (A-F). Five parallel tests were conducted for each matrix spiked with EPM at three different levels (0.005, 0.01, and 0.1 mg kg⁻¹). After sample pretreatment by the optimized QuEChERS procedure, the recovery of EPM in the various matrices ranged between 90.95% and 110.12%, with RSDs of 1.3% – 9.8% for repeatability (Table 1), and with RSDs of 3.63% – 8.49% for repeatability (Table 2). Five parallel tests were conducted for the blank matrix of paddy water samples spiked at 0.005, 0.01 and 0.1 mg kg⁻¹ of EPM, respectively, and the chromatograms were shown in Figure 2 (A-C).

Chromatograms of EPM on the five columns of one batche and on the five columns of the different batches were shown in Figure 3 (A-B). Thus, the developed analytical method fulfills the requirements of SANTE/11813/2017 guidelines and fall within the range of 70 – 120 % for recovery and less than 20% for RSD (Sante, 2017).

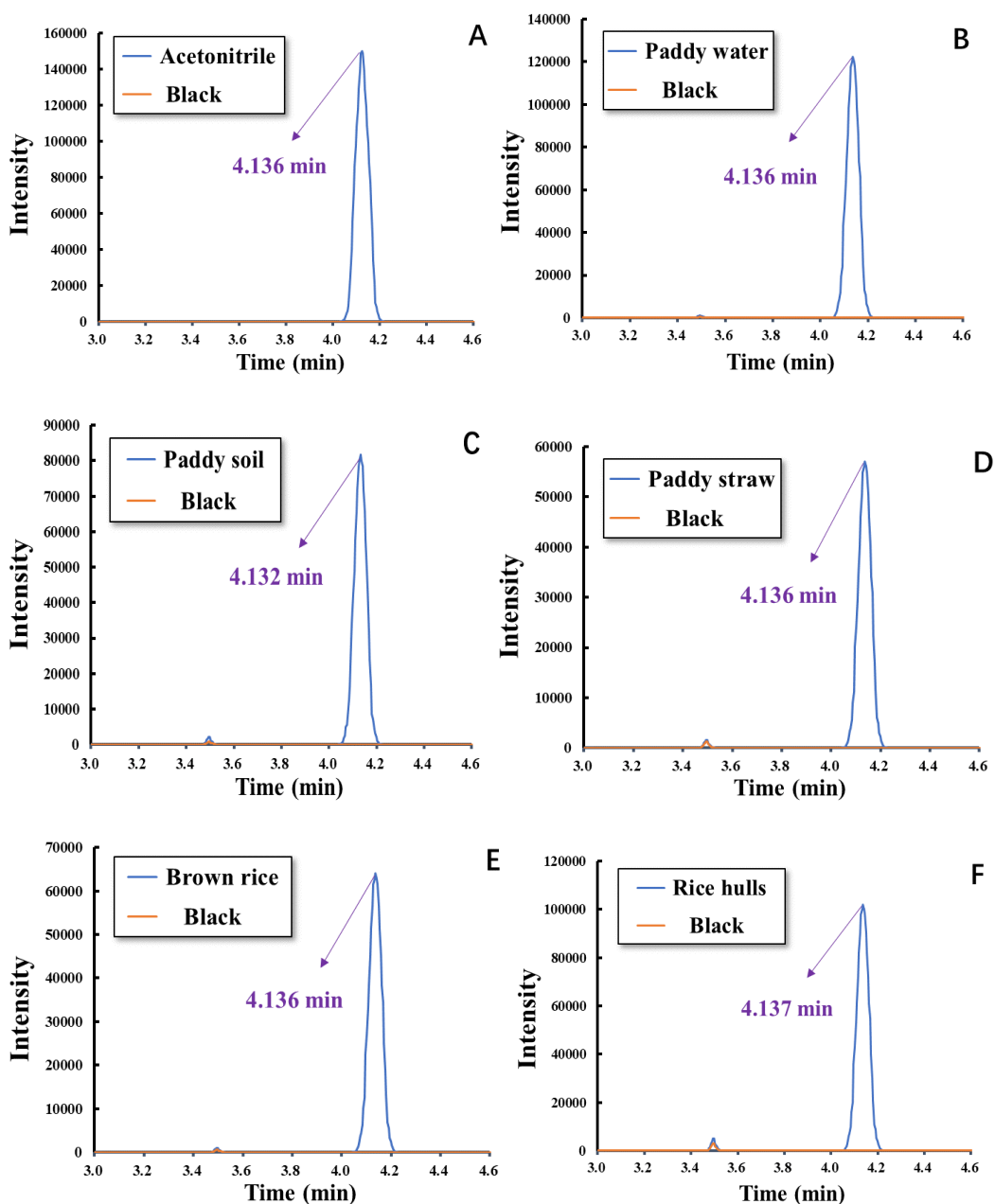


Figure 1 The selective ion chromatograms of blank (A) acetonitrile, (B) paddy water, (C) paddy soil, (D) paddy straw, (E) brown rice and (F) rice hulls samples spiked at 0 and 0.1 mg kg⁻¹.

Table 1 Recovery and relative standard deviation (RSD) of EPM in various matrices spiked at levels of 0.005, 0.01, and 0.1 mg kg⁻¹ (n=5)

Matrix	Spiked level (mg kg ⁻¹)	Recovery(%)					Mean recovery (%)	RSD (%)
		1	2	3	4	5		
Paddy water	0.005	93.51	107.03	91.03	90.95	104.35	97.37	7.93
	0.01	91.96	96.10	97.76	101.58	110.12	99.50	6.90
	0.1	93.93	92.88	103.45	108.15	109.67	101.62	7.72
Paddy soil	0.005	104.94	99.57	100.35	102.84	102.35	102.01	2.09
	0.01	108.01	93.85	94.10	94.22	104.79	98.99	6.93
	0.1	98.22	102.26	108.82	97.82	97.26	100.88	4.82
Rice straw	0.005	100.73	109.29	91.89	93.56	108.38	100.77	8.02
	0.01	102.67	95.22	93.22	103.30	102.91	99.46	4.87
	0.1	93.09	95.27	109.84	90.19	95.39	96.76	7.87
Brown rice	0.005	91.10	91.72	104.98	107.54	104.22	99.91	7.87
	0.01	92.17	109.97	108.62	91.22	97.30	99.86	8.95
	0.1	108.94	92.88	91.52	95.18	95.98	96.90	7.18
Rice Hulls	0.005	100.09	98.40	104.96	105.42	97.88	101.35	3.55
	0.01	97.21	102.68	94.47	96.10	92.12	96.52	4.09
	0.1	100.09	92.77	93.50	93.92	98.87	95.83	3.53

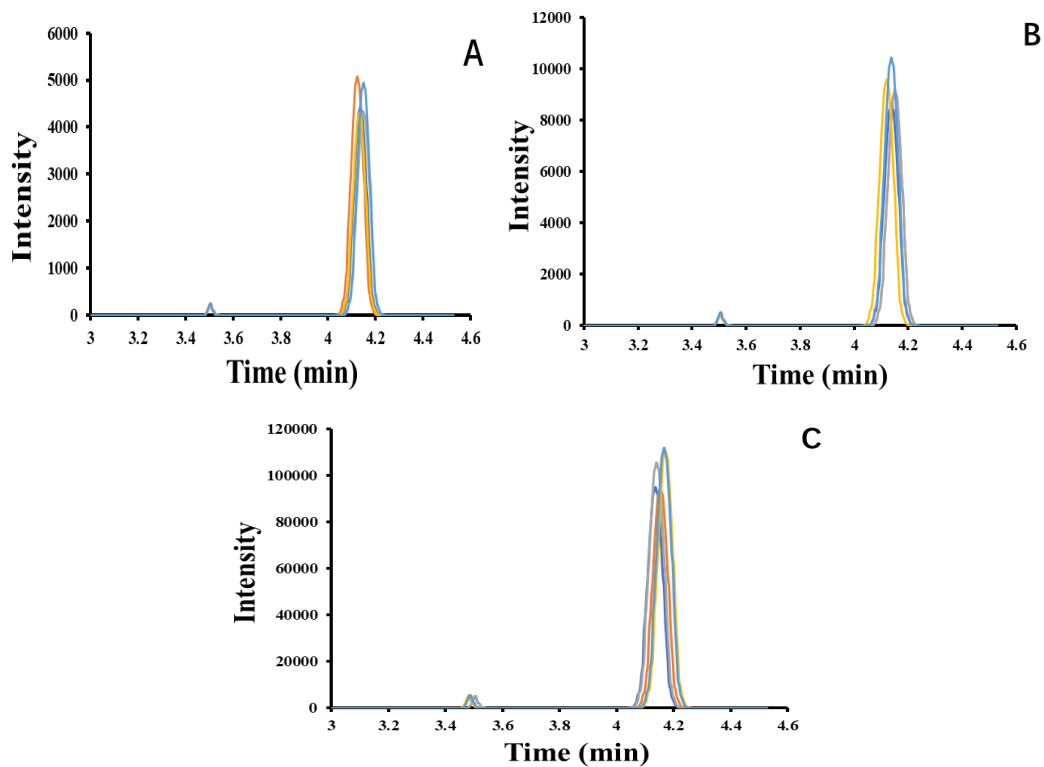


Figure 2 Recovery of EPM in paddy water spiked at levels of 0.005, 0.01, and 0.1 mg kg⁻¹ (n=5)

Table 2 Reproducibility of the retention time, precursor signal, and retention factor of EPM

	RSD (%)	
	Column-to-column reproducibility on five columns	Batch-to-batch reproducibility on six batches
Retention time	4.89	6.01
Precursor signal	7.27	8.49
Retention factor	3.63	5.87

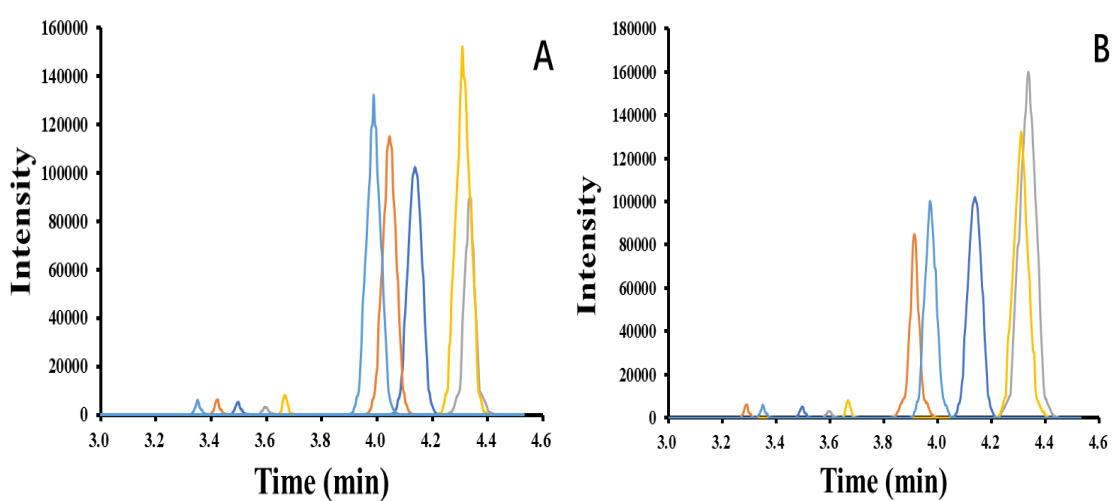


Figure 3 Chromatograms of EPM in paddy water on the five columns of the first batch (A) and on the five columns of the different batches (B) samples spiked at 0.1 mg kg^{-1}

Reference:

SANTE: Guidance document on analytical quality control and method validation procedures for pesticide residues analysis in food and feed, 2017.

Thanks again for your kindly comments.