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Dissipation and Residues of 2,4-D-dimethylammonium in Wheat and Soil

He Jiang · Shen Yan · Wang Donglan · Sun Xing · Fan Mingtao · Liu Xianjin

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Abstract The environmental behavior of 720 g/L 2,4-D-dimethylammonium AS under wheat field conditions was studied in detail. The dissipation behaviors of 2,4-D-dimethylammonium in wheat and soil were fitted to first-order kinetic equation ($C = C_0e^{-\lambda t}$). But different experimental points and years have different initial deposits and half-lives ($T_{1/2}$). After applying 720 g/L 2,4-D-dimethylammonium AS at recommended dosage and 1.5-fold recommended dosage during 3–4 leaf stage, the residues of 2,4-D-dimethylammonium in wheat plant, wheat grain and soil were all lower than 0.02 mg/kg. The obtained results suggested that, it is safety for application at the recommended dosage during 3–4 leaf stage to control weeds in wheat field.

Keywords 2,4-D-dimethylammonium · Dissipation · Residues · Wheat · Soil

Wheat is an important food crop in the world, but the impact of plant diseases, pests and field weeds on wheat production are also highlighted. To control these adverse factors, pesticides play an important role with new formulations being introduced on a regular basis. Amongst these, chlorinated phenoxyacetic acid herbicides, such as 2,4-D-dimethylammonium is commonly used for control of weeds in wheat field (Legrouri et al. 2005). However, the abuse of pesticides in agriculture is a matter of both food safety and environmental concern because these chemicals are recognized as a source of potential adverse impact (Alexandre Prado and Claudio 2002). So, environmental behavior research of pesticides is urgently needed to ensure their using safely and effectively. Although there are lost of papers about environmental behavior of pesticides have been published (Cao et al. 2008; Guo et al. 2010; Sondhia 2009), relevant work about 2,4-D-dimethylammonium is still absent. The present investigation was therefore undertaken to study the dissipation and residues of 2,4-D-dimethylammonium in wheat and soil under open field conditions.

H. Jiang · S. Yan · W. Donglan · S. Xing · L. Xianjin (✉)
Key Laboratory of Food Quality and Safety of Jiangsu Province, Key Laboratory of Food Safety Monitoring and Management, Ministry of Agriculture, 210014 Nanjing Jiangsu, People's Republic of China
e-mail: xianjin_liu@yahoo.cn

H. Jiang · F. Mingtao
Department of Food Science and Engineering, Northwest A&F University, 712100 Yangling Shaanxi, People's Republic of China

Materials and Methods

The field experiments were laid out in China at cities of Nanjing (118.78°E, 32.04°N), Chengdu (104.06°E, 30.67°N) and Yantai (121.39°E, 37.52°N) during 2008 and 2009 in a plot size of 30 m^2 with three replications per treatments. Herbicide 720 g/L 2,4-D-dimethylammonium AS was sprayed, recommended dosage (540 mL/hm^2) and 1.5-fold r ecommended dosage (810 mL/hm^2) were tested. In dissipation experiments, high dosage was applied during 3–4 leaf stage, then wheat plant (about 1 kg) and soil (0–10 cm, about 2 kg) were sampled randomly form each plot at 0 (2 h), 1, 3, 5, 7, 10, 14, 21, 28, 35 and 45 days after application. In residues experiments, both high and low dosages were applied in the same period as dissipation experiments, then wheat plants (about 1 kg), wheat grains (about 2 kg) and soil (0–10 cm, about 2 kg) were sampled

Table 1 Recovery of 2,4-D-dimethylammonium in each substrate

Substrate	Spiked level (mg/kg)	Recovery % (Mean ± SD, n = 5)
Wheat plant	0.02	107.12 ± 9.41
	0.50	84.69 ± 3.13
Wheat grain	0.02	101.23 ± 5.95
	0.50	86.42 ± 3.58
Soil	0.02	95.34 ± 10.12
	0.50	90.56 ± 6.02

randomly in harvest time. The period between pesticide application and wheat harvest was about 65 days. For control treatment, no pesticide was sprayed and samples were collected as experimental treatment. All samples were stored in deep freeze (−20°C) until detection.

The representative each samples from the stored replication were powered, and 10 mg each samples were extracted with 40 mL acetone for 60 min on a mechanical shaker (200 r/min). The contents were then filtered and 10 mL filtrates were evaporated to dryness by nitrogen blowing, then residues were dissolved in 1 mL chromatographic pure methanol and filtered (0.22 μm) for LC–MS/MS analysis. The contents of 2,4-D-dimethylammonium were determined by liquid chromatography coupled to tandem quadrupolar mass spectrometry (LC-QQQ) (Agilent G6410A QQQ, US) under multiple-reaction monitoring (MRM) mode, using m/z 219 ≥ 124.9 as qualifier and m/z 219 ≥ 160.9 as quantifier. Matrix-matched calibration standards with a series of concentration were used to developing calibration curve. The concentrations of 2,4-D-dimethylammonium in different samples were calculated with the help of their respective matrix calibration curve. The limit of quantification (LOQ) of 2,4-D-dimethylammonium was calculated as previous reference (Sondhia 2008) and found to be 0.02 mg/kg in all substrates.

Wheat plant, wheat grain and soil (CK) were spiked with 2,4-D-dimethylammonium at 0.02 and 0.50 mg/kg level and analyzed as per the methodology described above. Percent recovery of 2,4-D-dimethylammonium in those samples were found to be consistent and more than 80% (Table 1).

Results and Discussion

The results of dissipation of 2,4-D-dimethylammonium in wheat plant and soil are presented in Table 2. After application of 720 g/L 2,4-D-dimethylammonium AS at 810 mL/hm^2 level, the initial deposits of 2,4-D-dimethylammonium on wheat plant and soil were slightly different between different experimental points and years. It also showed that more than 95% of the residue in wheat plant and soil had dissipated in 10 days after treatment. The dissipation behavior of 2,4-D-dimethylammonium in both wheat plant and soil were fitted to first-order kinetic equation ($C = C_0 e^{-\lambda t}$) and the determination coefficients (R^2) of kinetic equation were all higher then 0.92 (Table 3). The half-lives ($T_{1/2}$) of 2,4-D-dimethylammonium in wheat and soil were different between different experimental points and years as the initial deposits. These differences might due to the different weather conditions in each experiment point.

Table 2 Dissipation of 2,4-D-dimethylammonium in wheat plant and soil after the application of 720 g/L 2,4-D-dimethylammonium AS at 810 mL/hm^2 level

Days after application	Content of 2,4-D-dimethyl amine salt (mg/kg) (n = 3)											
	Nanjing				Chengdu				Yantai			
	2008		2009		2008		2009		2008		2009	
	Wheat	Soil	Wheat	Soil	Wheat	Soil	Wheat	Soil	Wheat	Soil	Wheat	Soil
0(2 h)	14.16	1.66	16.05	0.83	13.37	1.36	15.36	0.59	10.69	0.68	17.11	0.64
1	13.02	0.76	14.60	0.51	12.37	0.75	11.55	0.56	8.87	0.48	11.62	0.41
3	6.05	0.30	9.47	0.26	6.32	0.36	9.90	0.26	5.06	0.39	9.98	0.27
5	3.16	0.14	4.16	0.14	3.40	0.25	4.32	0.14	2.61	0.23	4.68	0.14
7	0.95	0.05	0.61	0.06	1.00	0.16	0.61	0.13	0.59	0.09	0.63	0.07
10	0.38	<0.02	0.32	<0.02	0.42	0.04	0.35	<0.02	0.23	<0.02	0.37	<0.02
14	<0.02	<0.02	<0.02	<0.02	0.18	<0.02	0.13	<0.02	<0.02	<0.02	<0.02	<0.02
21	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
28	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
35	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
45	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02

Table 3 Fitting analysis of the dissipation behavior of 2,4-D-dimethylammonium in wheat plant and soil

Experimental point	Year	Substrate	Curve-fit equation	R^2	$T_{1/2}$ (days)
Nanjing	2008	Wheat	$C = 17.24e^{-0.38T}$	0.985	1.82
		Soil	$C = 1.423e^{-0.48T}$	0.992	1.44
	2009	Wheat	$C = 22.63e^{-0.43T}$	0.939	1.61
		Soil	$C = 0.799e^{-0.37T}$	0.995	1.87
Chengdu	2008	Wheat	$C = 14.95e^{-0.33T}$	0.980	2.10
		Soil	$C = 1.185e^{-0.32T}$	0.976	2.17
	2009	Wheat	$C = 18.17e^{-0.37T}$	0.946	1.87
		Soil	$C = 0.605e^{-0.24T}$	0.952	2.89
Yantai	2008	Wheat	$C = 13.58e^{-0.40T}$	0.971	1.73
		Soil	$C = 0.723e^{-0.27T}$	0.941	2.56
	2009	Wheat	$C = 21.47e^{-0.41T}$	0.922	1.69
		Soil	$C = 0.620e^{-0.30T}$	0.993	2.31

The results of residues experiments indicated that, after applying 720 g/L 2,4-D-dimethylammonium AS during 3–4 leaf stage, the ultimate residues of 2,4-D-dimethylammonium in wheat plant, wheat grain and soil were all lower than 0.02 mg/kg, no matter high or low dosages were applied (data not show). Fresh or processed wheat straw is used as feeds for ruminants in many regions of China, and wheat grain is a staple food used to make flour for further process. The maximum residue limit (MRL) of 2,4-D in wheat grain is set at 0.5 mg/kg by China, USA, Japan and Korea government, and 2.00 mg/kg by Codex Alimentarius Commission (CAC). The undetectable residues of 2,4-D-dimethylammonium indicated that, it is safety for application 720 g/L 2,4-D-dimethylammonium AS at the recommended dosage during 3–4 leaf stage to control weeds in wheat field.

In the work of Cessna and Hunter (1993) a tank mixture of 2,4-D and dicamba was applied in wheat in the period of emergence. Their result indicated that the initial residues immediately after application decreased rapidly and, at 5–6 week after application, residues of 2,4-D were less than 0.1 mg kg^{-1} and residues of 2,4-D were not detectable in the straw or seed at maturity. A combination of both degradation and dissipative mechanisms control overall persistence of a chemical in the natural environments and these aspects are influenced both by the physico-chemical characteristics of the chemical compound and of the environmental matrix (Chandra et al. 2009).

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