Electronic Supplementary Material (ESI) for Environmental Science: Nano.

Supplementary material

Field evaluation of the potential effects of polymer and silica-based nanopesticides on strawberries and agricultural soils

Juliana A. Galhardi[1,2]*, Peiying Wang[3]*, Vinicius Bueno[4], Subhasis Ghoshal[4], Valérie Gravel[5], Kevin J. Wilkinson[2], Stéphane Bayen[3¥]

1. Institute of Science and Technology, Laboratory of Environmental Nanotechnology, São Paulo State University (UNESP), 18087-180 Sorocaba, SP, Brazil
2. Dept. of Chemistry, Université de Montréal, 1375 Ave. Thérèse-Lavoie-Roux, Montréal, QC, CANADA, H2V 0B3
3. Department of Food Science and Agricultural Chemistry, McGill University, Ste-Anne-de-Bellevue, QC, Canada
4. Department of Civil Engineering, McGill University, H3A 0C3 Montreal, QC, Canada
[5] Department of Plant Science, McGill University, Ste-Anne-de-Bellevue, QC, Canada

*co-first authors

[¥] Corresponding author current address and email:
Department of Food Science and Agricultural Chemistry
McGill University, 21111 Lakeshore, Ste-Anne-de-Bellevue
Quebec, Canada, H9X 3V9
Email: stephane.bayen@mcgill.ca

This file includes:

A) Methods to characterize the soil properties

(i) pH: A 1:1 (w/v) solution of soil to water was shaken for 30 min, then left to rest for 1 hour. The measurements were done on an Accumet AR15 research pH meter (Thermal Fisher).

(ii) pH buffering: the SMP single-buffer procedure (1) is applied to estimate the lime requirement.

(iii) % Organic matter by Loss on Ignition (LOI): A (previously heated to 105℃ for 24 hours) sample is burned at 360 ℃ for 4 hours. The difference in weight between the two steps is attributed to loss of organic matter (expressed as a percent).

(iv) Available P, K, Ca, Mg, Na, Al, Cu, Zn, Mn and Fe: A multi-element extraction was performed using the Mehlich III solution (a mixture of acetic acid, ammonium nitrate, ammonium fluoride, nitric acid and EDTA). A colorimetric technique was used for the determination of P (Lachat flow injection analysis). P was measured at 880 nm following complexation with ammonium molydate in a reducing solution of ascorbic acid. The determination of metals was performed by Atomic Absorption Spectrophotometry using standards prepared in adequate matrices and dilutions. Quantification was performed on a Varian 220FS.

(v) Extractable Ammonium and Nitrates in soils: An extraction is performed using a 1:10 soil-to-2M KCl solution, which was shaken for 1 hour. The filtrate is analyzed by colorimetry for the determination of N as NH_4 and N as NO_3 on a multi-channel Lachat autoanalyser. Ammonium is determined following heating of the solution with salicylate and hypochlorite in an alkaline phosphate buffer. The green color is measured colorimetrically at 660 nm using flow injection analysis. Nitrates were measured following reduction to nitrites in a copperized cadmium column. The magenta color is measured colorimetrically at 520 nm on a Lachat flow injection instrument.

(vi) Particle Size Distribution (hydrometer method): The hydrometer is used to measure the density of the material in suspension. Readings were performed at specific intervals according to settling times of grain sizes (considering temperature).

B) Nanoencapsulated $nSiO_2$ pesticides Synthesis. The PHSNs were synthesized following a previously reported protocol (2). Briefly, 300 mg of CTAB, 850 mg of Pluronic P123 and 15 mL of NH_4OH (30% v/v) were added to ethanol (37.5% v/v). Then, the SiO_2 precursor, TEOS, was added dropwise at 0.0125 mL/s for 800 s. After 5 hours under vigorous mixing, the suspension was dried overnight at 80°C and the resulting powder was calcined at 550°C for 5 hours to remove the remaining surfactants and ammonia. These NPs were fully characterized in a previous work (3, 4). The encapsulation of AZOX or BFT within the nanocarriers was achieved by suspending the PHSNs in a methanol-water mixture (20% v/v methanol) containing AZOX or BFT (0.1 mg/mL), as previously reported (4).

C) Pesticide treatments

Table - Pesticide treatments applied on strawberry plants

Formulation	Composition			
	Pesticide AI[a] (mg)	Nanocarrier	Dispersant[b]	Water
control	-	-	Yes	1 L
Conventional[c]	3.95 AZOX or 4.06 BFT	-	Yes	1 L
Allosperse®[d]	3.95 AZOX or 4.06 BFT	Proprietary Information	Yes	1 L
$nSiO_2$[c]	3.95 AZOX or 4.06 BFT	200 mg	Yes	1 L

[a]The amount of pesticide AI were for each application in each pot. The pesticides were applied in the field twice per year (2 years).
[b]Dispersants: surfactants, antifreeze, biocide and others. Dispersants were added in order to reproduce the amounts that were present in the nanoformulations provided by Vive Crop Protection.
[c]The pesticide AI in the conventional and $nSiO_2$ formulations were pesticide standards (3.95mg AZOX 96.5% or 4.06mg BFT 98.5%). For $nSiO_2$, the AI were added to a glass vial containing solvent and nanocarrier for the

synthesis (section B). After the loading process, the dispersants were added to the $nSiO_2$ and conventional formulations.
[d]The Allosperse® pesticides (AZOX 18.4% and BFT 19.3%) were commercial products from Vive Crop Protection..

D) Pesticide analysis in soils and plants

AZOX and BFT were analyzed in the ***strawberry plant tissues and soil*** based on a LC-QTOF-MS method developed by Wang et al. (5), which was an approach adapted from the QuEChERS technique (6), and validated for both the conventional and nanoformulations of the pesticides. In short, 2 g of homogenized fruit (n = 3) was weighed in 15-mL plastic centrifuge tubes in which 4 mL of 1% acetic acid in acetonitrile, 0.8 g of magnesium sulphate (Fisher Chemicals) and 0.2 g of sodium acetate (Fisher Chemicals) were added. Internal standards (40 µg kg^{-1} of D_4-AZOX and 60 µg kg^{-1} of D_5-BFT) were spiked into each sample. Solutions were vortexed for 15 minutes then centrifuged at 2240 x *g* (5 min, 20°C). One mL of the extract was transferred to centrifuge tubes containing 50 mg of a Primary Secondary Amine (PSA, Agilent) and 150 mg of $MgSO_4$. Solutions were then vortexed for 1 min, centrifuged (2240 x g, 20°C) for 5 min and filtered through a 0.22 µm PTFE filter (Polytetrafluoroethylene, Chrom4; Thuringen, Germany) into HPLC vials (Agilent) for analysis.

Soils were dried at room temperature until constant weight, sieved through a 2-mm nylon mesh, then ground to a fine powder. Prior to the extraction, soils (n = 3) were spiked with internal standards (40 µg kg^{-1} of D_4-AZOX and 60 µg kg^{-1} of D_5-BFT). Samples were then vortexed for 1 min and left at least one hour prior to extraction. The extraction method was adapted from Kah et al. (7) and consisted of shaking (rotary shaker, 20 rpm) 1 g of dried and sieved (2 mm) soil in 2 mL of ACN for 1 hour at room temperature. Samples were then centrifuged (1882 × *g*; 5 min, 20°C) and the supernatant was filtered through 0.22 µm filters into glass HPLC vials. Leachate solutions from the pots were sampled in the field using a glass syringe, stored in glass flasks, and filtered through 0.22 µm filters into glass HPLC vials.

E) LC-QTOF-MS instrumental analysis

Leachate, soil and plant extracts were analyzed on an Agilent 1290 Infinity II liquid chromatograph (LC) coupled to a 6545 QTOF mass spectrometer (Agilent Technologies, Santa Clara, USA) operating in positive electrospray ionization mode. The LC separation was conducted on a Poroshell 120 phenyl hexyl column (Agilent Technologies; 2.7 μm × 3.0 mm × 100 mm) fitted with a Poroshell 120 EC-C18 (2.7 μm × 3.0 mm × 5 mm) guard column. Elution was performed in gradient mode (0.4 mL min^{-1}) using A = water and B = Acetonitrile: Methanol (1:1), both containing 0.1% formic acid and 5 mM NH_4Ac (0 min: 70% A; 0-3 min: B increased from 30 to 100%; 3-6 min: 100% B; 6-8 min: B decreased from 100% to 30%). The injection volume was 10 μL and the column temperature was maintained at 30°C. Nitrogen was used as the drying gas (110°C, 12 L min^{-1}). Samples were run in the *All Ions MS/MS* mode. The fragmentor voltage was 110 V and MS data was acquired in the 50-750 *m/z* range. The following *m/z* were extracted from total ion chromatogram (TIC) (±10 ppm) for quantification: 404.1247 for AZOX and 440.1604 for BFT. The qualifier ions for AZOX and BFT are 372.0971 *m/z* and 181.1009 *m/z*, respectively.

F) Soil enzyme activity analysis

Glucosidases are widely responsible for the supply of energy in soil microorganisms through the decomposition of organic matter. **Phosphatases**, originating from soil microorganisms, hydrolyzes phosphorus into its bioavailable forms, which is important to maintain crop (8). The mineralization of sulfur, an essential element for plant growth, from organic sulfates is mediated by the hydrolase **arylsulfatase** (9). **Leucine aminopeptidase** are metallopeptidases that cleave N-terminal residues from proteins and peptides (10). Therefore,

these four enzymes provide a sensitive indicator of soil microbial changes which could be induced by nanomaterials or pesticides in agricultural soils (11).

The activity of soil phosphomonoesterase, arylsulfatase, β-D-glucosidase, and leucine-aminopeptidase were determined according to Peyrot et al. (12) using the fluorescent substrates 4-methylumbelliferone-phosphate (MUB-P), 4-methylumbelliferone-sulfate (MUB-S), 4-methylumbelliferone-glucopyranoside (MUB-C), and L-leucine-7-amino-4-methyl coumarin (AMC-N), respectively (Glycosynth, England). The fluorophores 4-methylumbelliferone (MUB) and 7-amino-4-methyl coumarin (AMC) were purchased from Sigma-Aldrich. Stock solutions of MUB (5 mM) and AMC (15 mM) were prepared in dimethylsulfoxide. Working solutions of the MUB and AMC (10.0, 8.0, 6.0, 4.0, 2.0, 1.0, 0.5, 0.1 μM) and the substrates (50 μM MUB-P, 100 μM MUB-C, 500 μM MUB-S, 50 μM AMC-N) were prepared in the buffer solution, using a similar pH as the soils (phosphate buffer, pH 7.2).

Enzymes were extracted from the soils by adding 0.5 g of soil (n = 3) to 25 mL of the buffer solution and then rotating the solutions for 30 min on a tube rotator (Fisher Scientific Tube Rotator) at 20 rpm. Mixtures were subsequently centrifuged for 5 min at 1882 × g and the supernatants were filtered over 0.22 μm filters into glass HPLC vials. For each sample, there were 6 analytical replicates, and after the addition of 150 μL of enzyme substrates and 50 μL of the soil extract solution to multiwell plates, samples were incubated under constant stirring (24 h, 30 °C). Fluorescence intensities were measured using excitation wavelengths of 330 nm (MUB) or 360 nm (AMC), with a fluorescent emission of 460 nm (Infinite M200, Tecan). The results were calculated by subtracting the average signal of both the blanks (soils) and the background wells from each sample. Enzyme activities were expressed as nmol MUB or AMC g^{-1} h^{-1} and

normalization was performed against the control samples (no treatment added) to obtain a relative percentage of enzyme activity.

G) Figures

Figure S1 Field set-up and the development of the strawberry plants over the duration of the experiment.

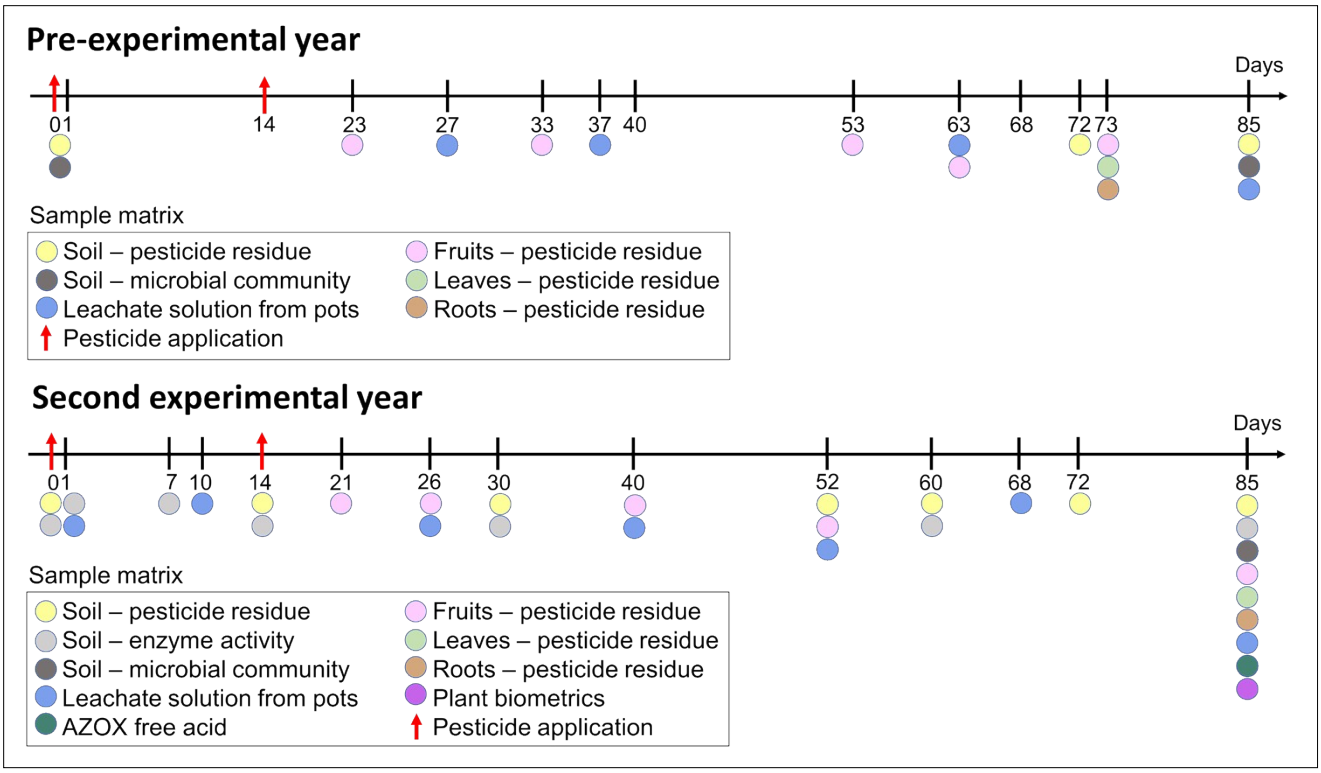


Figure S2 Overview of the sample collection timeline.

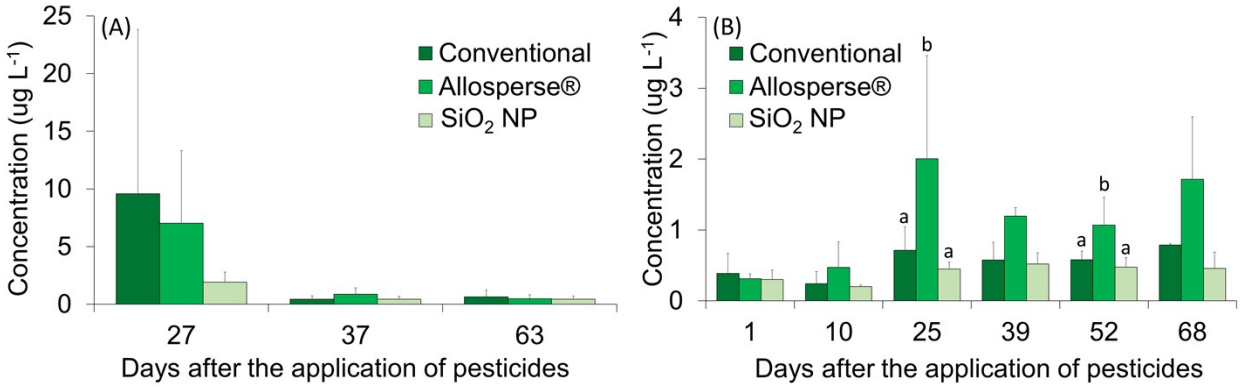


Figure S3 Azoxystrobin levels in the leachate solution sampled from each pot (n = 3) on different days counting from the application of the pesticides in the field. Data from the first experimental year are presented in (A), whereas datain (B).

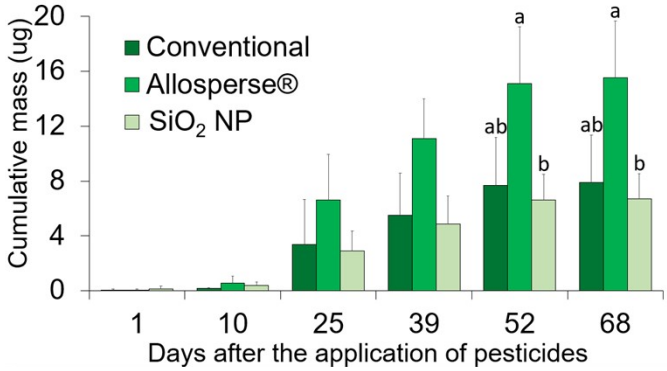


Figure S4 Cumulative azoxystrobin mass in the leachate solution sampled from each pot (n = 3) in different days counting from the pesticide application on field in the second experimental year.

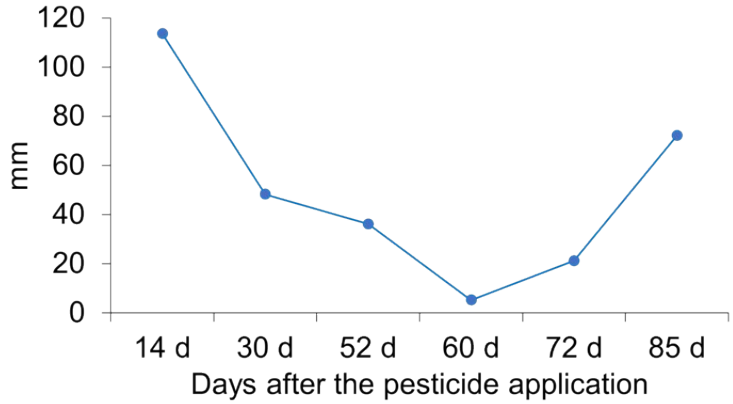


Figure S5 Cumulative precipitation records between the sampling days for the second growing season.

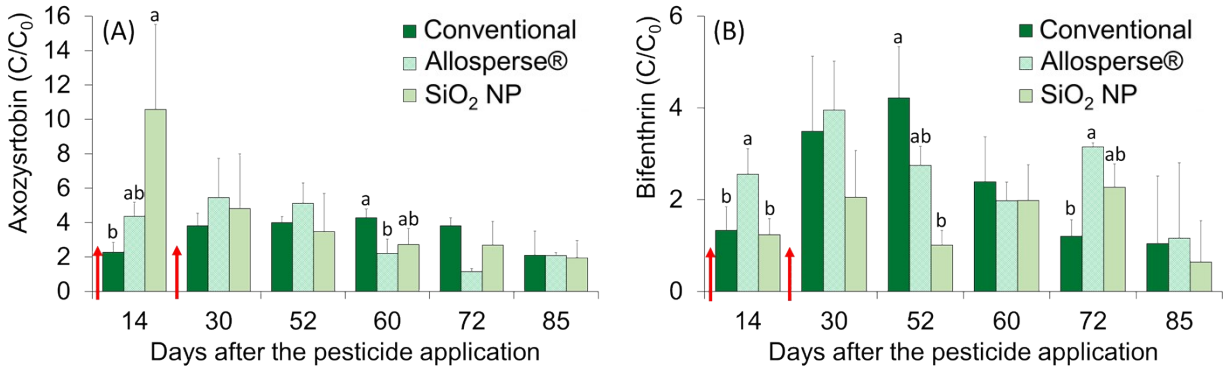


Figure S6 Normalized concentrations (concentration at a given time, C, divided by the concentration at day zero, C_0) of AZOX (A) and BFT (B) (conventional and nano formulations) in the soils in the second experimental year as a function of time following the application of the formulations. Red arrows indicate when the addition of the treatments to the soils occurred. Significant differences (ANOVA) between different formulations are represented by different letters, according to Fisher's least significant test. Data are means ± standard deviations (SD), n = 3.

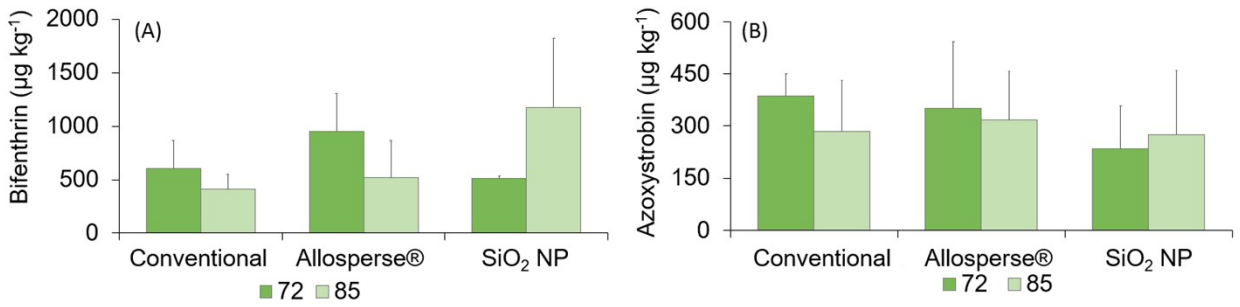


Figure S7 Concentrations of AZOX (A) and BFT (B) (conventional and nano) in the soils in the pre-experimental year as a function of time following the application of the pesticide formulations. Samples on day 14 were sampled just before the second application of the treatments to the soils. Statistically significant differences between the different formulations are represented by different letters, according to Fisher's least significant test. Data are means ± standard deviations (SD), n = 3.

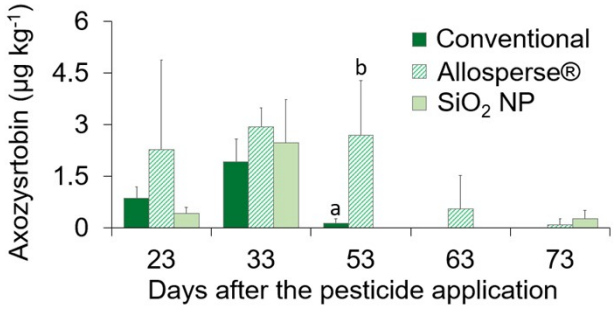


Figure S8 Concentration of Azoxystrobin (conventional and nano formulations) in the fruit samples from the first experimental year at different exposure times (days) counting from the first dosage application. Statistically significant differences between different formulations at the same sampling dates are represented by different letters, according to Fisher's least significant test. Data are means ± SD, n = 3.

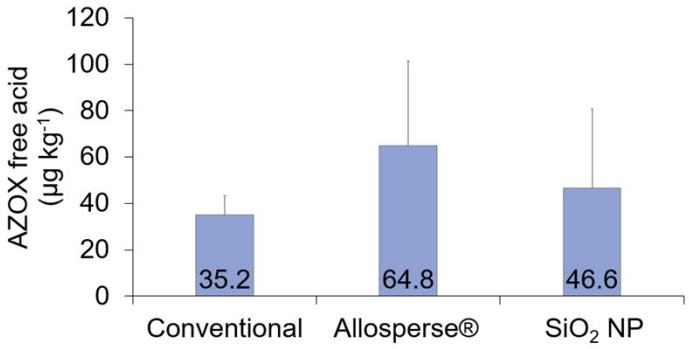


Figure S9 Concentrations of Azoxystrobin free acid in the strawberry roots sampled on the last day of the second experimental year. (Data are means ± SD, n = 3)

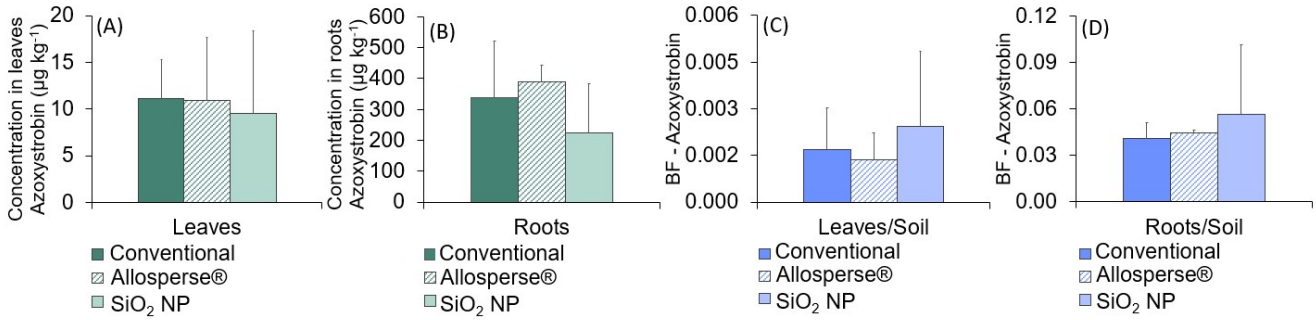


Figure S10 Concentrations of Azoxystrobin (conventional and nano formulations) in the leaves (A) and root samples (B) and bioaccumulation factors (BF, concentration in the plant divided by the concentration in the soils) from soil to leaves (C) and soil to roots (D) on day 85, i.e., the last sampling day of the second experimental year. No significant differences were found between the different formulations at $p < 0.05$. Data are means ± SD, n = 3.

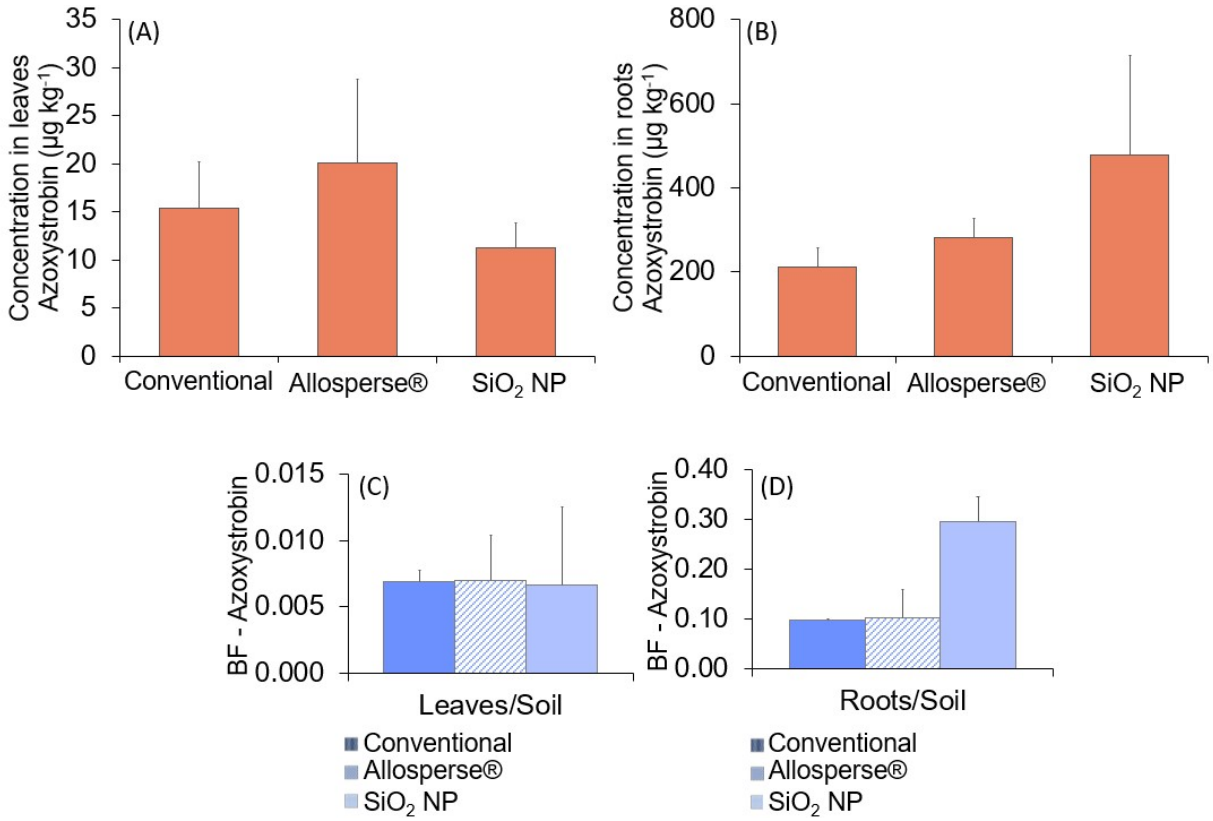


Figure S11 Concentration of azoxystrobin (conventional and nano formulations) in the leaves (A) and roots (B) and bioaccumulation factors (ratios of the concentrations in the plant tissue vs. soil) for leaves (C) and roots (D) from the last sampling day of the pre-experimental year. No significant differences were found between the different formulations at $p < 0.05$. Data are mean ± SD, n = 3.

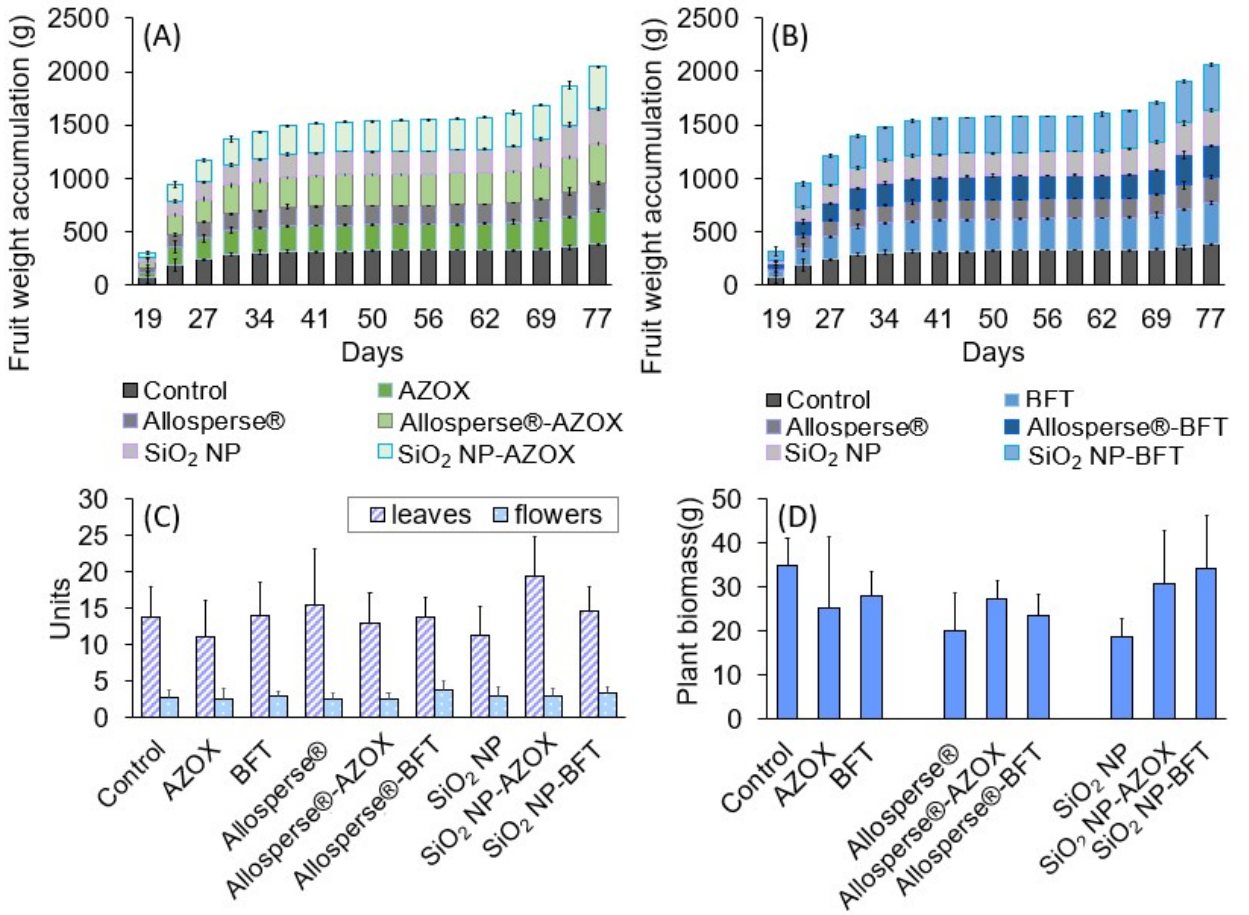


Figure S12 A) and B) Accumulation of the strawberry fruit (g/per pot) mass over time (days) counting from the first dosage application (A=AZOX; B=BFT) of the second experimental year. C) Number of flowers (units/per plant) and leaves for the strawberry plants at the end of the experiment. D) Biomass (g dry weight) of the strawberry plants analyzed at the end of the experiment. Control samples refer to the nanoparticle-free and pesticide-free samples. AZOX = Azoxystrobin; BFT = Bifenthrin.

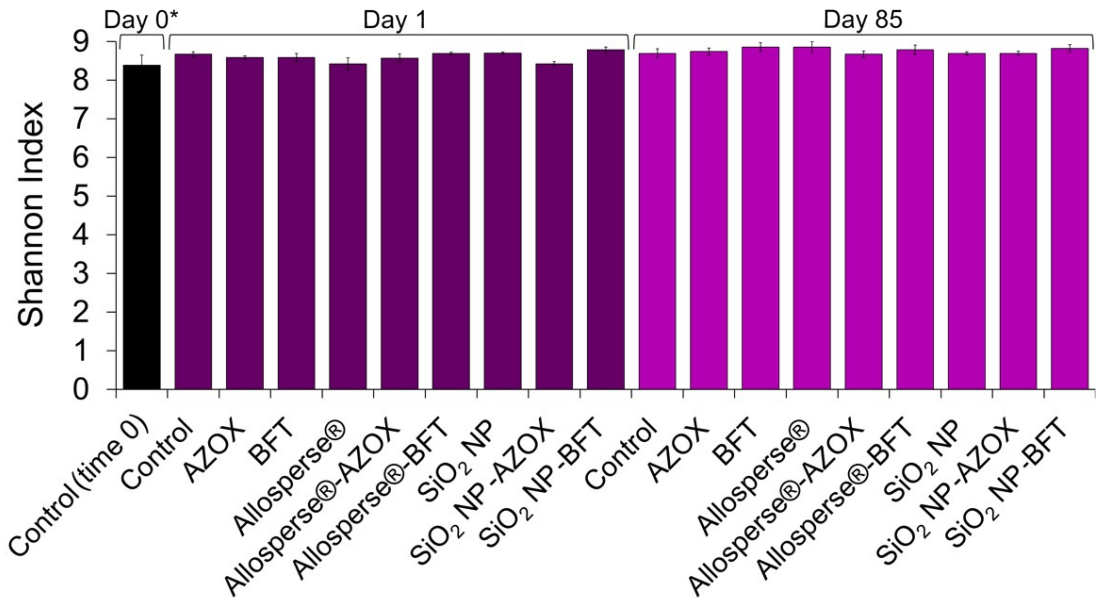


Figure S13 Shannon index for all of soil treatments from the second experimental year. *Day zero of the first experimental year. Days 0 and 85 of the second experimental year.

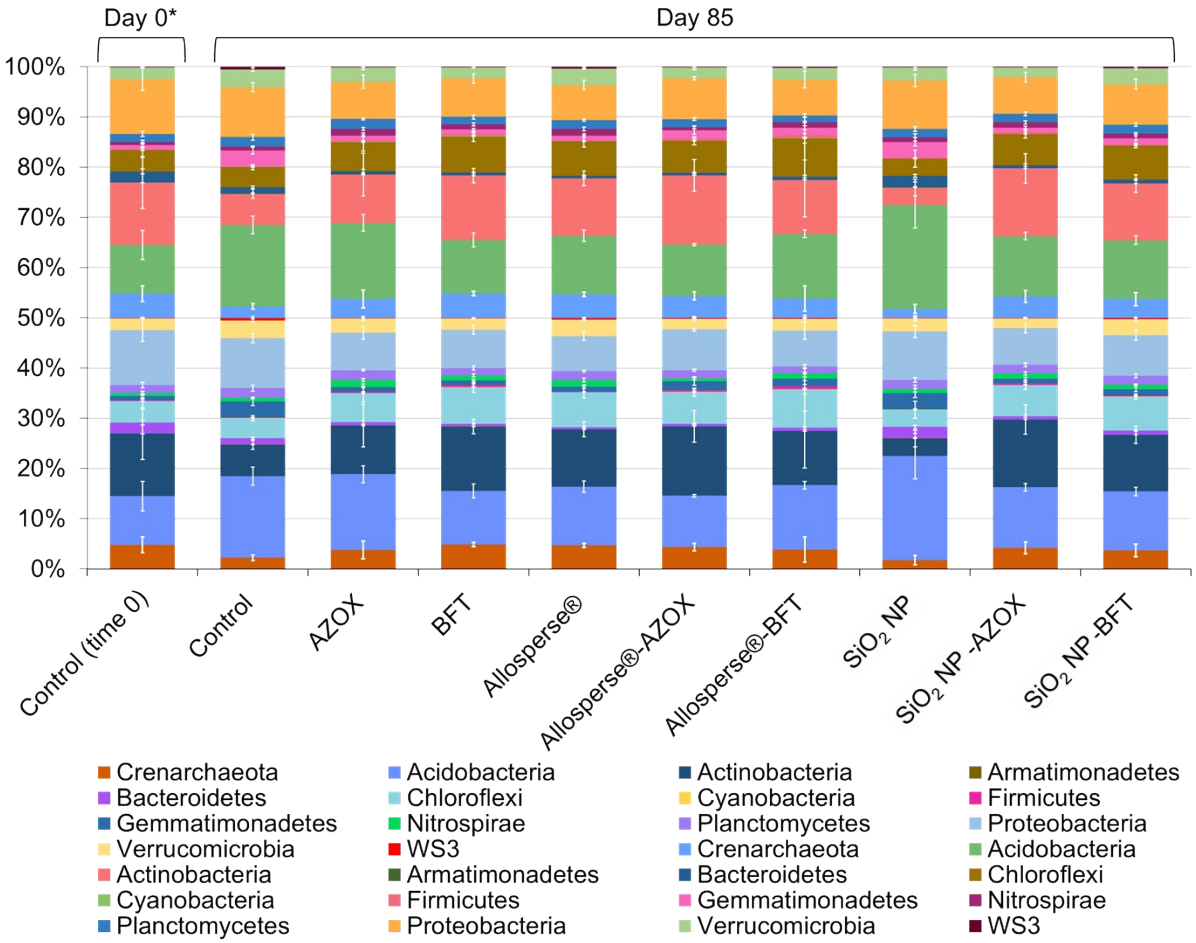


Figure S14 Relative abundance plot of the soil microbial community composition analyzed in the first experimental year. *Day zero of the first experimental year refers to the soils before the pesticide application.

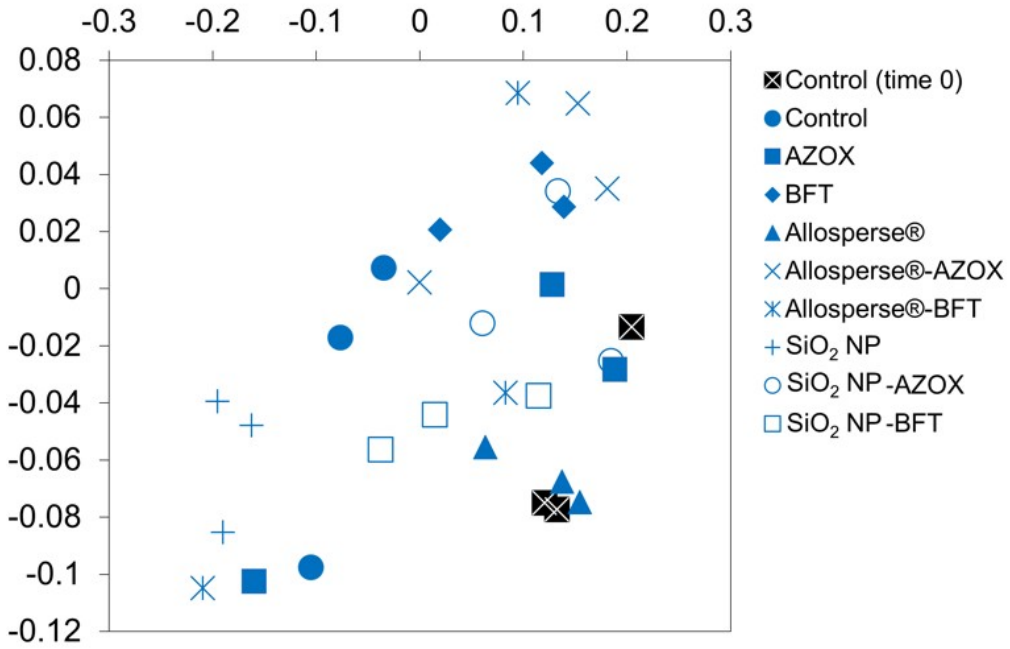


Figure S15 PCoA plot of the soil microbial community composition analyzed in the first experimental year.

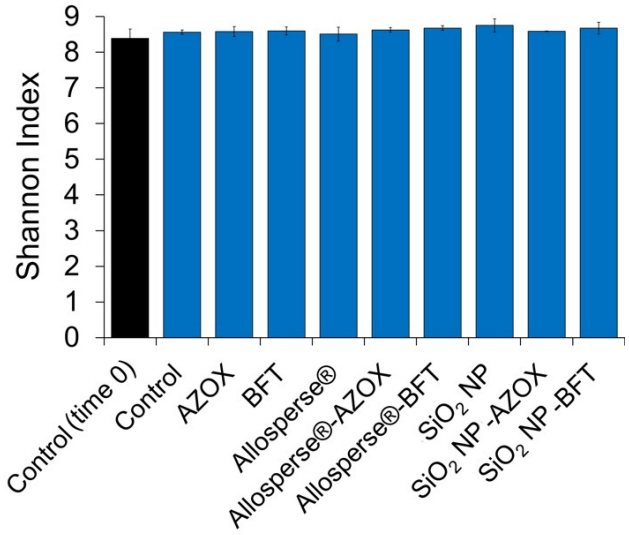


Figure S16 Shannon index for all of the soil treatments analyzed in the first experimental year.

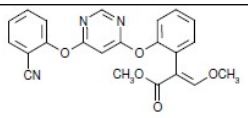
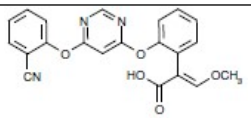
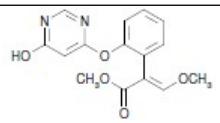
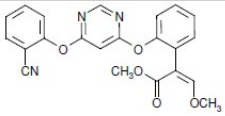
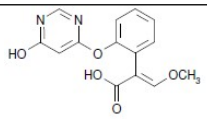
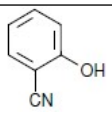
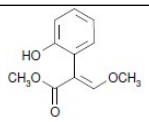
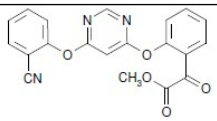
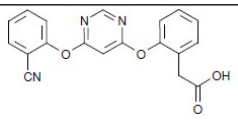
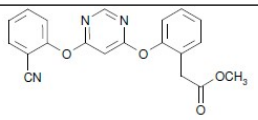
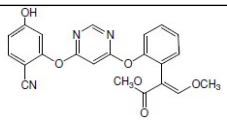
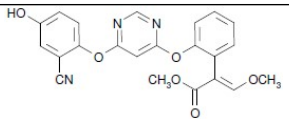
H) Tables

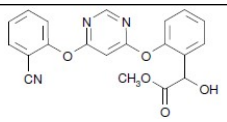
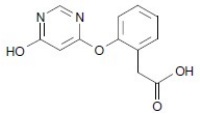
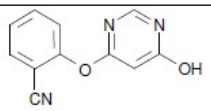
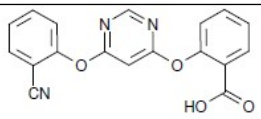
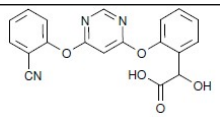
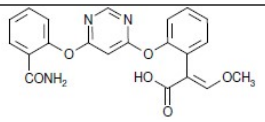
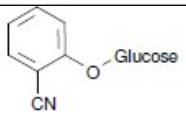
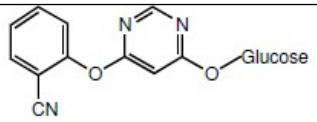
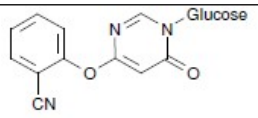
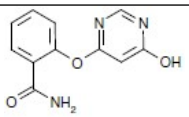
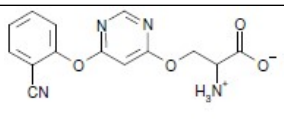
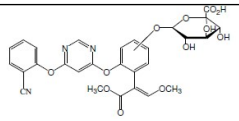
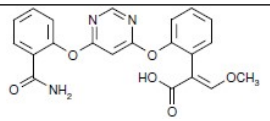
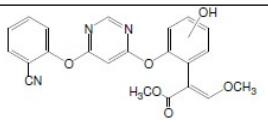
Table S1 - Transfer factors for the different pesticide formulations calculated on day 85 of the second experimental year.

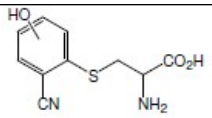
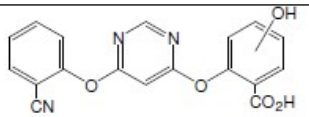
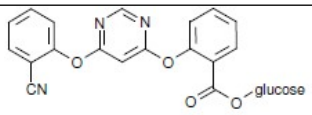
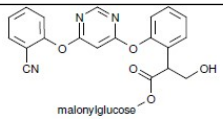
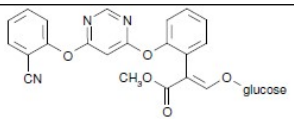
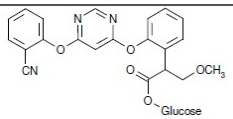
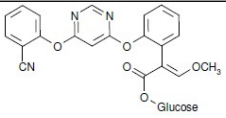
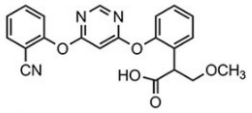
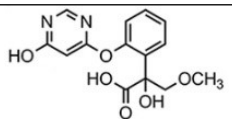
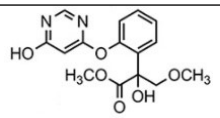
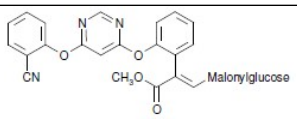
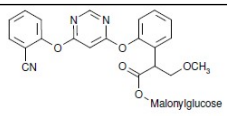
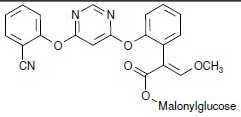
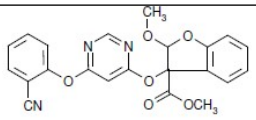
	Formulation	First experimental year	Second experimental year
$TF_{fruits/leaves}$ AZOX	Conventional	n.a.	$2.5 \times 10^{-2} \pm 2.2 \times 10^{-2}$
	Allosperse®	$1.1 \times 10^{-4} \pm 1.1 \times 10^{-4}$	$2.1 \times 10^{-2} \pm 1.3 \times 10^{-2}$
	$nSiO_2$	$3.6 \times 10^{-4} \pm 3.9 \times 10^{-4}$	$2.8 \times 10^{-3} \pm 3 \times 10^{-3}$
$TF_{fruits/roots}$ AZOX	Conventional	n.a.	$6.5 \times 10^{-4} \pm 5.9 \times 10^{-4}$
	Allosperse®	$4.6 \times 10^{-6} \pm 4.6 \times 10^{-6}$	$4.9 \times 10^{-4} \pm 1.9 \times 10^{-4}$
	$nSiO_2$	$1.2 \times 10^{-5} \pm 1.5 \times 10^{-5}$	$9.6 \times 10^{-5} \pm 1.1 \times 10^{-4}$
$TF_{leaves/roots}$ AZOX	Conventional	$7.5 \times 10^{-2} \pm 2.7 \times 10^{-2}$	$3.8 \times 10^{-2} \pm 2.1 \times 10^{-2}$
	Allosperse®	$7.7 \times 10^{-2} \pm 4.6 \times 10^{-2}$	$2.8 \times 10^{-2} \pm 1.6 \times 10^{-2}$
	$nSiO_2$	$2.6 \times 10^{-2} \pm 9.1 \times 10^{-3}$	$3.9 \times 10^{-2} \pm 8.5 \times 10^{-3}$

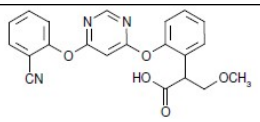
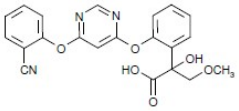
No significant differences were found between the different formulations at $p<0.05$. Data are mean ± SD, n = 3.
n.a. = not available since levels were below the MDLs in most samples.

Table S2- Azoxystrobin metabolites and degradation products (including identification number or letter, manufacturer code number, formula, *m/z* and structure) in the environment reported in the literature.

Compound[a]	Manufacturer code[b]	Formular	Molecular Weight	Structure	Reference
Compound 01 (azoxystrobin)	ICIA5504	$C_{22}H_{17}N_3O_5$	403.1168		(13)
Compound 02 (azoxystrobin free acid)	R234886	$C_{21}H_{15}N_3O_5$	389.1012		(13)
Compound 03	R219227	$C_{15}H_{14}N_2O_5$	302.0903		(13)
Compound 09	R230310	$C_{22}H_{17}N_3O_5$	403.1168		(13)
Compound 10	R232493	$C_{14}H_{12}N_2O_6$	304.0695		(13)
Compound 13	R71395	C_7H_5NO	119.0371		(13)
Compound 18	R176586	$C_{11}H_{12}O_4$	208.0736		(13)
Compound 19	R230309	$C_{20}H_{13}N_3O_5$	375.0855		(13)
Compound 20	R400050	$C_{19}H_{13}N_3O_4$	347.0906		(13)
Compound 21	R400051	$C_{20}H_{15}N_3O_4$	361.1063		(13)
Compound 22	R400297	$C_{22}H_{17}N_3O_6$	419.1117		(13)
Compound 23	R400299	$C_{22}H_{17}N_3O_6$	419.1117		(13)

Compound 24	R400753	$C_{20}H_{15}N_3O_5$	377.1012		(13)
Compound 26	R401487	$C_{12}H_{10}N_2O_4$	246.0641		(13)
Compound 28	R401553	$C_{11}H_7N_3O_2$	213.0538		(13)
Compound 30	R402173	$C_{18}H_{11}N_3O_4$	333.0750		(13)
Compound 35/U3	R402987	$C_{19}H_{13}N_3O_5$	363.0855		(13)
Compound 36	R403314	$C_{21}H_{17}N_3O_6$	407.1117		(13)
Compound 40	R405270	$C_{13}H_{15}NO_7$	297.0849		(13)
Compound 41	-	$C_{17}H_{17}N_3O_8$	391.1016		(13)
Compound 42	R405287	$C_{17}H_{15}N_3O_8$	389.0859		(13)
Compound C	-	$C_{11}H_9N_3O_3$	231.0644		(13)
Compound G2	-	$C_{14}H_{12}N_4O_4$	300.0859		(13)
Compound K1	-	$C_{29}H_{29}N_3O_{12}$	611.1751		(13)
Compound K2	-	$C_{21}H_{17}N_3O_6$	407.1117		(13)
Compound L1	-	$C_{23}H_{21}N_3O_6$	435.1430		(13)

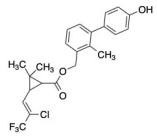
Compound L4	-	$C_{11}H_{14}N_2O_3S$	254.0725		(13)
Compound L9	-	$C_{19}H_{15}N_3O_5$	365.1012		(13)
Compound M1	-	$C_{24}H_{21}N_3O_9$	495.1278		(13)
Compound M2	-	$C_{29}H_{27}N_3O_{13}$	625.1544		(13)
Compound M3	-	$C_{27}H_{25}N_3O_{10}$	551.1540		(13)
Compound N1	-	$C_{27}H_{27}N_3O_{10}$	553.1696		(13)
Compound N2	-	$C_{27}H_{25}N_3O_{10}$	551.1540		(13)
Compound New M3	-	$C_{21}H_{17}N_3O_5$	391.1168		(14)
Compound New M4	-	$C_{14}H_{14}N_2O_6$	306.0852		(14)
Compound New M6	-	$C_{15}H_{16}N_2O_6$	320.1008		((14)
Compound O1	-	$C_{30}H_{27}N_3O_{13}$	637.1544		(13)
Compound O2	-	$C_{30}H_{29}N_3O_{13}$	639.1700		(13)
Compound O3	-	$C_{30}H_{29}N_3O_{13}$	639.1700		(13)
Compound U13	-	$C_{22}H_{17}N_3O_6$	419.1117		(13)

Compound U5	-	$C_{21}H_{17}N_3O_5$	391.1168		(13)
Compound U6	-	$C_{21}H_{17}N_3O_6$	407.1117		(13)

[a] The compound: number and letters were commonly used in the literature, except the "new M3, M4, and M6", which is found in the study of (14).

[b] Manufacturer codes of azoxystrobin metabolites were usually used as compounds ID in the literature.

Table S3 - Bifenthrin metabolites and degradation products (including compounds name, formula, *m/z* and structure) in the environment reported in the literature.

Compound	Formular	Structure	Molecular Weight	Reference
4'OH-BFT	$C_{23}H_{22}ClF_3O_3$		438.12096	(15, 16)
TFP acid	$C_9H_{10}ClF_3O_2$		242.03214	(15, 16)
Biphenyl alcohol (BP alcohol)	$C_{14}H_{14}O$		198.10447	(15, 16)
BP aldehyde	$C_{14}H_{12}O$		196.08882	(15, 16)
Biphenyl acid (BP acid)	$C_{14}H_{12}O_2$		212.08374	(15, 16)

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