

Dicamba residue persistence in processing tomato

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Research Article

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Abstract

There is zero tolerance for dicamba and dicamba metabolite residue in tomato (*Solanum lycopersicum* L.) fruit following exposure to dicamba. Field trials were conducted in 2020 and 2021 to determine the persistence of dicamba and metabolite (5-hydroxy dicamba and 3,6-dichlorosalicylic acid [DCSA]) residue in processing tomato shoots and fruits. Dicamba was applied 49 d after transplanting at 0, 0.53, 5.3, and 53 g ae ha⁻¹. Tomato plants were harvested 5, 10, 20, 40, and 61 d after treatment (DAT). No 5-hydroxy dicamba was recovered from any sample. In 2020, the DCSA metabolite was detected from tomato shoot tissue when dicamba was applied at the 53 g ha⁻¹ rate at 0 (14 µg kg⁻¹), 5 (3 µg kg⁻¹), and 20 DAT (5 µg kg⁻¹) and from tomato fruit tissue at 53 g ha⁻¹ at 20 (2 µg kg⁻¹) and 61 DAT (2 µg kg⁻¹). In 2021, DCSA was not detected from tomato shoot or fruit tissues at any harvest date. By 5 DAT, dicamba was only detected from tomato shoot tissues treated with 53 g ha⁻¹. At 0 DAT, dicamba residue was detectable only from tomato fruit on plants treated with 53 g ha⁻¹. Tomato fruit dicamba residue from plants treated with 5.3 g ha⁻¹ had a predicted peak of 19 µg kg⁻¹ at 11.3 DAT. Tomato fruit dicamba residue from plants treated with 53 g ha⁻¹ decreased from 164 to 8 µg kg⁻¹ from 5 to 61 DAT. Furthermore, this study confirms that dicamba is detectable from tomato fruits at 61 DAT following exposure to 5.3 or 53 g ha⁻¹ dicamba. Growers who suspect dicamba exposure should include tomato fruit tissue with their collected sample or sample tomato fruits separately.

Introduction

Dicamba use in the United States increased 6-fold between 2015 and 2019 (USGS 2021), and the increase can be attributed to applications made in soybeans [*Glycine max* (L.) Merr.] and cotton (*Gossypium hirsutum* L.). Portions of the north-central United States experienced the greatest dicamba use in 2019, equivalent to >6.6 g ae ha⁻¹ averaged across all cropped and non-cropped land (USGS 2021). The increase in dicamba usage has resulted in a corresponding increase in dicamba exposure to sensitive crops (USEPA 2021). In the 2021 growing season, there were 3,461 incidence reports of off-target dicamba injury to sensitive plant species, including fruit and vegetable crops (USEPA 2021). Concurrently, results from a 2021 survey of specialty crop producers in the north-central United States revealed that 69% are more concerned about herbicide drift than they were 5 yr earlier, and 66% reported confirmed or suspected drift between 2016 and 2020 (D Doohan, unpublished data). Of the survey respondents, 31% grew solanaceous crops, including tomatoes (*Solanum lycopersicum* L.). Indiana is the leading tomato-producing state in the midwestern United States. In 2017, processing tomato production in Indiana consisted of more than 2,800 ha across 74 farms with a value of \$28 million (USDA-NASS 2017).

Tomato sensitivity to herbicides, including dicamba, is well documented. Knezevic et al. (2018) reported that at 28 d after treatment (DAT) 50% injury of ‘Better Boy’ tomato plants was achieved with 3.98 to 5.35 g ha⁻¹ dicamba. Kruger et al. (2012) reported that the response of processing tomato varieties exposed to 0.56 to 56 g ha⁻¹ varied according to the stage of crop development (early vegetative vs. early flowering) and that dicamba exposure can result in tomato flower abortion, reduced yield, and delayed fruit ripening. Zangouinejad et al. (2019) reported as much as 89% and 99% crop injury to ‘Money Maker’ and Better Boy tomato at 28 DAT with 3 g ha⁻¹ dicamba. In addition to crop injury following exposure to low-dose dicamba, commercial producers also must be aware of potential dicamba residue in tomato fruit.

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There are currently only two vegetable crops with an allowable level of dicamba and dicamba metabolite residue on the raw agricultural commodity at the point of sale: asparagus (*Asparagus officinalis* L.) (4.0 mg kg⁻¹) and sweet corn (*Zea mays* L.) (0.04 mg kg⁻¹) (U.S. Code of Federal Regulations 2022). By default, all other fruit and vegetable crops have a zero tolerance. Zero-tolerance residue limits are established for one of four reasons: (1) a safe level of a pesticide has not been determined; (2) the chemical is carcinogenic or results in other physiological effects when fed to test animals; (3) toxicity of a pesticide is documented, but it is normally used in a manner that raw agricultural commodities will not contain it; and (4) all pesticide residue is normally removed through good agricultural practices or weathering before the raw agricultural commodity is offered for sale. In the case of tomato, the zero tolerance for dicamba exists because dicamba is not registered for use in the crop and, according to federal regulation, it should therefore not contain dicamba residue.

Given the zero tolerance for dicamba and dicamba metabolite residues in processing tomato, the impact of off-target dicamba exposure is not limited solely to a potential reduction in crop yield and delayed crop maturity. Instead, off-target dicamba exposure resulting in detectable concentrations of dicamba or its metabolites will result in a crop-destruct scenario and a complete crop loss. Recent studies in watermelon [*Citrullus lanatus* (Thunb.) Matsum. & Nakai] and sweetpotato [*Ipomoea batatas* (L.) Lam.] document that exposure to reduced rates of dicamba resulted in detectable dicamba residues in raw agricultural commodities. Culpepper et al. (2018) reported that dicamba applied at 2.2 and 7.5 g ha⁻¹ to watermelon 40 or 60 d after transplanting resulted in dicamba residue of 0.01 to 0.03 mg kg⁻¹ in watermelon fruits. Shankle et al. (2021) reported that dicamba residues ranging from 5.3 to 14.3 µg kg⁻¹ were recovered from sweetpotato storage roots following applications of 8.65 to 70 g ha⁻¹ of dicamba to sweetpotato plants 5 or 7 wk after transplanting.

Growers who suspect a tomato field was subjected to an off-target dicamba exposure event need to know how best to sample the field and how to interpret lab results related to dicamba and its metabolite residues. Additionally, growers need a more quantitative understanding of how dicamba residue persists in the crop to forecast whether tomato plants exposed during the growing season will yield fruit with detectable concentrations. Based on the interactions of the authors with specialty crop producers, producer consensus is that it is not possible to detect dicamba residue once exposed plants become symptomatic. If this is true, the specialty crop farmer may not be able to fully document the off-target movement event(s). Research-based information on the persistence of dicamba and its metabolites in both tomato shoots and fruit is lacking. Sirons et al. (1982) reported the persistence of dicamba residue on tomato plants exposed to five rates of dicamba ranging from 1.1 to 112 g ha⁻¹, but the studies were conducted in a growth chamber, and it is unclear whether the homogenized plant sample used for residue testing included tomato fruits. Zangouejad et al. (2020) applied 2.8 g ha⁻¹ dicamba to susceptible and resistant tomato lines in a greenhouse study and documented differences in herbicide uptake and partitioning within young plants at 1, 3, and 7 DAT, but did not report on dicamba residue partitioning in tomato fruits. Therefore, the objective of this study was to determine the persistence of dicamba and select metabolites from tomato shoot and fruit tissues following exposure to reduced rates of dicamba under field conditions.

Materials and Methods

Trials were conducted at commercial processing tomato fields in 2020 and 2021. In 2020, the trial was conducted near Fairmount, IN (40.3931°N, 85.7647°W) on a Pewamo silty clay loam (fine, mixed, active, mesic Typic Argiaquolls) soil with 3.3% organic matter (OM) and pH 7.1. The site in 2021 was located near Swayzee, IN (40.4366°N, 85.7876°W) on a Pewamo silty clay loam with 3.9% OM and pH 7.0. Tomato varieties '611' and '331' were transplanted on June 18, 2020, and May 21, 2021, respectively, in a double-row configuration with 30 cm between rows and 45 cm between plants within each row. Each set of double rows was spaced 1.5 m apart on-center. Plots consisted of a single double row that was 7.6-m long in 2020 and 5.5-m long in 2021. To limit cross-contamination of plots, there was a nontreated buffer row between each treated row.

Dicamba (XtendiMax®, Bayer CropScience, St Louis, MO, USA) was applied 49 d after transplanting on August 6, 2020, and July 9, 2021, at four rates: 0 (nontreated check), 0.53, 5.3, and 53 g ae ha⁻¹. These rates were based on a 1× rate of 530 g ha⁻¹ and equated to 1/1,000×, 1/100×, and 1/10× rates, respectively. Dicamba applications were made using a CO₂-pressurized backpack sprayer equipped with a two-nozzle boom fitted with TeeJet® TTI11002 tips (Spraying Systems, Wheaton, IL, USA) and calibrated to deliver 140 L ha⁻¹ at 193 kPa. At the time of application, tomato plants were in the initial reproductive stage of growth and contained an average of 9.8 fruits plant⁻¹ with a mean fruit size of 44 g. The experimental design was a randomized complete block with four replications. In 2020, plots were allowed to remain in the field through 61 DAT, whereas the last observations made in 2021 occurred at 40 DAT.

A visual estimate of total crop injury was recorded at 5, 20, 40, and 61 DAT on a scale of 0% to 100% relative to the nontreated check, where 0% indicates no crop injury and 100% indicates crop death. Aboveground plant tissues were sampled at 0, 5, 10, 20, 40, and 61 DAT. One tomato plant per plot was cut at the soil surface using a pair of pruners at each sample timing. Visible soil was removed from the plant by gently shaking the plant. Then tomato fruits were hand removed from the sample plant, and fruits and shoot tissues were placed into separate resealable plastic bags. At 20, 40, and 61 DAT, flowers were counted on each sampled plant before bagging. Bagged samples were placed into a cooler, covered with ice packs, and returned to the lab, where they were stored at -20 C until analysis. During sampling, gloves and pruners were changed between different dicamba rates to avoid cross-contamination.

Frozen samples were transported to the Mississippi State Chemical Laboratory (33.4517°N, 88.7888°W) for analysis using liquid chromatography with tandem mass spectrometry (LC-MS/MS) by a modified version of the quick, easy, cheap, effective, rugged, and safe (QuEChERS) method (AOAC 2007) to quantify residues of dicamba, 5-hydroxy dicamba, and 3,6-dichloro-2-hydroxybenzoic acid (DCSA). Samples were homogenized, and a 5-g subsample was placed into a 50-ml polypropylene centrifuge tube (Corning 430291, Corning Life Sciences, Glendale, AZ, USA). Fifteen milliliters of homogenizing bead (Agilent 5610-2142, Agilent Technologies, Santa Clara, CA, USA) followed by 10 ml of HPLC-grade water (Optima water, Thermo Fisher Scientific Incorporated, Ottawa, ON, Canada) were added to each tube, and tubes were then placed in an automated homogenizer (GenoGrinder®, SPEX SamplePrep, Metuchen, NJ, USA) for 5

min. The samples underwent a derivatization process by adding a basic solution (0.6 M NaOH, SS272-20, Fisher Scientific, Fair Lawn, NJ, USA) at 0.200 ml and were then allowed to rest for 30 min at room temperature. Then, 0.200 ml of an acidic solution (0.6 M HCl, SA50-20, Fisher Scientific) was added to neutralize the samples, and samples were placed into a heat bath (TSCIR19, Thermo Fisher Scientific Incorporated, Waltham, MA, USA) at 65 C for 30 min. Samples were removed from the heat bath and allowed to cool to room temperature. Ten milliliters of 1% formic acid in acetonitrile (Optima A117-50, Fisher Scientific) were added to the sample tube and placed in the homogenizer for 5 min; this was followed by the addition of QuEChERS citric salt (4,000 mg magnesium sulfate, 1,000 mg sodium chloride, 500 mg sodium citrate dibasic sesquihydrate, and 1,000 mg sodium citrate tribasic dihydrate; ECQUEU7-MP, UCT, Bristol, PA, USA) to accelerate the separation of the aqueous and organic phases and homogenization for 5 min. Samples were then placed into a centrifuge (Eppendorf 5810R, Eppendorf North America, Enfield, CT, USA) for 10 min at 4,000 rpm, and the extract liquid was decanted into a new 15-ml polypropylene centrifuge tube (Corning 430790, Corning Life Sciences). Approximately 1 ml of the extracted liquid was filtered into an autosampler vial (Agilent 5182-0715, Agilent Technologies) with a Millex Fluoropore polytetrafluoroethylene syringe filter (SLFCX13NK, MilliporeSigma, Burlington, MA, USA) and analyzed using an Agilent 1290 liquid chromatograph (Agilent Technologies) coupled to an Agilent 6460 mass spectrometer (Agilent Technologies). Quantification was completed using a multipoint calibration curve ($R^2 \geq 0.998$), and no result was reported or used if the results fell outside the multipoint calibration curve. Liquid chromatograph conditions, solvent gradients, and MS conditions and transitions are described in Table 1.

To determine the effect of dicamba rate, the data for crop injury, flower number, fruit number, and fruit weight were subjected to ANOVA using SAS PROC GLM (SAS 9.4, SAS Institute, Cary, NC) with the fixed effects of dicamba rate and random effects of year and replication within year. Crop injury data were subjected to arcsine square-root transformation, but were back-transformed to facilitate the interpretation of results. Crop injury of the nontreated check was excluded from ANOVA due to a lack of variance. When a significant ($P \leq 0.05$) dicamba rate-by-year interaction existed, data were analyzed separately by year.

Mean dicamba and metabolite residue data from tomato shoots and fruits were subjected to regression analysis with sampling timing (DAT) as the independent variable using the nonlinear curve-fitting function in JMP (JMP Pro v. 15, SAS Institute). Data were fit to either a two-parameter exponential model (Equation 1):

$$y = a * \exp(b * x) \quad [1]$$

where y is the predicted dicamba residue (in $\mu\text{g kg}^{-1}$), a is the y -intercept, b is the slope of the line, and x is days after dicamba application; or a gaussian peak model (Equation 2):

$$y = a * \exp\left(-\left\{0.5 \left[\frac{(x-b)}{c}\right]^2\right\}\right) \quad [2]$$

where y is the predicted dicamba residue (in $\mu\text{g kg}^{-1}$), a is the peak value, b is the DAT at which the peak value is predicted, c is the growth rate, and x is the days after dicamba application. To be a good fit, each parameter estimate of the model had to be significant ($P \leq 0.05$).

Results and Discussion

Tomato Injury

Injury data at 5 and 20 DAT were pooled across both 2020 and 2021. Injury data at 40 DAT were analyzed separately by year due a significant dicamba rate-by-year interaction ($P = 0.02$). Injury data at 61 DAT were only collected in 2020. Tomato crop injury presented as a combination of epinasty, stunting, leaf distortion, and necrosis (Figure 1). At 5 DAT, injury ranged from 6% to 29%, with each dicamba rate resulting in significantly greater injury than the previous rate (Table 2). At 20 DAT, tomato crop injury was minimal from 0.53 g ha^{-1} (3%), moderate from 5.3 g ha^{-1} (19%), and severe from 53 g ha^{-1} dicamba (47%). By 40 DAT, injury was $\leq 7\%$ at dicamba rates of 0.53 and 5.3 g ha^{-1} , but was 25% and 16% from 53 g ha^{-1} dicamba in 2020 and 2021, respectively. By 61 DAT in 2020, injury was $\leq 3\%$ for all rates of dicamba used in this study (data not shown).

Visible tomato injury in the present study was much lower than in previous reports. The maximum injury observed in this study was 47% at 20 DAT with 53 g ha^{-1} dicamba. At 21 DAT, Knezevic et al. (2018) reported 50% tomato injury at dicamba rates ranging from 3.98 to 5.35 g ha^{-1} . At 28 DAT, Zangouejnejad et al. (2019) reported more than 89% tomato injury from 3 g ha^{-1} dicamba.

Tomato Flower Number

Due to a lack of significant dicamba rate-by-year interaction, data for tomato flower number at 20 and 40 DAT were analyzed across both years; flower number at 61 DAT was only recorded in 2020. At 20 DAT, flower number was greatest in the nontreated check (26.5 flowers plant^{-1}) and 0.53 g ha^{-1} dicamba (24.5 flowers plant^{-1}) (Table 3). Tomato plants exposed to 53 g ha^{-1} dicamba had 1 flower plant^{-1} , which was statically similar to plants treated with 5.3 g ha^{-1} dicamba (9.3 flowers plant^{-1}). At 40 DAT, plants exposed to 53 g ha^{-1} had the most flowers (28.5 flowers plant^{-1}) and significantly more flowers than the nontreated check (10.4 flowers plant^{-1}). Flower number for plants exposed to 0.53 and 5.3 g ha^{-1} dicamba was statistically similar to both the nontreated check and 53 g ha^{-1} dicamba. By 61 DAT in 2020, flower number was greatest with 53 g ha^{-1} dicamba (27.8 flowers plant^{-1}), which was statically greater than all other treatments (1.3 to 8.5 flowers plant^{-1}). Results from the present study differ from those of Kruger et al. (2012), who estimated 5%, 10%, 25%, and 50% flower losses at 1.5, 2.7, 6.4, and 15.4 g ha^{-1} , respectively, applied at an early flowering stage of growth. In our study, we observed a flower reduction only at the 20 DAT observation, and flower reduction was 1%, 65%, and 96% at rates of 0.53, 5.3, and 53 g ha^{-1} . By 40 DAT, the date most similar to that used by Kruger et al. (2012), we observed a 34% to 174% increase in flower number in dicamba-treated tomatoes compared with the nontreated check.

Tomato Fruit Number and Weight

Due to a lack of significant dicamba rate-by-year interaction, data for fruit number and weight plant^{-1} were pooled across 2020 and 2021. Fruit weight per plant did not differ by dicamba rate at any of the sampling timings (data not shown). Tomato fruit number did not differ by dicamba rate at 0, 5, 10, or 61 DAT (Table 3). At 20 DAT, tomato fruit number of the nontreated check was 30.0 fruits plant^{-1} , which was statistically greater than tomato fruit number for plants exposed to 53 g ha^{-1} (12.3 fruits plant^{-1}). At 40 DAT, the nontreated check contained 37.9 fruits plant^{-1} , which was

Table 1. Liquid chromatograph conditions, solvent gradients, and mass spectrometry conditions and transitions for tomato shoot and fruit residue sampling.^a

LC conditions					
Column	Agilent ZORBAX Eclipse Plus C18, RR HT, 2.1 × 50 mm, 1.8 μm (cat. no. 959741-902) or equivalent				
Injection volume	5.00 μl				
Column temperature	40.00 C				
Flow rate	0.300 ml min ⁻¹				
Solvent A	100% water + 0.1% formic acid				
Solvent B	100% acetonitrile + 0.1% formic acid				
Stop time	5 min				
Post time	3 min				
Solvent gradients					
Time	% B				
0 min	10				
1 min	10				
3 min	90				
MS conditions					
Gas temperature	200 C				
Gas flow	10 L min ⁻¹				
Nebulizer	276 kPa				
Sheath gas temperature	350 C				
Sheath gas flow	11 L min ⁻¹				
Capillary	4,000 V				
MS transitions					
Analyte	Precursor ion	Product ion	Fragmentor	Collision energy	Polarity (+/-)
Dicamba	219.0	175.0	65	1	—
Dicamba	219.0	145.0	60	5	—
5-hydroxy dicamba	235.0	190.9	74	1	—
5-hydroxy dicamba	235.0	155.0	74	9	—
DCSA	204.9	160.9	79	5	—
DCSA	204.9	124.9	79	21	—

^aDCSA, 5-hydroxy dicamba and 3,6-dichlorosalicylic acid; LC, liquid chromatography; MS, mass spectrometry.



Figure 1. Processing tomato plants at 5 d after treatment (DAT) showing epinasty (A) and flower necrosis (B) from 53 and 5.3 g ha⁻¹ dicamba, respectively, in 2020. Leaf distortion at 20 DAT from 5.3 g ha⁻¹ dicamba in 2020 (C).

Table 2. Influence of dicamba rate on processing tomato injury in 2020 and 2021.

Dicamba rate g ae ha ⁻¹	Tomato plant injury ^a			
	5 DAT		20 DAT	
	2020 and 2021		2020	2021
	% ^b			
0.53	6 c	3 c	0 b	7 ab
5.3	13 b	19 b	3 b	5 b
53	29 a	47 a	25 a	16 a

^aInjury ratings: 0% = no injury; 100% = crop death. DAT, days after treatment.

^bMeans followed by the same lowercase letter do not differ from one another according to Fisher's protected LSD ($P \leq 0.05$).

Table 3. Influence of dicamba rate on processing tomato flower and fruit number per plant pooled across 2020 and 2021.^a

Dicamba rate g ae ha ⁻¹	Tomato flowers			Tomato fruit					
	20 DAT	40 DAT	61 DAT ^b	0 DAT	5 DAT	10 DAT	20 DAT	40 DAT	61 DAT ^b
	plant ⁻¹ c								
0	26.5 a ^c	10.4 b	1.3 b	9.5	20.0	20.3	30.0 a	37.9 ab	57.8
0.53	24.5 a	13.9 ab	0.3 b	10.3	14.5	18.3	23.8 a	46.1 a	61.3
5.3	9.3 b	14.8 ab	8.5 b	7.9	18.0	14.0	21.6 ab	30.3 ab	59.8
53	1.0 b	28.5 a	27.8 a	11.6	13.4	18.0	12.3 b	26.4 b	60.5

^aDAT, days after treatment.

^bTomato flower and fruit number data at 61 DAT were only collected in 2020.

^cMeans followed by the same lowercase letter do not differ from one another according to Fisher's protected LSD ($P \leq 0.05$).

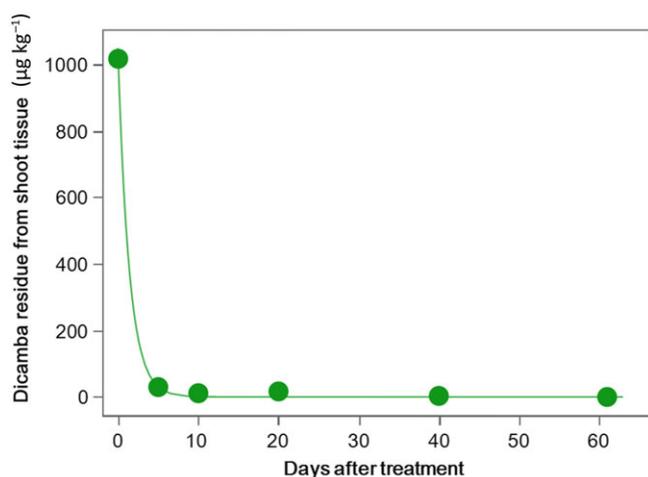


Figure 2. Persistence of dicamba residue from tomato shoot tissues treated with 53 g ae ha⁻¹ dicamba pooled across 2020 and 2021. Points represent the observed mean data; the line represents the predicted value based on a two-parameter exponential model (Equation 1). Parameter estimates with standard errors in parentheses: $a = 1,016.4$ (9.9); $b = -0.7031$ (0.0655); $R^2 = 0.99$.

statically similar to all of the other dicamba treatments. In the present study, we did not segregate tomato fruit into marketable and nonmarketable. Nor did we segregate the fruit into ripe and non-ripe. For this reason, direct comparisons with the findings of others as they relate to the effect of reduced-rate herbicide exposure on processing tomato fruit ripening and marketability is not possible.

Dicamba and Metabolite Residues

In 2020, the DCSA metabolite was only detected in tomato shoot tissue at the 53 g ha⁻¹ rate at 0 (14 µg kg⁻¹), 5 (3 µg kg⁻¹), and 20 DAT (5 µg kg⁻¹) and in tomato fruit tissue from 53 g ha⁻¹ dicamba

at 20 (2 µg kg⁻¹) and 61 DAT (2 µg kg⁻¹) (data not shown). In 2021, DCSA was not detected in tomato shoot or fruit tissues at any sampling date. No 5-hydroxy dicamba was detected in any shoot or fruit sample in either year.

No dicamba residue was detected from any nontreated check plant shoots or fruits. In plants treated with 0.53 g ha⁻¹ dicamba, dicamba residue (2 µg kg⁻¹) from tomato shoots was only detected in 2020 at the 0 DAT sampling timing (data not shown). Similarly, for tomato plants treated with 5.3 g ha⁻¹ dicamba, dicamba residue was only detected from tomato shoots at the 0 DAT sampling timing (43 and 10 µg kg⁻¹ in 2020 and 2021, respectively) (data not shown). Tomato shoot dicamba residue from plants treated with 53 g ha⁻¹ dicamba fit a two-parameter exponential model (Equation 1; Figure 2). Pooled across both years, predicted dicamba residue from tomato shoots decreased steeply from 1,016 to 30 µg kg⁻¹ between 0 and 5 DAT. Between 5 and 61 DAT, predicted dicamba residue from tomato shoot tissues decreased from 30 to 2.42×10^{-16} µg kg⁻¹. Similarly, when Sirons et al. (1982) applied of 22.4 g ha⁻¹ dicamba, they reported a decrease in dicamba residue from 1,007 µg kg⁻¹ to 370 µg kg⁻¹ from 0 to 7 DAT. In the present study, dicamba residue was not detectable from 5 to 61 DAT with applications of 5.3 or 0.53 g ha⁻¹. This result is similar to that of Sirons et al. (1982), who reported that dicamba applied at 1.1 g ha⁻¹ resulted in no detectable dicamba residue by 14 DAT.

The persistence of dicamba residue from tomato fruit tissue differed from that of tomato shoots. In plants treated with 0.53 g ha⁻¹ dicamba, dicamba residue was only detected once during the course of the study: 2 µg kg⁻¹ at 5 DAT in 2020 (data not shown). Dicamba residue data from plants treated with 5.3 g ha⁻¹ fit a gaussian peak model (Equation 2) with a predicted peak dicamba residue of 19 µg kg⁻¹ at 11.3 DAT (Figure 3). On either end of the peak model, predicted dicamba residue was 4 and 2×10^{-14} µg kg⁻¹ at 0 and 61 DAT, respectively. Dicamba residue from plants treated with 53 g ha⁻¹ were fit to a two-parameter exponential model

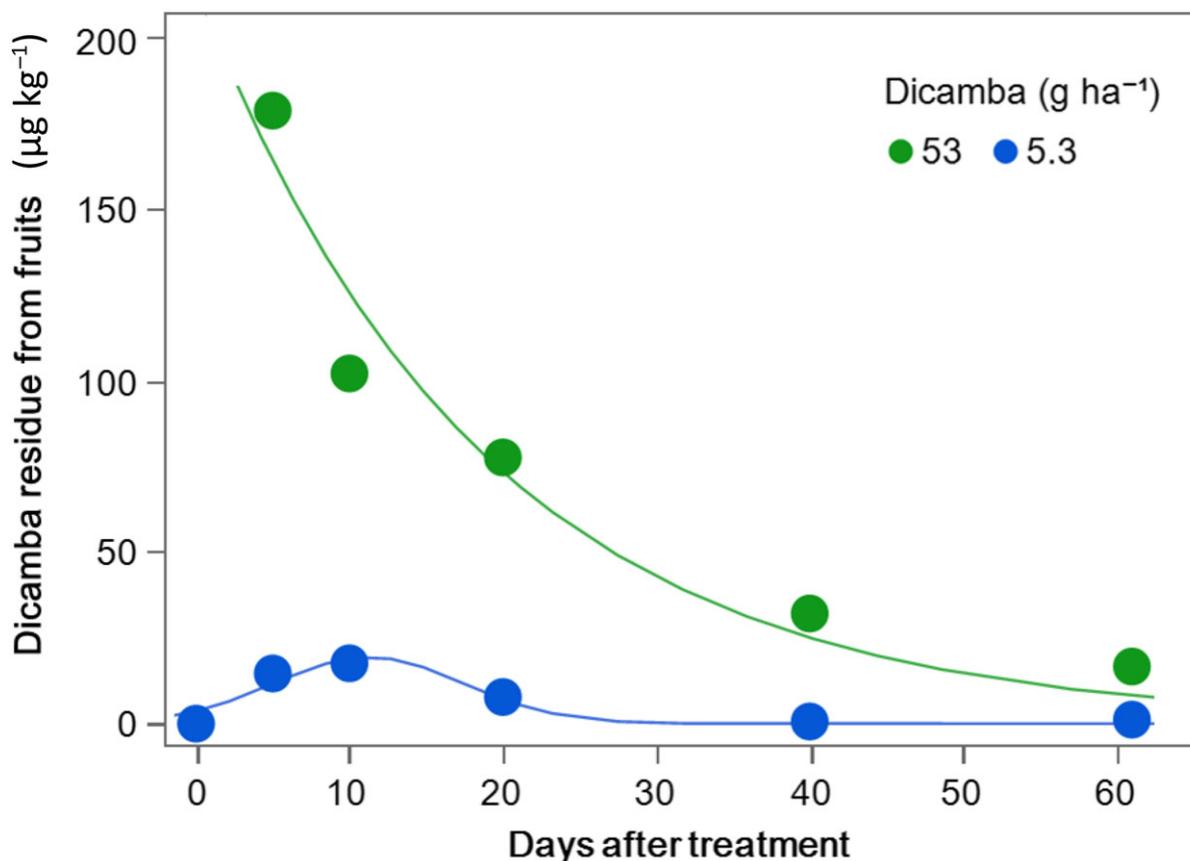


Figure 3. Persistence of dicamba residue on tomato fruits treated with 5.3 or 53 g ae ha⁻¹ dicamba pooled across 2020 and 2021. Points represent the observed mean data; the lines represent the predicted value based on a two-parameter exponential model (Equation 1) for the 53 g ha⁻¹ and a gaussian peak (Equation 2) for 5.3 g ha⁻¹. Parameter estimates with standard errors in parentheses when dicamba was 5.3 g ha⁻¹: $a = 19.4$ (3.2); $b = 11.3$ (1.2); $c = 6.2$ (1.0); $R^2 = 0.91$. Parameter estimates with standard errors in parentheses when dicamba was 53 g ha⁻¹: $a = 214.6$ (29.2); $b = -0.0537$ (0.0126); $R^2 = 0.95$.

(Equation 1), and predicted dicamba residue decreased from 164 to 8 µg kg⁻¹ from 5 to 61 DAT.

These results support the specialty crop grower concern that plant samples collected once dicamba symptoms are observed may not contain detectable levels of dicamba or dicamba metabolite residues. In this study, dicamba at reduced rates of 0.53 to 5.3 g ha⁻¹, equivalent to 1/1,000× and 1/100× rates, respectively, were only detectable from tomato shoots on the day of exposure. Dicamba residue from tomato fruits was more persistent than residue from the shoot tissues. Our findings suggest that growers who suspect dicamba exposure should include tomato fruit tissue with their collected sample or sample tomato fruits separately.

Based on the findings of this study, tomato fruit on plants exposed to 5.3 and 53 g ha⁻¹ of dicamba contained minute, but detectable amounts of dicamba residue through 61 DAT, rendering them not marketable. The authors propose a modification to the current zero-tolerance rule to allow for some level of dicamba residue to be permitted in cases of off-target exposure. Currently tolerances for dicamba residue exist for numerous agricultural commodities, including sweet corn (0.04 mg kg⁻¹); asparagus (4.0 mg kg⁻¹); milk (0.2 mg kg⁻¹); and cattle, hog, and sheep meat (0.25 mg kg⁻¹) (U.S. Code of Federal Regulations 2022). Establishing a low-level tolerance for dicamba and dicamba metabolite residues in cases of off-target dicamba exposure would alleviate some of the financial burden on tomato growers and applicators deemed responsible for an off-target movement event.

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References

- [AOAC] Association of Official Analytical Chemists (2007) AOAC Official Method 2007.1: Pesticide Residues in Foods by Acetonitrile Extraction and Partitioning with Magnesium Sulfate. https://nucleus.iaea.org/sites/fcris/Shared%20Documents/SOP/AOAC_2007_01.pdf. Accessed: April 12, 2022
- Culpepper AS, Sosnoskie LM, Shugart J, Leifheit N, Curry M, Gray T (2018) Effects of low-dose applications of 2,4-D and dicamba on watermelon. *Weed Technol* 32:267–272
- Knezevic SZ, Osipitan OA, Scott JE (2018) Sensitivity of grape and tomato to micro-rates of dicamba-based herbicides. *J Hortic* 5:229
- Kruger GR, Johnson WG, Doohan DJ, Weller SC (2012) Dose response of glyphosate and dicamba on tomato (*Lycopersicon esculentum*) injury. *Weed Technol* 26:256–260
- Shankle MW, Harvey LM, Meyers SL, Morris CJ (2021) Sweetpotato response to reduced rates of dicamba. *Weed Technol* 35:748–752
- Sirons GJ, Anderson GW, Frank R, Ripley BD (1982) Persistence of hormone-type residue in tissue of susceptible crop plants. *Weed Sci* 30:572–578

- U.S. Code of Federal Regulations (2022) Dicamba; tolerances for residues. 40 U.S.C. § 180.227. <https://www.ecfr.gov/current/title-40/chapter-I/subchapter-E/part-180/subpart-C/section-180.227>. Accessed: April 4, 2022
- [USDA-NASS] U.S. Department of Agriculture–National Agricultural Statistics Service (2017) Table 36. Vegetables, Potatoes, and Melons Harvested for Sale: 2017 and 2012. https://www.nass.usda.gov/Publications/AgCensus/2017/Full_Report/Volume_1,_Chapter_1_State_Level/Indiana/st18_1_0036_0036.pdf. Accessed: April 4, 2022
- [USEPA] U.S. Environmental Protection Agency (2021) Status of over-the-top dicamba: summary of 2021 usage, incidents and consequences of off-target movement, and impacts on stakeholder-suggested mitigations. <https://www.regulations.gov/document/EPA-HQ-OPP-2020-0492-0021>. Accessed: August 24, 2022
- [USGS] U.S. Geological Survey (2021) Estimated Annual Agricultural Pesticide Use. https://water.usgs.gov/nawqa/pnsp/usage/maps/show_map.php?year=2019&map=DICAMBA&hilo=L. Accessed: April 4, 2022
- Zangouejad R, Alebrahim MT, Tseng TM (2019) Evaluation of auxin tolerance in selected tomato germplasm under greenhouse and field conditions. *Weed Technol* 33:815–822
- Zangouejad R, Alebrahim MT, Tseng T (2020) Absorption and translocation of dicamba in dicamba-tolerant wild tomato. *Can J Plant Sci* 101:30–38