

Dicamba volatility assessment in a tropical environment

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Abstract: Background: The use of the herbicide dicamba has significantly increased in Brazilian agriculture, has potential for volatilization and risk of injury to sensitive agricultural crops and environmental contamination. **Objective:** The objective of this research was to evaluate the behavior of dicamba volatility under application conditions in commercial crops in tropical climate conditions in Brazil. **Methods:** Six field experiments were conducted from 2018 to 2020 in 3 different states of Brazil to provide a quantitative assessment of dicamba volatility. Aerodynamic and integrated horizontal flux methods were used to estimate the volatile flux and mass loss of dicamba within

72 hours after application. **Results:** The results showed that the flux of volatilized dicamba peaked in the first few hours after application, decreasing drastically approximately 20 hours after application. The cumulative mass loss of dicamba was $0.15 \pm 0.08\%$ of the applied dose. The average in-field air concentration ($38.66 \pm 9.52 \text{ ng m}^{-3}$) represented only 28% of the regulatory no observable adverse effect concentration (NOAEC) established by the USEPA. **Conclusions:** Based on these results, vapor drift related to dicamba volatility under application conditions in tropical regions in Brazil was similar to or lower than the results reported in the literature for temperate climate regions.

Keywords: Auxinic Herbicide; Off-Target Movement; Vapor Drift; Volatilization

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1. Introduction

Herbicide use is the main method of weed control present in production areas, with glyphosate as one of the most used herbicides to control weeds in grain cultivation (Constantin et al., 2013). However, due to intense and continuous glyphosate use, many weeds are increasingly acquiring resistance to this chemical (Moreira et al., 2010; Mendes et al., 2012; Schneider, 2018; Baldini et al., 2021). Therefore, the use of glyphosate combined with different herbicides through cropping systems with glyphosate-resistant transgenic stacked with other mechanisms of action is increasing, with emphasis on the use of auxin mimicking herbicides. These herbicides act mainly on dicotyledonous weeds (broadleaves), with dicamba as one of the molecules that fall in this class (Mithila et al., 2011).

Dicamba (3,6-dichloro-2-methoxybenzoic acid) belongs to the benzoic acid chemical group. This compound has postemergence activity with a long history of use in some geographies including the United States and Argentina. This herbicide is being used again in Brazil after a few decades what leads to some challenges due to the lack of agronomic experience in using this herbicide appropriately and sustainably for weed control. This is particularly significant since reports of off-target movement of dicamba have been re-reported in the United States of America, the first country to launch dicamba-tolerant (DT) soybean varieties (US Environmental Protection Agency, 2020).

When receiving deposits of synthetic auxins, the initial effects on sensitive dicotyledonous plants are mainly phenomena that affect plant growth, such as epinasty. This phenomenon can be followed by chlorosis and subsequent tissue necrosis (Alves et al., 2021). Some cultivated plants are extremely sensitive to hormonal herbicides; therefore, care should be taken to avoid drift in this type of application. The best practices and the best adjustments in the application technology parameters must be followed, such as the selection of the appropriate spray tip and volume, as well as the weather conditions and the characteristics of each type of spray formulation of these herbicides (Radons et al., 2021).

New technologies of genetically modified soybean plants are being introduced with tolerance to auxins such as dicamba and 2,4-D. The mixture of dicamba and glyphosate is an important tool in the management of broadleaf resistant weeds and has been

extensively studied in terms of control effectiveness and off-target movement (Mueller, Steckel 2019; Egan, Mortensen, 2012; Riter et al., 2020; Sall et al., 2020; Carbonari et al., 2020; Bish et al., 2019).

Dicamba is a weak acid (pKa 1.87) that is formulated as a salt (Carbonari et al., 2020). A small portion of dicamba DGA can dissociate, and the acid form may be lost via volatilization to the atmosphere; hence, strategies to avoid acid formation are essential for the mitigation of volatilization (Bish et al., 2019; Carbonari et al., 2022a). Dicamba is classified as moderately volatile and has the potential to cause adverse effects in nontarget plants if not used appropriately (Riter et al., 2012; Carbonari et al., 2022b).

Some alternatives can be used to reduce the potential for losses caused by the drift of dicamba through volatilization. The choice of salt that is used to neutralize dicamba acid has been an area of research with a focus on selecting those that further reduce volatility. Dicamba formulated as dimethylamine salt has been reported to have a negative effect on soybean at a 0.1% dose 21 m from the application area (Egan, Mortensen, 2012). The use of diglycolamine salt (DGA) has shown better results in terms of volatilization (94% reduction), consequently reducing the potential effects of low dicamba concentrations created by this type of drift in sensitive cultures (Mueller et al., 2013). By associating adjuvants with the DGA formulation, an improvement in volatilization was also identified (Mueller, Steckel, 2019).

With the importance of dicamba in the weed control scenario, concerns about the quality and safety in the applications of these products also arise to minimize contamination problems and losses due to spray drift or volatilization. Knowing the degree of volatilization of dicamba becomes more important because of the potential effects that this molecule can cause in sensitive cultures, even at low concentrations (Oliveira Jr, 2011). In 2018, the U.S. Environmental Protection Agency (USEPA, 2018) determined that an airborne concentration of 138 ng m⁻³ could be used as the most sensitive threshold with no observable adverse effects when assessing risk, specifically regarding the height of plants (US Environmental Protection Agency, 2018). In the case of deposition of dicamba by physical drift (particles), a concentration equivalent to 1% of the commercial dose was sufficient to reduce soybean yield by 50%, as well as to reduce cotton and peanut yield (Johnson et al., 2012). Yield reduction with low concentrations of dicamba has also been identified in grape crops (Dixon et al., 2021). In addition to reduced productivity, low concentrations of dicamba can reduce seed germination and lead to unevenness in crop plant stands (Silva et al., 2018; Costa et al., 2020).

With the new dicamba tolerance biotech traits, the industry has invested resources to develop new formulations that have a low risk profile. In addition, volatility reducers were also developed to further decrease the volatility profile of dicamba by scavenging free protons in the dicamba spray solution and avoiding the formation of dicamba acid (Carbonari et al., 2022a).

Riter et al. (2020) developed a robust set of methodologies for the assessment of dicamba volatility drift under field conditions. According to the authors, the quantitative assessment of herbicide volatility under commercial field conditions imposes specific requirements on sampling logistics, detailed monitoring of meteorological conditions, precise analytical methods, and sophisticated modeling techniques. The method described by Riter et al. (2020) details the study design, sample collection, analytical chemistry, and flux modeling. Based on this method, Sall et al. (2020) carried out a series of studies consisting of 23 field trials in the United States over three years of work in different locations and environmental conditions, aiming to quantify the post application volatilization of different dicamba formulations. In all cases, the vertical flux peaked within 24 hours of application, declining to much lower levels in the following hours. The total losses of volatiles among all dicamba formulations and conditions evaluated ranged from 0.023 ± 0.003 to $0.302 \pm 0.045\%$ of the dicamba applied.

The footprint of DT soybean is expected to increase greatly in Brazil in the coming years with the release of the I2X system. Although there is relevant information cited above on the volatilization of dicamba under field conditions, such this information is practically non-existent in the literature for tropical climate conditions. Tropical conditions are characterized according to the Köppen-Geiger climate classification by warm temperatures with an alternating rainy and dry season, or by an equatorial climate with humid conditions (Beck et al., 2028). Gentil et al. (2020) showed that many environmental processes related to pesticides, such as degradation and volatilization, have higher kinetic rates under tropical conditions, mainly due to higher temperatures, solar radiation, intense rainfall, and microbial activity. Product volatilization is related to its vapor pressure, that is, the pressure of the vapor state of a given molecule in equilibrium with the liquid or solid phase, indicating the ability of a compound to change its physical state from liquid or solid form to the gaseous form (Silva, Monquero, 2013). The temperature increase enhances volatilization because of higher vapor pressure (Sanchez-Bayo, Hyne, 2011).

Considering this scenario, the objective of this research was to evaluate and describe the behavior of dicamba volatility under application conditions in commercial crops in tropical climate conditions in Brazil and compare it with the risks of damage in dicamba non-tolerant soybeans and other sensitive crops according to references found in the literature.

2. Material and Methods

2.1 Experimental fields

Six trials were conducted in areas with a long history of soybean production (Table 1) from 2018 to 2020. Five out of the six trials were conducted over the top of dicamba-

sensitive soybean, and one was conducted on bare ground soil. Dicamba-sensitive soybean fields were chosen to run this research because by the time the six trials were set up, there were no commercial fields of Intacta 2 Xtend® (I2X) in Brazil. All information related to the trial details, including locations, soybean growth stage, application dates and time, chemistry used in each field (dicamba and glyphosate salts used, application rates and adjuvants) and basic information related to the application technology can be found in Tables 1–3. For each field, the herbicides and adjuvants shown in Table 2 were tank mixed.

In all trials, a 4.0 ha experimental field (200 m by 200 m) was set up for dicamba application. The fields were flat and essentially uniform with respect to slope, soil texture, and

agronomic and pesticide historical use. All areas were placed at least 150 m away from any major wind obstruction. All trials were set up following a basic design and methods as described by Riter et al. (2020) for volatility assessment. According to this method (Riter et al., 2020) for each test, within the area that received dicamba application, air samplers were installed in different horizontal and vertical positions.

2.2 Herbicide application

Herbicide applications were performed with a basic tank mixture of dicamba diglycolamine salt (Xtendicam - 480 g L⁻¹, Bayer) plus glyphosate potassium salt (Roundup Transorb R - 480 g L⁻¹, Bayer). The rates of herbicides and

Table 1 - Description of each field, soybean growth stage, application dates and time

Field number ¹	Location	Soybean growth stage ⁴	Application date	Time of application
1	Primavera do Leste, MT ²	V6	11/27/2018	08:50 a.m. – 09:00 a.m.
2	Primavera do Leste, MT	V4	11/14/2019	01:37 p.m. – 01:47 p.m.
3	Primavera do Leste, MT	V4	11/18/2019	11:35 a.m. – 11:43 a.m.
4	Primavera do Leste, MT	Bare Ground	11/22/2019	09:20 a.m. – 09:30 a.m.
5	Douradina, MS	V2	11/05/2020	09:25 a.m. – 09:43 a.m.
6	Quarto Centenário, PR ³	V3	10/03/2020	10:04 a.m. – 10:22 a.m.

¹ Each trial corresponded to a field. ² MT: Mato Grosso State, Brazil. ³ PR: Prana State, Brazil. ⁴ V2, V3, V4 and V6: Growth Stages in Soybeans, respectively, with 2, 3, 4 and 6 fully developed trifoliolate

Table 2 - Chemistry used for each trial, including herbicide and adjuvant dose rates

Field number ¹	Dicamba ² rate (g ae ha ⁻¹)	Glyphosate ³ rate (g ae ha ⁻¹)	Adjuvant 1	Adjuvant 1 rate (% v v ⁻¹)	Adjuvant 2	Adjuvant 2 rate (% v v ⁻¹)
1	560	1,120	Vapor Grip ⁴	0.5	AgRHO Starguar ⁶	0.50
2	720	1,440	Xtend Protect ⁵	1.5	-	-
3	720	1,440	Vapor Grip	0.5	OnSite ⁷	0.75
4	720	1,440	Vapor Grip	0.5	OnSite	0.75
5	480	960	Xtend Protect	1.0	-	-
6	480	960	Xtend Protect	1.0	-	-

¹ Each trial corresponded to a field. ² Dicamba diglycolamine salt dicamba (Xtendicam® - Bayer); the different doses are recommended on the label and were chosen according to the recommendation of each field. ³ Glyphosate potassium salt (Roundup Transorb R® - Monsanto); the different doses are recommended on the label and were chosen according to the recommendation of each field. ⁴ Volatility reducer - potassium acetate (Vapor Grip® - Monsanto). ⁵ Premix of vapor grip volatility reducing adjuvant and a drift reducing adjuvant (Xtend Protect® - Solvay). ⁶ Guar gum (AgRHO® Starguar - Solvay). ⁷ Polymer (OnSite® - Brandt)

Table 3 - Information related to application technology used in each trial

Field number ¹	Sprayer model	Nozzle ²	Nozzle spacing (cm)	Number of nozzles	Average travel speed (km h ⁻¹)	Application rate (L ha ⁻¹)
1	John Deere 4730	TTI 11004	50	60	19	98.3
2	John Deere 4730	TTI 11004	50	60	18	100
3	John Deere 4730	TTI 11004	50	60	18	100
4	John Deere 4730	TTI 11004	50	60	18	100
5	Stara Gladiator	TTI 11004	50	54	18	100
6	Uniport 3030	TTI 11002	35	93	13	100

¹ Each trial corresponded to a field. ² The spraying pressure was 280 kPa in all trials providing ultracoarse (UC) droplets

adjuvants used for each field are described in Table 2. Specific information related to dates and time for each application, self-propelled sprayer models, nozzle orifice sizes, spray volume rate and chemical dose rates used in each trial (herbicides and adjuvants) are described in Tables 1 and 3. Ultracoarse (UC) droplets were used in all trials, keeping the spray boom at 0.5 m above the targets.

2.3 Field and equipment layout for air sample collection

In all fields, one air sampling mast was placed at the center of the sprayed plot after approximately 30 minutes of application, while a weather station for wind, temperature and relative humidity data sampling was placed 15 m outside of an upwind field edge. The mast was equipped with air pumps that were attached to a glass tube with polyurethane foam (PUF) collectors designed to capture dicamba in the air at heights of 0.15, 0.33, 0.55, 0.9, and 1.5 m above the canopy in field 1 (2018 trial) and 0.33, 0.55, 0.9, and 1.5 m above the ground or canopy in fields 2 to 6 (2019 and 2020 trials) (Figure 1).

The flow rate of each air pump was set to deliver a flow rate of 2.9 to 3.1 L min⁻¹. Flow calibration was recorded for each sampler as well as the time and date taken. To avoid contamination from physical drift, air sampling started 30 minutes after the end of application and lasted for up to 72 hours (~3 days) or ended when there was more than 2 mm of rain. During this 3-day sampling duration, the sampling interval was set to approximately 3 to 5 hours on day 1 and approximately 10 to 12 hours on the following days.

The shorter sampling duration on day 1 provides increased resolution when volatility is expected to be higher.

All samples were collected in a manner to avoid the potential for cross contamination. Samples (PUFs) were placed in labeled plastic 50 mL plastic centrifuge tubes and placed in labeled cardboard boxes to isolate samples by distance and direction. Boxes were double bagged using plastic bags and placed into an appropriate cooler. All samples were kept isolated based on type and distance from the application area and stored and shipped in coolers containing dry ice until transfer to storage at -20°C prior to analysis.

2.4 Dicamba extraction and quantification

To perform dicamba extraction from PUF traps a water:methanol (75/25) solution was prepared containing a labeled internal standard (0.075 ppb of (13C6)-dicamba) (CAS no.: 1173023-06-7; Sigma Aldrich, St. Louis, MO). The PUFs were removed from the plastic centrifuge tubes and placed in 60 mL syringes. The plastic centrifuge tubes were then shaken with 10 mL of the extraction solution for one minute. The washing solution from the plastic centrifuge tubes was then poured into 60 mL syringes followed by another 10 mL of the extraction solution, totaling 20 mL in 60 mL syringes. This 20 mL of solution was then pushed and pulled from the syringe and a beaker 15 times. After this process, all the solution was placed in new 50 mL plastic centrifuge tubes. All samples were carefully managed during the extraction and analysis process to avoid the potential for cross-contamination and stored at -20°C until analysis.



Figure 1 - Air samplers (right) and meteorological station to collect meteorological parameters (left) at different heights and detail of the PUF collectors (bottom box)

Dicamba was quantified using a liquid chromatography–tandem mass spectrometry (LC–MS/MS) system composed of a high-performance liquid chromatograph (Prominence UFLC, Shimadzu, Kyoto, Japan) coupled to an AB Sciex API 5000 triple-quadrupole mass spectrometer (Applied Biosystems, Foster City, USA) with an electrospray ionization (ESI) source in negative ionization mode. Extracted samples were separated using a gradient from 25 to 61% B (mobile phase A = 0.1% formic acid in water and mobile phase B = 0.1% formic acid in methanol) at a flow rate of 0.3 mL min⁻¹ on a Phenomenex Kinetex Phenyl Hexyl column (100 mm × 2.1 mm, 2.6 μm) held at 50°C. Dicamba and (13C6)-dicamba were monitored at unit resolution in multiple reaction monitoring (MRM) mode at m/z 219–175 and m/z 225–181, respectively, with confirmatory transitions at m/z 221–177 and m/z 225–181 as described by Riter et al (2020).

2.5 Weather data collection

For all trials, a meteorological station was positioned near an upwind edge of the trial plot at approximately 15 m from the edge of the field. Wind speed was measured

by WindSonic Option 3 black ultrasonic sensors (Gill® Instruments Limited), temperature and humidity were measured by Smart Sensor S-THB-M008 sensors (Onset® Computer Corporation), and the data were sent and recorded in an HOBO® RX3000 datalogger (Onset® Computer Corporation). All the factors were measured and recorded every minute at 0.15, 0.33, 0.55, 0.9, and 1.5 m height above the canopy for field 1 (2018 trial) and 0.33, 0.55, 0.9, and 1.5 m height above the canopy or ground for fields 2 to 6 (2019 and 2020 trials), which correspond to the same heights as the PUFs. The range of temperature, relative air humidity and windspeed are shown in Table 4. Aerial and meteorological samples were conducted for 72 hours after dicamba application for the different fields. Only for fields 2 and 4 sampling was terminated 48 and 52 hours after sampling due to rain at these times.

2.6 Determination of dicamba concentration

Dicamba air concentrations (μg m⁻³) were calculated from the laboratory-provided sample residues (μg) based on the volume of air passing through each PUF sample collector. Pump flow rates were recorded at the start and

Table 4 - Range of meteorological conditions measured throughout the entire sampling period for each trial								
Field	Sample height							
	1.5 m		0.9 m		0.55 m		0.33 m	
	Temperature (°C)							
	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
1	-	-	20.56	31.28	20.56	31.46	20.51	31.71
2	19.34	31.69	19.34	33.00	19.27	32.30	19.32	31.05
3	20.39	32.51	20.41	32.92	20.34	32.66	20.39	32.07
4	20.20	32.92	20.10	33.94	20.08	33.21	20.27	32.28
5	14.15	38.90	14.00	39.35	13.91	40.17	13.71	40.57
6	13.33	30.95	13.16	31.31	12.97	32.07	12.94	32.59
Relative air humidity (%)								
	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
1	-*	-	57.20	99.10	56.80	97.80	56.60	99.10
2	54.10	98.10	56.30	99.20	56.40	98.10	55.80	98.80
3	52.00	97.60	55.50	98.70	54.80	97.60	53.40	98.20
4	46.10	96.50	48.30	97.60	48.80	96.80	48.20	97.10
5	25.50	92.40	23.40	92.30	25.70	94.40	25.30	92.70
6	32.10	89.90	30.80	89.80	32.30	91.50	32.80	92.70
Wind speed (km h ⁻¹)								
	Min.	Max.	Min.	Max.	Min.	Max.	Min.	Max.
1	-	-	-	-	0.57	15.32	0.50	13.04
2	0.60	17.83	0.51	15.46	0.44	13.49	0.36	10.81
3	0.93	26.68	0.63	15.62	0.58	13.62	0.57	12.54
4	0.21	16.68	0.21	13.04	0.21	12.79	0.21	9.78
5	0.65	19.39	0.64	17.26	0.56	15.00	0.56	13.95
6	1.32	18.08	1.25	15.80	1.01	14.13	0.97	12.01

*Data not collected for field 1 because of equipment failure

end of each sample. The average pump flow rate and the sample duration were used to calculate the volume of air passing through the PUF sample collector.

The working range of the analytical method used for this study was 0.03 to 7.5 ng PUF⁻¹, with a limit of quantitation (LOQ) of 0.1 ng PUF⁻¹. If the sample mass was below 0.03 ng, the limit of detection (LOD), that mass and concentration were reported as non-detect (ND). Sample air concentrations resulting from analytical results between the LOD and the LOQ were evaluated for suitability by visualizing the log-linear fit against sample height. In the case of a pump malfunctioning or the end time was unknown for some other reason, an air concentration was not calculated and was reported as no answer (N/A).

Duplicate samples were taken at a height of 1.5 m on the mast sample. Air concentrations were calculated independently for each of these samples. The average of the two air concentrations was used in subsequent analyses, including outlier analysis.

2.7 Flux estimation methods

The integrated horizontal flux (IHF) and aerodynamic (AD) methods were employed to estimate the dicamba flux. These methods are further described in the following sections.

2.8 Integrated horizontal flux (IHF) method

The IHF method or “mass-balance” method and related equations used to estimate flux are presented in literature [21-23]. The IHF method can be used to estimate surface flux when the air concentration, $c(z)$, and horizontal wind speeds, $u(z)$, in the atmosphere are known as a function of height. Method requirements include a minimum in-field fetch requirement of 20 meters, as well as a maximum surface roughness length requirement of 0.1 meters.

This method was used to estimate flux during post-application periods using air concentrations and horizontal wind speeds measured at several heights above the crop canopy at the approximate center of the treated field. Based on mass balance principles, this method assumed that dicamba mass emitted from the soil/crop surface in an area upwind of the sampling mast was equal to the mass passing through a vertical plane at the sampling mast. A log-linear regression was conducted to relate the natural logarithm of the sample height to the concentration and wind speed. These variables were integrated over height in equations described by Majewski et al. (1990) to estimate flux. Assuming a spatially uniform source (e.g., flux(x,y) is constant), the flux ($\mu\text{g m}^{-2} \text{s}^{-1}$) was estimated from:

$$\text{flux} = \frac{1}{X} \int_{Z_0}^{Z_p} \bar{u}(\bar{z}) + \bar{c}(\bar{z}) dz$$

where X is the fetch - the air trajectory across the field (meters) from the upwind edge to the sampling location, $\bar{c}$ is the average residue concentration ($\mu\text{g m}^{-3}$) at height

z (m), $\bar{u}$ is the wind speed (m s^{-1}) at height z, Z_0 is the AD surface roughness length (m), and Z_p is the height of the top of the plume (m), where dicamba concentrations become negligible.

For cases where concentrations and wind speed were well represented by a straight line regression against $\ln(\text{sampler height (m)})$, the integral in the above equation for flux had an analytical solution that was useful for calculating the flux values. The above equation can therefore be simplified to:

$$\text{flux} = \frac{1}{X} \int_{Z_0}^{Z_p} (m_u \ln(z) + b_u) (m_c \ln(z) + b_c) dz$$

where m_u is the slope of the wind speed regression line by $\ln(z)$, b_u is the intercept of the wind speed regression line by $\ln(z)$, m_c is the slope of the concentration regression line by $\ln(z)$, and b_c is the intercept of the concentration regression line by $\ln(z)$. The analytical solution to this integral is given by:

$$\text{flux} = \frac{Z_p}{X} (m_u b_c - m_c b_u + 2m_c m_{cu}) + \frac{Z_0}{X} (m_u b_c - m_c b_u + 2m_c m_{cu})$$

Except for occasional and minor differences, the analytical solution to the integral equation was in exact agreement with the alternative numerical integration.

The parameter Z_p is set by the natural upper limit to the indicated integration as the height at which the air concentration falls to zero and can be determined by the following equation:

$$Z = \exp\left[\frac{(-b_c)}{m_c}\right] \text{ if } \leq 25 \text{ m}$$

$Z_p = 25 \text{ m}$ otherwise

where b_c is the intercept of the concentration regression by $\ln(z)$ and m_c is the slope of the concentration regression by $\ln(z)$. In the US EPA's spreadsheet template for the IHF method, the numerator for Z_p in the above equation is $(0.1 - b_c)$ instead of $(-b_c)$. However, for semi volatile compounds, such as dicamba, $0.1 \mu\text{g m}^{-3}$ is not an appropriate reference concentration for the plume top because measured concentrations in the plume may be lower than this. Therefore, the plume top reference concentration was set to 0 in this study. If the equation for Z_p , which is sensitive to the intercept/slope ratio, yields a calculated Z_p value that is unrealistically high, then Z_p is limited to 25 m (based on US EPA method recommendation). The result is that flux integration limits represent the height at which the air concentration reaches zero or 25 m at the upper end, Z_p , and the height at which wind speed reaches zero at the lower end, Z_0 . The Z_0 parameter can be determined similarly to Z_p by solving for z in the regression equation of wind speed to $\ln(z)$, where the wind speed is set to 0.

The fetch distance, X, is the distance between the sample mast (at center of field) and the upwind edge of the treated plot. This fetch distance was calculated every minute during

the sampling period according to the wind direction and the plot orientation and averaged over the entire sampling period to give an average value used for calculation of the average flux during that period.

2.9 Aerodynamic (AD) method

The AD method or “flux-gradient” method and related equations used to estimate flux are also presented in literature [28,29,30]. As with the IHF method, the AD method also uses air concentration and wind speed, as well as temperature, measured at multiple heights above the soil surface or crop canopy from near the center of the treated field. A log-linear regression was conducted to relate period-averaged wind speed, temperature, and concentration values with the natural logarithm of height at which measurements were conducted. The AD method has a minimum external fetch, referring to the upwind distance to the nearest large-scale structure or other wind obstacle, not the edge of the treated field (Thibodeaux, 1980), requirement of 100 times the height of the highest air sampler.

The log-linear relationships between wind speed, temperature, and concentration are then incorporated into the Thornthwaite-Holzman equation to estimate flux:

$$\text{flux} = \frac{-(0.42)^2 (c_{z_{\text{top}}} - c_{z_{\text{bottom}}})(u_{z_{\text{top}}} - u_{z_{\text{bottom}}})}{\phi_m \phi_p \left[\ln \left(\frac{Z_{\text{top}}}{Z_{\text{bottom}}} \right) \right]^2}$$

where ϕ_m and ϕ_p are internal boundary layer (IBL) atmospheric stability correction terms determined according to the following conditions based on the calculated Richardson number, Ri:

$$Ri = \frac{(9.8)(Z_{\text{top}} - Z_{\text{bottom}})(T_{z_{\text{top}}} - T_{z_{\text{bottom}}})}{\left[\left(\frac{T_{z_{\text{top}}} - T_{z_{\text{bottom}}}}{2} \right) + 273.16 \right] \times (u_{z_{\text{top}}} - u_{z_{\text{bottom}}})^2}$$

where $T(Z_{\text{top}})$ and $T(Z_{\text{bottom}})$ are the regressed temperatures at the top and bottom of the vertical profile in units of °C. Ri is determined as:

if $Ri > 0$ (for Stagnant/Stable IBL):

$$\phi_m = (1 + 16Ri)^{0.33} \text{ and } \phi_p = 0.885(1 + 34Ri)^{0.4}$$

if $Ri < 0$ (for Convective/Unstable IBL):

$$\phi_m = (1 - 16Ri)^{-0.33} \text{ and } \phi_p = 0.885(1 - 22Ri)^{-0.4}$$

The values of $T(Z_{\text{top}})$ and $T(Z_{\text{bottom}})$ used in this work are sampling heights of 0.9 m and 0.33 m, respectively, based on the USEPA IHF spreadsheet template calculations.

2.10 Mass loss calculations

The mass of dicamba lost due to volatilization during each period of the study was estimated based on the flux estimates from each of the methods described above, according with Sall et al. (2020). The calculation of mass

loss was based on the target application rate in each field (Table 2) and the actual size of the test field (200 m x 200 m),

3. Results and Discussion

Table 4 presents the meteorological data (maximum and minimum values) recorded during each of the 6 trials. Although the volatility simulation models used in this work (IHF and AD) do not use relative air humidity values in their processing, these values are provided in Table 4 to better contextualize the real conditions in the field in each region. In general, the values presented show how the weather conditions can be quite variable in the Midwest and South regions of Brazil, the main soybean producing regions in the country (Empresa Brasileira de Pesquisa Agropecuária, 2023). Except for field 5, which presented higher maximum temperatures, the other areas presented similar temperatures after application, during the air sampling period in the different fields (Table 4).

Taking the records obtained closer to the ground (0.33 m high), in the region of Mato Grosso (fields 2, 3 and 4), as an example, the minimum and maximum temperatures were 19.32 and 32.28 °C, respectively, while in Mato Grosso do Sul (field 5), these values were between 13.71 and 40.57 °C, and in Paraná (field 6), they were between 12.94 and 32.59 °C. Regarding relative air humidity, the minimum values recorded were 48.20% for fields 1, 2, 3 and 4 in MT, 25.30% in field 5 (MS), and 32.80% in field 6 (PR). In the case of wind, the maximum values obtained at a height of 0.33 m were similar to each other in the different regions (13.04 km h⁻¹ for MT, 13.95 km h⁻¹ for MS, and 12.01 km h⁻¹ for PR). However, if the highest sampling (1.5 m above ground level) was considered, the speeds reached higher and more diverse values, with 26.68 km h⁻¹ for MT (fields 1, 2, 3 and 4), 19.39 km h⁻¹ for MS (Field 5), and 18.08 km h⁻¹ for PR (Field 6).

The dicamba flux (ng m⁻² s⁻¹) and the total cumulative mass loss (% of the dose applied in the field) presented in Figure 2 correspond to the average between the data generated with the application of the two simulation models (AD and IHF). The flux profile of dicamba starts relatively high (above 1 ng m⁻² s⁻¹ in the first evaluation in all fields), reaching a maximum value of 3.8 ng m⁻² s⁻¹ in field 6. This flux decays quickly in the first 20 hours, reaching virtually null values in some fields. The lower values found for fields 2 and 4 regarding accumulated mass losses are due to the rainfall that occurred during the sampling period in these two fields, as previously reported.

Table 5 shows an analysis of the behavior of dicamba emissions considering the mean and standard deviation of the data from the six fields evaluated. The initial flux was 2.15±0.99 ng m⁻² s⁻¹, decreasing in the period approximately 20 hours after application to 0.30±0.25 ng m⁻² s⁻¹, a reduction of 85.9%. In the hours that followed until the end of the collections, the value of dicamba emissions continued to decrease, ending with a value of 0.18±0.17

$\text{ng m}^{-2} \text{s}^{-1}$ on average for the six fields (89.7% reduction compared to the average initial flux). It is worth noting that each trial had a specific duration, varying between 43 and 72 hours, since the completion of the collections in each field always occurred 72 hours after application or at the time of the first rain with precipitation greater than 2 mm following application.

The mean and standard deviation related to the accumulated dicamba loss data for the six fields are also presented in Table 5. Based on the initial period approximately 20 hours after application, when the flux was higher, the average accumulated loss reached $0.10 \pm 0.04\%$ of the dose applied. The average final value after the end of the collections was $0.15 \pm 0.08\%$ of the dose applied.

Figure 3 presents the values of the maximum concentration of dicamba in the air measured during the data collection period in each of the six fields considered

in this research. Values ranged from 20.21 ng m^{-3} (field 2) to 47.18 ng m^{-3} (field 6), with a mean of $38.66 \pm 9.52 \text{ ng m}^{-3}$ for all tests.

Sall et al. (2020) conducted a statistical analysis comparing 157 paired data from the application of AD and IHF models on data from 23 dicamba volatility trials in the field, noting that the AD model returned a higher vertical flux estimate than the IHF only in the initial sampling period, with no differences between the AD and IHF methods in any of the subsequent sampling periods up to 72 hours after application.

The range of values and the general behavior of the dicamba emission flux in this study repeat the pattern observed in the 23 trials described by Sall et al. (2020) in the United States. As a comparison parameter in relation to the data generated with the same methodology in the United States (Soltani et al., 2020). Sall et al. (2020) recorded

Table 5 - Mean values and standard deviation of the dicamba flux ($\text{ng m}^{-2} \text{s}^{-1}$) and the accumulated dicamba (% of the applied dose) considering the data from the six trials

Data considering the 6 trials	Initial flux ($\text{ng m}^{-2} \text{s}^{-1}$)	Flux after approximately 20 hours* ($\text{ng m}^{-2} \text{s}^{-1}$)	Flux at the end of collections ($\text{ng m}^{-2} \text{s}^{-1}$)	Accumulated after approximately 20 hours ¹ (% of dose applied)	Final cumulative (% of dose applied)
Mean	2.15	0.30	0.22	0.10	0.15
Standard deviation	0.99	0.25	0.15	0.04	0.08

¹ Time considered varied between 19 and 24.5 hours depending on the collection in each field

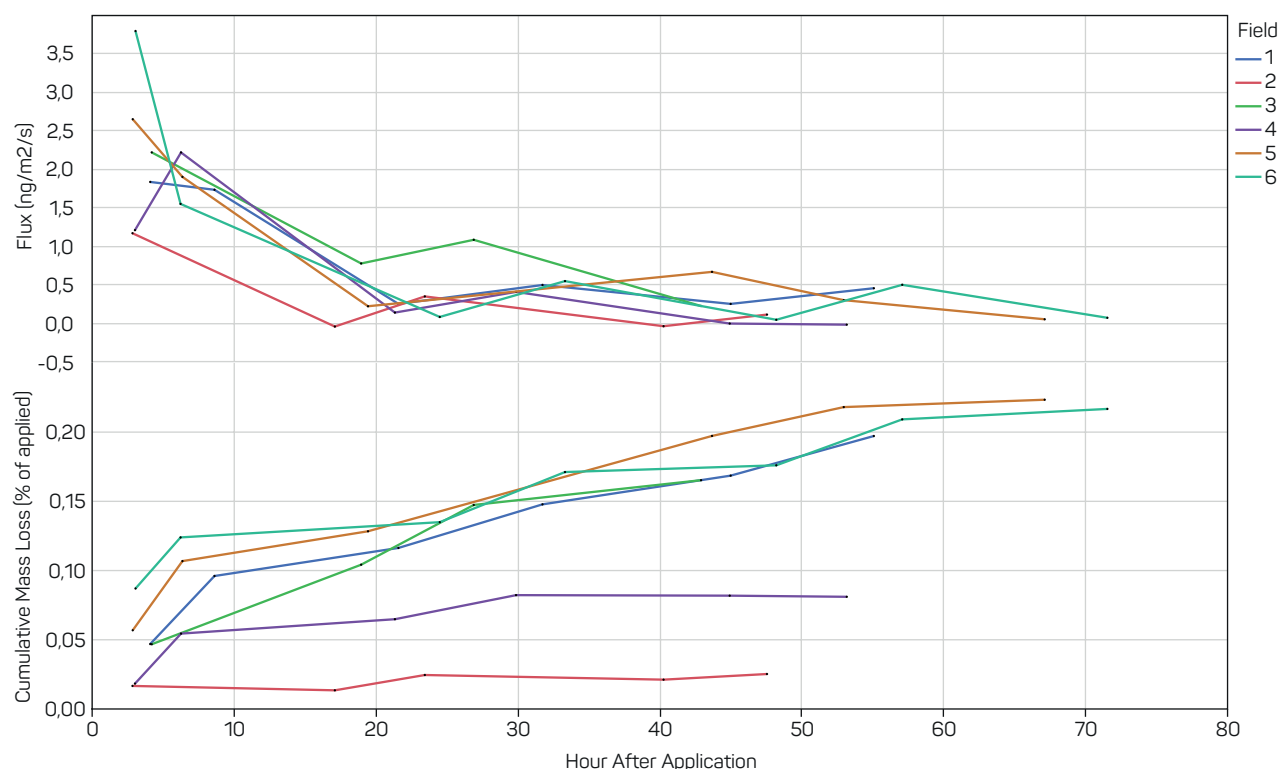


Figure 2 - Dicamba flux values ($\text{ng m}^{-2} \text{s}^{-1}$) and the cumulative mass loss of dicamba (% of the applied dose) over time for the 6 fields evaluated, corresponding to the 6 trials carried out in this research

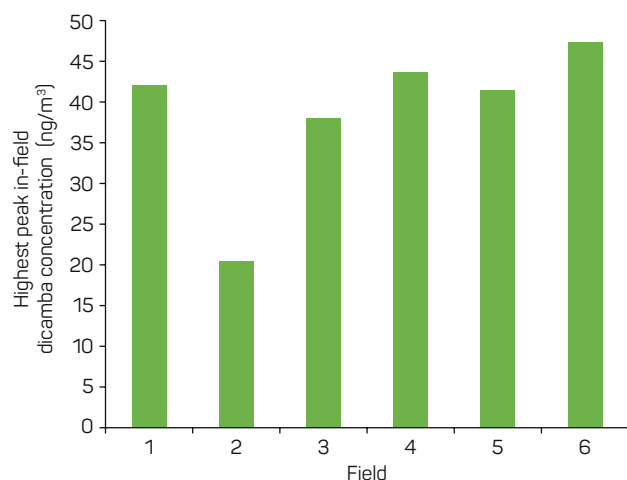


Figure 3 - The green bars show the values of the maximum in-field concentration of dicamba (ng m^{-3}) measured during the data collection period in each of the 6 fields in this study

accumulated losses of up to $0.30 \pm 0.05\%$, while Riter et al. (2020) reported an average cumulative loss of $0.2 \pm 0.05\%$ of the dose applied.

In all fields evaluated in this research, the maximum concentration observed was lower than the worst case observed among the 23 trials reported by Sall et al. (2020), which was 51 ng m^{-3} , and the mean value obtained in Brazil represented only 28% of the no observable adverse effect concentration (NOAEC) for non-dicamba tolerant soybean height or yield considered by the USEPA (2018), which is 138 ng m^{-3} . The concentration of dicamba in the air at different times after application were also similar to other studies found in the literature (Sall et al., 2020; Riter et al., 2020) with a very intense decay in the amount of dicamba in the air up to 20 hours after application.

Using similar methods, a large-scale experiment conducted from 2018 to 2021 investigated damage to nearby sensitive cotton from 2,4-D choline and glufosinate-ammonium applications (Hwang et al., 2022). In this study, the herbicides were applied to 0.4 ha in the center of a 4 ha non-2,4-D tolerant cotton field. Air samplers were placed at the treated area 30 minutes after application. According with the authors, the 72-hour cumulative air concentration of 2,4-D ranged from 3.3 to 7.1 ng m^{-3} , with higher volatile residues (5.0 – 25.5 ng m^{-3}) found downwind of the treated field. No damage was reported outside the treated area, except downwind during the application, despite wind direction changes. The study shows that 2,4-D choline volatilization is insufficient to damage nearby cotton, highlighting the need for applicators to consider wind direction and the proximity of sensitive crops during application.

In the 6 fields of this study, dicamba DGA was applied with a volatility reducer in association with potassium glyphosate. The literature indicates that the DGA dicamba salt is less susceptible to volatilization than the DMA dicamba salt under controlled or field conditions (Mueller,

Steckel, 2019; Egan, Mortensen, 2012; Dixon et al., 2021; Mueller et al., 2013). The volatilization of dicamba applied in the form of DGA salt was reduced by 94% compared to that of dicamba applied in the form of DMA salt (Majewski et al., 1991). Mueller and Steckel (2019) and carbonari et al., 2022a) evaluated the volatilization of the application of dicamba DGA alone or with different formulations (salts) of glyphosate and observed that the mixture with glyphosate increased the volatilization. Despite this, the author showed that volatility reducer was very efficient in reducing volatility for dicamba alone and in combination with all glyphosate salts. The combination of DGA dicamba salt with potassium salt of glyphosate and a volatility reducer was the blend with the lowest volatility and is the most suitable combination to recommend to farmers (Carbonari et al., 2022a; Carbonari et al., 2022b).

The results of this research were compared with those generated in the United States (Sall et al., 2020; Riter et al., 2020) using a similar methodology. It was found that the potential for dicamba volatilization under application conditions in tropical regions in Brazil was equal to or less than dicamba volatilization measured in temperate regions in the United States. In 2018, the (USEPA, 2018) determined that an air concentration of 138 ng m^{-3} could be used as the most sensitive concentration with no observable adverse effect for dicamba sensitive soybean in a risk assessment (US Environmental Protection Agency, 2018). To conduct a risk assessment, the NOAEC was compared with the air concentration outside of the field to calculate a buffer distance. The air concentration measured in the middle of the field at 33 cm above the application can be considered the worst case and an upper bound air concentration since off-field air concentrations are relatively low due to effects from dispersion and dilution. The results indicated that even the maximum in-field concentration in each field was lower than the NOAEC of 138 ng m^{-3} (Figure 3), thus demonstrating that the use of this dicamba tank mix is protective of soybean fields in Brazil from unreasonable adverse effects.

As a comparison, Bish et al. (2019) quantified dicamba in the air after application of the dicamba formulations dicamba DGA salt + volatility reducer (Vapor Grip®) and dicamba BAPMA salt with and without glyphosate potassium salt and under different weather conditions. The dicamba DGA salt + Vapor Grip® and BAPMA salt were quantified at similar levels over time when applied simultaneously Bish et al. (2019). The highest concentrations for each formulation occurred from 0.5 to 8 hours after application, and the concentrations of DGA + volatility reducer and BAPMA were 22.6 and 25.8 ng m^{-3} , respectively. Bish et al. (2019) found that both dicamba formulations had similarly rapid dissipation in air, with dicamba concentrations decreasing from $> 20 \text{ ng m}^{-3}$ in 0.5 to 8 hours after application (HAA) to $< 7 \text{ ng m}^{-3}$ between 8 and 16 HAA and 24 and 48 HAA and the dicamba concentrations were $< 2 \text{ ng m}^{-3}$ and remained at this level until 72 HAA.

4. Conclusions

The values of dicamba flux and accumulated volatile mass loss in the 6 fields under tropical agriculture conditions were equal to or lower than the results of studies carried out in temperate climate regions reported in the literature with similar methodology. The results indicated that even the maximum in-field concentration in each area was below the NOAEC of 138 ng m³, demonstrating that the use of this dicamba mixture in Brazil is potentially safe against unreasonable adverse effects in soybean fields.

Author's contributions

All authors read and agreed to the published version of the manuscript. URA, HB, RFLO, CAC and EDV: conceptualization of the manuscript and development of

the methodology. AM, RC, FC, URA, MP, NP and FC: data collection and curation. NP, MP and FC: data analysis. URA, CAC and NP: data interpretation. URA and RFLO: funding acquisition and resources. URA: project administration. URA and RFLO: supervision. URA, MP and CAC: writing the original draft of the manuscript. AM, RFLO, EDV and NP: writing, review and editing.

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