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ORIGINAL ARTICLE

Comparison of water-sensitive paper, Kromekote and Mylar collectors for droplet deposition with a visible fluorescent dye solution

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Abstract

The study was conducted at the University of Nebraska Pesticide Application and Technology Laboratory in North Platte, Nebraska in July 2015. Two application volume rates (100 and 200 $l \cdot ha^{-1}$) and three nozzle types (XR, AIXR, TTI) were selected at two flow rates (0.8 and 1.6 $l \cdot min^{-1}$) and at a single application speed of 7.7 $km \cdot h^{-1}$. Each collector type [Mylar washed (MW), Mylar image analysis (MIA), water-sensitive paper (WSP), and Kromekote (KK)] was arranged in a randomized complete block design. Each nozzle treatment was replicated twice, providing six cards of each collector type for each nozzle treatment. A water + 0.4% v/v Rhodamine WT spray solution was applied, given the fluorescent and visible qualities of Rhodamine, which allows it to be applied over all the collector types. MW had the highest coverage at 18.3% across nozzle type, followed by WSP at 18%, KK at 12% and lastly by MIA at 4%. MW resulted in a 58% increase in coverage, WSP in a 56% increase, and KK only an increase of 39% when the volume rate was doubled from 100 $l \cdot ha^{-1}$ to 200 $l \cdot ha^{-1}$ across nozzle type. MW coverage was similar to KK for half of the nozzles (XR 11002, XR 11004, AIXR 11002). Droplet number density fixed effects were all significant for nozzle type and collector type ($p < 0.001$) as was the interaction of nozzle type and collector type ($p < 0.001$). Results from this study suggest a strong correlation to data produced with WSP and MW collectors, as there was full agreement between both types except for the TTI 11004. Using both collector types in the same study would allow for a visual understanding of the distribution of the spray, while also giving an idea of the concentration of that distribution.

Keywords: artificial collector, droplet density, droplet size, Kromekote, pesticide spray coverage, water-sensitive paper

Introduction

Effective crop protection product application requires the use of techniques and technologies that maximize coverage and droplet deposition on plants. Pesticide use increased by 23.5 million kilograms a.i. (active ingredient) between 2002 and 2010 in the US alone, which was a 10% increase during that time (Osteen and Fernandez-Cornejo 2013). Inefficiencies from poor application lead to a reduction of pest control and more off-target movement of sprays through spray drift (EPA 1999; Hewitt 2000) or when sprays are not well distributed within crop canopies (Wolf *et al.* 2000). Sprays that are not properly distributed through the canopy can lead to variable rates of pest control and the need to reapply the pesticide (Uk and Courshee 1982).

Many studies have been done on the use of collectors for droplet deposition characterization (Johnstone 1960; Higgins 1967; Turner and Huntington 1970; Hill and Inaba 1989; Hewitt and Meganasa 1993). Artificial collectors are diverse, ranging from alpha-cellulose cards, to glass slides, water-sensitive paper (WSP), Mylar collectors (Lee *et al.* 1978), and Kromekote collectors. WSP is a specific type of paper coated with bromoethyl blue, a dye that appears yellow, but turns blue when hydrated by water-containing droplets impacting the coated paper collectors (Turner and Huntington 1970). The use of WSP for characterization of sprays in the field has been on-going for 45 years (Turner and Huntington 1970) and is the most widely used collector type for canopy penetration studies (Knoche 1994; Derksen *et al.* 2008; Wolf and Daggupati 2009; Hanna *et al.* 2009; Derksen *et al.* 2014).

Kromekote collectors are a specialty type of photo paper that stain when a spray solution containing dye deposits on it (Johnstone 1960). Mylar collectors have been used in numerous spray drift studies due to their ability to release sprayed material when rinsed (Creech 2015). The physical properties of a surface have a significant effect on the ability of the impacting droplet to deposit, bounce, or shatter thereby repeating the deposit, bounce or shatter process (Spillman 1984; Dorr *et al.* 2014, 2015). The size of the impacting droplet will also influence its final fate (Spillman 1984). Mylar, WSP, and Kromekote collectors have different surface properties, which cause identical droplets to behave differently upon deposition (Forster *et al.* 2014). Collector types also have different wettabilities, which further affect the spreading and retention of a droplet, especially with differing liquid physical properties (Forster *et al.* 2014).

Canopy penetration is the ability of sprayed droplets to move through a canopy to provide adequate control of pests. Previous studies have examined canopy penetration in several cropping systems to identify which application techniques and technologies maximize canopy penetration (Knoche 1994; Zhu *et al.* 2004; Derksen *et al.* 2008; Hanna *et al.* 2009; Derksen *et al.* 2014; Creech 2015; Ferguson *et al.* 2016). These studies observed that coarser sprays improved canopy penetration within several crop canopy types. Only the trends from these studies have been compared since different methods to analyze droplet deposition were used. WSP collectors were used in most of these studies (Knoche 1994; Zhu *et al.* 2004; Derksen *et al.* 2008; Wolf and Daggupati 2009; Hanna *et al.* 2009; Derksen *et al.* 2014). One study used a fluorescent tracer dye and Mylar collectors and rinsed them, obtaining droplet deposition results based on the fluorescence of each collector (Creech 2015). Another study used Kromekote collectors and a visible dye in the spray solution to characterize deposition (Ferguson *et al.* 2016). Although the results from these studies appear to correlate, so far it has not been possible to pool them.

Therefore, the following experiment was conducted to understand the differences in droplet deposition results from WSP, Mylar, and Kromekote collectors using a common spray solution across five spray quality producing nozzles. The objectives of this study were: 1) to assess the coverage and droplet number densities (droplets cm^{-2}) from six different nozzles that span five different spray qualities across four collector types, 2) to quantify the differences that exist in the droplet deposition results from the different collector types.

Materials and Methods

Spray application of the artificial collectors

A study to compare the coverage and droplet number density was conducted at the Pesticide Application Technology Laboratory (PAT Lab) at the University of Nebraska West Central Research and Extension Center in North Platte, Nebraska, USA. Four collector types were studied: water-sensitive paper (WSP) (Novartis International AG, Basel Switzerland), Kromekote (KK), and two methods of Mylar (Grafix Plastics, Cleveland, OH, USA) analysis [one washed (MW) and one analyzed through image analysis software (MIA) as with the WSP and KK collectors]. Each collector measured 76 × 26 mm and was sprayed with water plus a 0.4% v/v addition of Rhodamine WT dye (Liquid Red, Cole-Parmer, Vernon Hills, IL, USA). Applications were made using a six-nozzle spray boom (50 cm nozzle spacing) attached to an all-terrain vehicle (ATV) (Polaris Xplorer 400 4x4, Polaris Industries, Medina, MN, USA). Dye was added since it is required by the KK and Mylar collectors to quantify droplet deposition. Nozzles used in the study included five different spray qualities (Fine, Medium, Coarse/Very-Coarse, Extremely-Coarse and Ultra-Coarse) (ASABE/ANSI 2018). Nozzles selected for the study were the XR 11002 & 11004; AIXR 11002 & 11004; TTI 11002 & 11004 (Spraying Systems Inc, Wheaton, IL, USA).

Though nozzles had varying flow rates, each treatment was applied at 7.7 km and 207 kPa. Application volume rates in the study were 100 l · ha^{-1} for the 0.8 l · min^{-1} (02) flow rate nozzles, and 200 l · ha^{-1} for the 1.6 l · min^{-1} (04) flow rate nozzles. Spray applications were made in a rye (*Secale cereale* L.) stubble field on July 28th, 2015. Collectors were placed on flat metal plates 50 cm underneath the boom with collector types arranged in a randomized complete block design (RCBD) with three replications. The block was comprised of a center driving line for the ATV sprayer with two of each collector on both sides of the driving line at 50 cm interval spacing. Each block line was spaced at 1 m intervals. Thus, total block size for the study was 2 by 2 m. Each nozzle treatment had two runs, producing six sprayed collectors of each collector type for each treatment.

Image analysis of the artificial collectors

Individual collectors of each type were scanned into the computer using a 6400 dpi flatbed scanner (Epson Perfection V600, Epson America Inc., Long Beach, CA, USA). Each sprayed collector image was analyzed using ImageJ software (Rasband 2008). The sprayed collector image was cropped to remove background area, changed into 8-bit format, and then into binary mode which makes the image black and white allowing coverage to be measured (Ferguson *et al.* 2016). Droplet number density was quantified by using the count function in ImageJ to obtain total droplets on the collector. This number was divided by 19.76 (area in cm^2 of each collector) to obtain the droplets' cm^{-2}. Each image was analyzed for droplet number density and percent coverage.

Washed Mylar® (MW) protocol

One MW collector in each block (three per treatment) was rinsed with 39 ml of a water plus 10% propan-2-ol solution measured from a bottle top dispenser (Model 6000-BTR, LabSciences Inc., Reno, NV, USA). Each MW collector was rinsed in the solution and agitated for 30 sec to release all Rhodamine dye from the collector and a 2 ml sample was pipetted into a glass cuvette. Cuvettes were analyzed for raw fluorescence units (RFU) using a fluorimeter (Trilogy Laboratory Fluorimeter, Turner Designs, Sunnyvale, CA, USA) with a Rhodamine filter and results were recorded.

Calculating coverage on MW

Coverage was calculated by determining the amount of dye captured on collectors by the amount of dye emitted from the application. Based on the fluorescence the captured dye was already known and, based on dilutions from tank samples (1 ml of the tank sample, added to 39 ml of water plus 10% propan-2-ol), and fluorescing the dilution, the amount of dye emitted was calculated.

Droplet size analysis

Each nozzle was analyzed for droplet size and distribution at the Pesticide Application Technology Laboratory (PAT Lab) at the University of Nebraska West Central Research and Extension Center in North Platte, NE, USA on July 27th, 2015. Wind speed was constant at 6.7 $m \cdot s^{-1}$. Each treatment was analyzed on a laser diffraction instrument (Sympatec HELOS--VARIO/KR, Sympatec Inc., Clausthal, Germany) to measure droplet size from each nozzle type. The laser diffraction instrument was 30 cm downwind from the nozzle, to allow for complete sheet breakup. Nozzles were actuated upward or downward (only one direction per measurement), allowing for the entire spray plume to pass through the measurement area for 9 sec per measurement. The volumetric droplet size spectra parameters selected for data interpretation were the $D_{v0.1}$, $D_{v0.5}$, and the $D_{v0.9}$. These parameters were selected because they are widely used to assess spray drift potential ($D_{v0.1}$) (Hewitt 1997) and efficacy potential ($D_{v0.5}$). The $D_{v0.1}$ is the diameter at which 10% of the volume of droplets are contained in droplets at or below that diameter. The $D_{v0.5}$ (volume median diameter) is the diameter at which half of the volume is contained in droplets of larger or smaller diameters to help classify sprays for efficacy potential, and understand the size classification of each. The $D_{v0.9}$ is the diameter at which 90% of the volume of droplets are contained in droplets at or below that diameter. ASABE/ANSI reference nozzles were also analyzed for droplet size and distribution as per the protocol in ASABE/ANSI S572.2 (ASABE/ANSI 2018) to help classify the spray quality of each of the nozzle treatments used in this study.

Statistical analyses

Collector type coverage, droplet number density and MW fluorescence were each analyzed in separate generalized linear mixed models (PROC GLIMMIX) in SAS (Statistical Analysis Software, version 9.4, Cary, NC, USA) with means separations made at the $\alpha = 0.05$ level. Coverage and droplet number densities were analyzed by the model: coverage (or droplet number density) = nozzle type by collector type by replication. Fixed effects were nozzle type and collector type. MW fluorescence was analyzed using the previous model, without collector type included. Fixed effects in the fluorescence model were the nozzle type and volume rate. In all three models, replication was treated as a random effect. The denominator degrees of freedom (*df*) was protected from bias through the inclusion of the Kenward-Roger adjustment for the generalized linear mixed model (Kenward and Roger 1997). The Sidak adjustment was included in comparisons of variables to improve the power and confidence in reported differences (Sidak 1967).

Results

Coverage results across collector type

Coverage was significant for collector type and nozzle type ($p < 0.001$). MW had the highest coverage at 18.3% across nozzle type, followed by WSP at 18%, KK at 12% and lastly by MIA at 4%. MW resulted in a 58% increase in coverage, WSP in a 56% increase, and KK had only an increase of 39% when the volume rate was doubled from $100\,l \cdot ha^{-1}$ to $200\,l \cdot ha^{-1}$ across nozzle type (Table 1). All collector types resulted in similar coverage for only the XR 11002 nozzle. MW coverage was always similar to WSP coverage for every nozzle except the TTI 11004, where MW had a higher coverage. MW coverage was similar to KK for half of the nozzles (XR 11002, XR 11004, AIXR 11002) (Table 1). KK was similar to WSP for all but two nozzles (XR 11004, AIXR 11004). For the TTI and XR nozzles, across collector type, the resulting coverage was greater than 50% relative to a 50% volume rate decrease (from 200 to $100\ l \cdot ha^{-1}$, respectively) (Table 1). Coverage was similar across nozzle types where the XR had the highest coverage, followed by the AIXR and lastly the TTI (11.4 to 10.8 to 9.5% coverage, respectively).

Droplet number density across collector type

Droplet number density fixed effects were all significant for nozzle type and collector type ($p < 0.001$) as was the interaction of nozzle type and collector type ($p < 0.001$). MIA collectors had the highest droplet number density for each nozzle type, followed by KK and lastly WSP collectors (167 to 93 to 74 droplets cm^{-2}, respectively). WSP and KK collectors observed similar droplet number densities across all nozzle types except the TTI 11004 (Table 1). All three collectors resulted in similar droplet number densities for the AIXR 11002 and the TTI 11002. Observed droplet number densities for MIA collectors resulted in greater than 1.5× more droplets than the other two collector types, making it the most sensitive of any of the collector types. KK collectors for the AIXR and TTI 11002 and 11004 resulted in nearly identical densities, where the AIXR 11002 had a higher density than the AIXR 11004 (69 to 68, respectively) (Table 1). The XR had the highest droplet number density followed by the AIXR and the TTI had the lowest droplet number density across collector type (217 to 77 to 40 droplets cm^{-2}, respectively). Volume rate increases followed a similar trend with the coverage results where the higher volume resulted in an overall higheTable deposition result, but not to the same degree of the increase (e.g.: doubling volume did not double droplet number density) (Table 1).

Fluorescence results from MW collectors

MW fluorescence fixed effects resulted in significance (nozzle type $p = 0.004$ and volume rate $p < 0.001$). Results from MW fluorimetry followed the trend where the increased volume rate increased the result (coverage or droplet number density, respectively) but did not follow the trend with respect to nozzle type as the TTI had the highest fluorescence result, and the XR the lowest (1,767 to 1,144 RFUs, respectively) (Table 1).

Droplet size results

Droplet size results were not run through a statistical model due to significance at small droplet size changes, as observed with previous studies (Ferguson *et al.* 2015). The XR had the smallest droplet size distribution, followed by the AIXR, with the TTI producing the largest droplet size distribution (Table 2). The

Table 1. Results of the coverage and droplet number density per nozzle and collector type and the letter grouping with Sidak's adjustment at α = 0.05 across each of the six imaging systems used in the study

Nozzle	Pressure	Volume rate	$D_{v0.5}$	MW	MW	Kromekote	WSP	MIA	Kromekote	WSP	MIA
	[kPa]	[$l \cdot ha^{-1}$]	[μm]	[RFU]	coverage%				droplets per cm^2		
XR 11002	207	100	213	559 C	7 H–J	10 F–J	12 E–I	3 J	161 *CD*	133 *CD*	294 *AB*
XR 11004	207	200	296	1,730 AB	23 A–D	18 C–F	22 AB	4 IJ	209 *BC*	153 *CD*	351 *A*
AIXR 11002	207	100	416	693 C	11 E–I	9 G–J	11 E–I	2 J	69 *E*	48 *E*	105 *DE*
AIXR 11004	207	200	534	1,898 AB	25 A–C	14 E–H	28 A	6 H–J	68 *E*	70 *E*	112 *D*
TTI 11002	207	100	801	1,239 BC	16 D–G	7 H–J	10 F–J	4 IJ	24 *E*	18 *E*	59 *E*
TTI 11004	207	200	879	2,295 A	30 A	12 E–I	20 B–E	6 H–J	26 *E*	33 *FG*	85 *DE*

MW – Mylar washed; WSP – water-sensitive paper; MIA – Mylar image analysis
The Mylar washed data were analyzed in its own model.
Letter groupings represent statistical difference in the generalized linear mixed model with Kenward-Roger and Sidak's adjustments. Letters following means within a row indicate significant differences at α = 0.05. The letters are italicized with the droplet number density data to indicate a separate statistical model to the coverage results

Table 2. Droplet size distribution for nozzles used in the study compared and defined by their spray quality against the ASABE/ANSI S572.2 reference nozzles for the $D_{v0.1}$, $D_{v0.5}$ and $D_{v0.9}$

Nozzle	Pressure [kPa]	$D_{v0.1}$ [μm]	$D_{v0.5}$ [μm]	$D_{v0.9}$ [μm]	ASABE/ANSI Classification
11001	450	67	142	241	Very-Fine/Fine
XR 11002	207	104	213	342	Fine
11003	300	113	250	414	Fine/Medium
XR 11004	207	133	296	486	Medium
11006	200	168	363	594	Medium/Coarse
AIXR 11002	207	224	416	613	Coarse
8008	250	201	436	723	Coarse/Very-Coarse
AIXR 11002	207	224	416	613	Very-Coarse
6510	200	243	522	834	Very-Coarse/Extremely-Coarse
AIXR 11004	207	275	534	807	Extremely-Coarse
6515	150	315	661	1,044	Extremely-Coarse/Ultra-Coarse
TTI 11002	207	431	801	1,123	Ultra-Coarse
TTI 11004	207	455	879	1,296	Ultra-Coarse

The AIXR 11002 is classified as both Coarse and Very-Coarse as it was Coarse $D_{v0.5}$ (efficacy purposes) and Very-Coarse $D_{v0.1}$ (spray drift concerns)

0.8 l · min^{-1} (0.2 gal · min^{-1}) flow-rate nozzles produced a smaller droplet distribution than their 1.6 l · min^{-1} (0.4 gal · min^{-1}) flow-rate counterpart, given the larger orifice size consistent with higher flow-rate nozzles. After the nozzles were characterized for droplet size distribution, the ASABE/ANSI reference nozzles were measured with water at their reference spray pressure to help classify each of these nozzles into an ASABE/ANSI spray quality classification (Table 2). The XR 11002 was classified as a Fine spray, the XR 11004 a Medium spray, the AIXR 11002 a Coarse spray by the $D_{v0.1}$ and a Very-Coarse spray by the $D_{v0.5}$, the AIXR 11004 an Extremely-Coarse spray, and the TTI 11002 and 11004 both as an Ultra-Coarse spray (Table 2).

Application day weather

The weather conditions during application are summarized in Table 3. The study area comprised a 2 by 2 m block and applications were completed within 2 h, thus the weather conditions did not affect the droplet deposition results.

Discussion

Coverage results across collector type

MW, WSP and KK collectors resulted in the same trends across nozzle type, where the 1.6 l · min^{-1} (0.4 gal · min^{-1}) flow rate nozzle types had a higher coverage with all collectors. Though the 0.8 l · min^{-1} nozzles had half of the volume rate of the 1.6 l · min^{-1} nozzles, they resulted in a higher coverage relative to their application rate (Table 1). This suggests that flow rate volume has a linear relationship with application volume rate, and lower volumes can result in higher coverage as has been observed in prior studies (Fritz *et al.* 2005). MW had the highest coverage percentages, where WSP collectors resulted in a 2% decrease, KK collectors a 35% decrease in coverage and MIA collectors showed a 77% decrease in coverage compared to MWs across nozzle types. This difference is due to the spread factor and wettability differences of WSP compared to KK and Mylar (MW and MIA) collectors. WSP collectors have the greatest spread factor, and Mylar collectors the least, for any given solution. The

Table 3. Weather data for the day of application on July 28th, 2015. Weather data taken was summarized only for the hours of the day during which spraying occurred

		Average relative		
temperature	dew point	humidity	wind speed direction	gust
[°C]	[°C]	[%]	[km · h^{-1}]	[km · h^{-1}]
26	12	42	11 N	19

reverse is true due to the lack of wettability of Mylar collectors where droplets prior to liquid evaporation spread to greater areas than WSP collectors (Forster *et al.* 2014).

Nozzle type affected the coverage across collector types, where the lowest coverage was observed with Ultra-Coarse nozzles (Tables 1 and 2). Coverage was not different even with significant changes in droplet size across collector type. MIA observed no difference in coverage regardless of nozzle type (Table 1). MW collectors resulted in similar coverage regardless of droplet size among the same flow rate nozzles for all 04 sec and for 02 sec except the XR 11002. KK collectors followed the same trend, except that all 02 sec and all 04 sec resulted in similar coverage regardless of droplet size.

MW collectors had the highest coverage with the TTI 11004, WSP collectors with the AIXR 11004, KK collectors with the XR 11004, and MIA Collectors with the XR and TTI 11004. The AIXR and XR 11002 had identical coverage with KK collectors, and similar coverage with WSP collectors, but a visible decrease in coverage was observed with the TTI. This shows that with the right nozzle choice, coverage can be maintained, along with a reduction in drift potential of a given application.

Previous studies using Mylar collectors have used the washing and fluorescence method but did not analyze them using image analysis (Creech 2015). This study is the first of its kind to compare MIA, WSP and KK collectors for droplet deposition using identical methods. As the MIA cards do not stain like KK collectors, the coverage analyzed is most similar to results that would be observed on hard-to-wet leaf surfaces as observed in previous studies (Forster *et al.* 2014). The MIA collectors were the least susceptible to changes in droplet size, but still resulted in a clear trend of increased coverage with increased application volume rate.

Droplet number density across collector type

MIA collectors resulted in the highest droplet number densities followed by KK and finally WSP collectors. KK collectors showed a 45% decrease and WSP collectors a 55% decrease in droplet number densities compared to MIA collectors (Table 1). This result indicates that MIA collectors are able to detect droplets that are not visible with WSP or KK collectors. Previous research has shown that WSP collectors cannot detect droplets smaller than 50 μm (Hoffmann and Hewitt 2005) thereby suggesting the usefulness of using all three types to classify a spray.

There was not a clear increase in the droplet number densities with the increase in application volume rate observed with all three collector types, except the TTI 11004 which had a greater droplet number density than the TTI 11002.

Comparison of MW to MIA for coverage

MW collectors should have the same visible coverage that MIA collectors do if they too were allowed to dry. One drawback from MIA collectors is that they took the longest to dry and were not easy to scan as they are transparent. The differences observed between the coverage of MW and MIA collectors is due purely to the method of measurement for them. Image analysis can only provide two-dimensional understanding of coverage, but, does not quantify the deposition of the spray in terms of concentration – which may be of immense importance in efficacy situations. In prior canopy penetration studies, when the concentration of an active ingredient was quantified to determine canopy penetration, coarser sprays were observed to have the best canopy penetration (Zhu *et al.* 2004; Derksen *et al.* 2008, 2014). Results from this study confirm that result, as the TTI 11004, the coarsest spray in the study showed the greatest coverage with MW. The understanding of the dose applied and how evenly the application is made can be understood through a comparison of MIA and MW collectors. This result is also germane to the result where the TTI 11004 (an Ultra-Coarse spray quality) had the highest coverage from MW even though KK and WSP observed lower coverages than the AIXR or XR. This suggests that the TTI deposited more dye, even if it was not as widely distributed on collectors as XR and AIXR treatments.

Assessing the four collector type results

Results from this study suggest a strong correlation to data produced with WSP and MW collectors, as there was full agreement between both types except for the TTI 11004 (Table 1). Using both collector types in the same study would allow for a visual understanding of the distribution of the spray, while also giving an idea of the concentration of that distribution. Previous work with KK found it to be useful in multiple condition types, even in dense and wet canopies (Ferguson *et al.* 2016). Results suggest that previous work using the MW method (Creech 2015) can be compared to work using WSPs (Wolf and Daggupati 2009), if the same nozzle and pressure combination is featured.

Conclusions

Using multiple collector types helps to quantify all the droplet deposition occurring from a spray. While previous studies have utilized a single type of artificial collector for characterizing droplet coverage and droplet number density, results from this study suggest that these two-dimensional collectors are not presenting the full scope of the deposition. MW and WSP had nearly identical results, thus if used in tandem can provide an estimation of the spray coverage and the concentration of active ingredients to further improve dosing and application label recommendations. Using Mylar for image analysis provided an interesting snapshot in understanding the small droplets that deposit but are often not visible on WSP or KK cards. In order to optimize technology selection, using multiple artificial collectors can properly characterize the spray deposition and help in selecting the best technology to reduce spray drift, yet provide the best coverage for maximum efficacy.

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