FLUOPICOLIDE, INDOXACARB, AND OXATHIAPIPROLIN RESIDUES AFTER APPLICATION TO FLUE-CURED TOBACCO (*Nicotiana tabacum* L.)

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Pesticide residues found on cured tobacco (*Nicotiana tabacum* L.) remain a large concern to the allied tobacco industry. To quantify maximum expected cured leaf residues, 3 active ingredients (fluopicolide, indoxacarb, and oxathiapiprolin) were applied to flue-cured tobacco grown in 6 North Carolina environments from 2016 to 2018. Fluopicolide residues were consistently among the highest documented in this evaluation (7.25 mg/kg maximum), which was most likely a result of the compound having the shortest preharvest interval (PHI; 7 days) among the products tested. The highest indoxacarb residue was 2.15 mg/kg, which was identified in lower-stalk-position samples collected from 1 environment in 2018. Additional data suggest that indoxacarb residues are likely to be <2.0 mg/kg. Oxathiapiprolin was below the limit of quantification (0.09 mg/kg) in 98.6% of the samples analyzed and averaged 0.10 mg/kg in the lower-stalk position of 1 environment in 2017. It is plausible that residues from commercial farming operations would be lower than those reported because of integrated pest management (IPM) practices. Further investigations are warranted to better identify residues resulting from applications delivered using recommendations put forth by Cooperative Extension Services in the southern United States.

Additional key words: crop protection agents, insecticide, fungicide

INTRODUCTION

As part of an ongoing research program at North Carolina State University, the cured leaf residues of 2 fungicides (fluopicolide and oxathiapiprolin) and 1 insecticide (indoxacarb) were quantified in 6 growing environments over a 3-year period. Fluopicolide (FRAC group 43) and oxathiapiprolin (FRAC group 49) are currently registered in the United States for applications to commercially produced tobacco (5,7). Both active ingredients are primarily used to control black shank (*Phytophthora nicotianae*); however, fluopicolide may also be used to control blue mold (*Peronospora tabacina*; (5,7). Indoxacarb (IRAC group 22A; 4) is not labeled for commercial use in the United States, but has been reported to offer suppression of tobacco budworm (*Heliothis virescens* (Fabricius)), tobacco splitworm (*Phthorimaea operculella*), and tobacco hornworm (*Manduca sexta*; 6). The objective of this study was to establish the maximum expected residues on cured tobacco leaves that would result from a maximum labeled application and minimum PHI of the specified compounds.

METHODS AND MATERIALS

Field experiments were conducted in 2016, 2017, and 2018 at the Upper Coastal Plain Research Station (UCPRS) near Rocky Mount, NC and the Lower Coastal Plain Research Station (LCPRS) in Kinston, NC. Tobacco was produced using practices recommended by the North Carolina Cooperative Extension Service (3), with the exception of treatments imposed. The cultivar 'NC 196' (Goldleaf Seed Co., Hartsville, SC) was planted in all environments. Individual plots were treated with 1 of 3 pesticides: fluopicolide (Presidio®, Valent U.S.A. LLC, Walnut Creek, CA), indoxacarb (Steward® EC, FMC Corporation, Philadelphia, PA), or oxathiapiprolin (Orondis® Gold 200, Syngenta Crop Protection, LLC, Greensboro, NC).

Fluopicolide was applied 3 times during each growing season: 1 soil application immediately after lay-by (140 g active ingredient [a.i.]/ha) and 2 foliar applications (140 g a.i./ha per application) each with a targeted PHI of 14 and 7 days, respectively. Indoxacarb was applied in 4 foliar applications targeted to 32, 27, 22, and 17 days before first harvest. Each application of indoxacarb delivered 124 g a.i./ha. Oxathiapiprolin was applied once as a soil application immediately after lay-by (71 g a.i./ha). The PHI for oxathiapiprolin is 7 days; however, given that the single application of the pesticide occurred anywhere from 4 to 8 weeks after transplanting, the PHI was not considered for the purpose of this evaluation. Lay-by applications were done with a CO_2-pressurized backpack equipped with a single wide-angle flat spray nozzle (TK-VS2, TeeJet Spraying Systems Co., Wheaton, IL) calibrated to deliver 187 L/ha directed to the row middle. Foliar applications were made using a CO_2-pressurized backpack sprayer calibrated to deliver 187 L/ha at 172–207 kPa. A 3-nozzle boom with 26-cm nozzle spacing was equipped with TX-12 hollow cone (TeeJet) spray tips oriented 45 cm above the center of the plant. Outside nozzles were angled 45° toward the center of the boom. Nozzle arrangement and orientation were selected to ensure maximum exposure of plants to the selected materials.

Treatments were replicated 4 times and arranged in a randomized complete block design in all growing environments. All 4 rows in each plot were treated, with the 2 center rows being harvested, cured, and sampled for residue analysis. Row spacing and plot dimensions

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Table 1. Transplanting, pesticide application, harvest dates, and cumulative rainfall in each growing environment.

	2016		2017		2018	
Event[a]	LCPRS[b]	UCPRS[b]	LCPRS	UCPRS	LCPRS	UCPRS
Transplanting	Apr. 21	Apr. 26	Apr. 20	Apr. 21	Apr. 30	Apr. 19
Fluopicolide – App. 1	May 25	June 13	June 06	June 15	June 19	June 19
Fluopicolide – App. 2	July 06	July 18	July 06	July 13	July 20	July 16
Fluopicolide – App. 3	July 13	July 25	July 12	July 21	July 27	July 23
Indoxacarb – App. 1	June 17	June 30	June 16	June 26	July 03	June 28
Indoxacarb – App. 2	June 23	July 05	June 20	June 30	July 06	July 02
Indoxacarb – App. 3	June 28	July 11	June 27	July 07	July 11	July 09
Indoxacarb – App. 4	July 02	July 15	July 02	July 10	July 16	July 13
Oxathiapiprolin – App. 1	May 25	June 13	June 06	June 15	June 19	June 19
1st Harvest	July 20	Aug. 04	July 19	Aug. 02	Aug. 14	Aug. 09
2nd Harvest	Aug. 09	Aug. 24	Aug. 09	Aug. 15	Aug. 21	Aug. 24
3rd Harvest	Aug. 31	Sep. 09	Sept. 12	Aug. 22	Sept. 04	Sept. 14
4th Harvest	Sep. 01	Sep. 09	Sept. 21	Aug. 22	Sept. 04	Sept. 19
Cumulative Rainfall (mm)[c]	574	899	681	691	624	664

[a] App., application
[b] LCPRS, Lower Coastal Plain Research Station in Kinston, NC; UCPRS, Upper Coastal Plain Research Station near Rocky Mount, NC.
[c] Cumulative rainfall represents the period from transplanting to 4th harvest within each environment.

varied between locations, with plant spacing at the UCPRS being 55 cm by 121 cm and plant spacing at the LCPRS being 55 cm by 111 cm. Transplanting dates, pesticide application dates, and harvest dates varied by location as well and are presented in Table 1. Four stalk positions (lugs, cutter, leaf, and tip) were harvested and bulk-cured on each research station. After curing, 100-g lamina samples absent of midribs were collected from individual stalk positions (lower, middle, and upper stalk) and analyzed by Global Laboratory Services, Inc. in Wilson, NC. Harvest intervals 1 and 2 (lug + cutter) were combined and are represented in the "lower" stalk position, harvest 3 (leaf) is represented in the "middle" stalk position, and harvest 4 (Tip) is represented in the "upper" stalk position (Table 1; Figure 1). Analysis of variance was conducted using the PROC Mixed procedure in SAS ver. 9.4 (SAS Institute Inc., Cary, NC) to test pesticide residue differences among stalk positions. In the analysis, pesticide compound was considered to be a fixed factor, whereas environment and replication were considered as random factors. Means were separated using Fisher's Protected LSD at $P \leq 0.05$. Because of significant residue-by-environment interactions, results for each compound are presented by individual growing environments. Figures were created using SigmaPlot version 14.0 (Systat Software, Inc., San Jose, CA).

RESULTS AND DISCUSSION

In 3 growing environments (LCPRS-2016, LCPRS-2017, and UCPRS-2018), fluopicolide residues were greatest in samples collected from lugs and cutters, which comprised lower-stalk leaf samples (Figure 1). In each scenario, residues on lower-stalk positions were 0.85 to 5.37 mg/kg greater than those documented in the tip leaf (upper-stalk) position. Higher residue detection in the lower-stalk positions is most likely a result of the narrow window (7 to 18 days) between final application of fluopicolide and first harvest. Regardless, the residue data obtained in the present study are 3 to 5 times lower than tolerances established for U.S. food crops with similar morphology to tobacco. For example, the fluopicolide tolerances for *Brassica* and non-*Brassica* leafy vegetables are 18 and 25 mg/kg, respectively (1). Last, the application pattern used in this study does not reflect practical use patterns for flue-cured tobacco systems in North Carolina. Foliar applications of fluopicolide for blue mold control would not be commonly used because of low occurrence of the disease. In addition, consecutive applications of the active ingredient would not be recommended by Cooperative Extension to reduce the potential for pathogen resistance.

Similar trends were observed after indoxacarb application. In the LCPRS-2016 and UCPRS-2018 environments, residues were significantly lower in upper- and middle-stalk leaves relative to those reported in lower-stalk leaves (Figure 1). Cured leaf residues were not different among stalk positions in the other environments. The targeted PHI within indoxacarb treatments was 17 days, but ranged from 17 to 28 days among all environments. It is plausible that the longer PHI may prove favorable for low indoxacarb residues. Overall, the results from this study indicate that multiple applications of indoxacarb should result in cured leaf residues that are much less than the CORESTA guidance residue limit (15 mg/kg; 2) but greater than the limit of quantification (0.09 mg/kg). Given the low residues resulting from repeated indoxacarb applications and the compound's reported efficacy against lepidoptera insects, it appears to be a suitable candidate for commercial registration in the United States and may be a viable option for commercial farmers.

Oxathiapiprolin residues were below the limit of quantification (0.09 mg/kg) in all stalk positions sampled among 5 growing environments (data not shown).

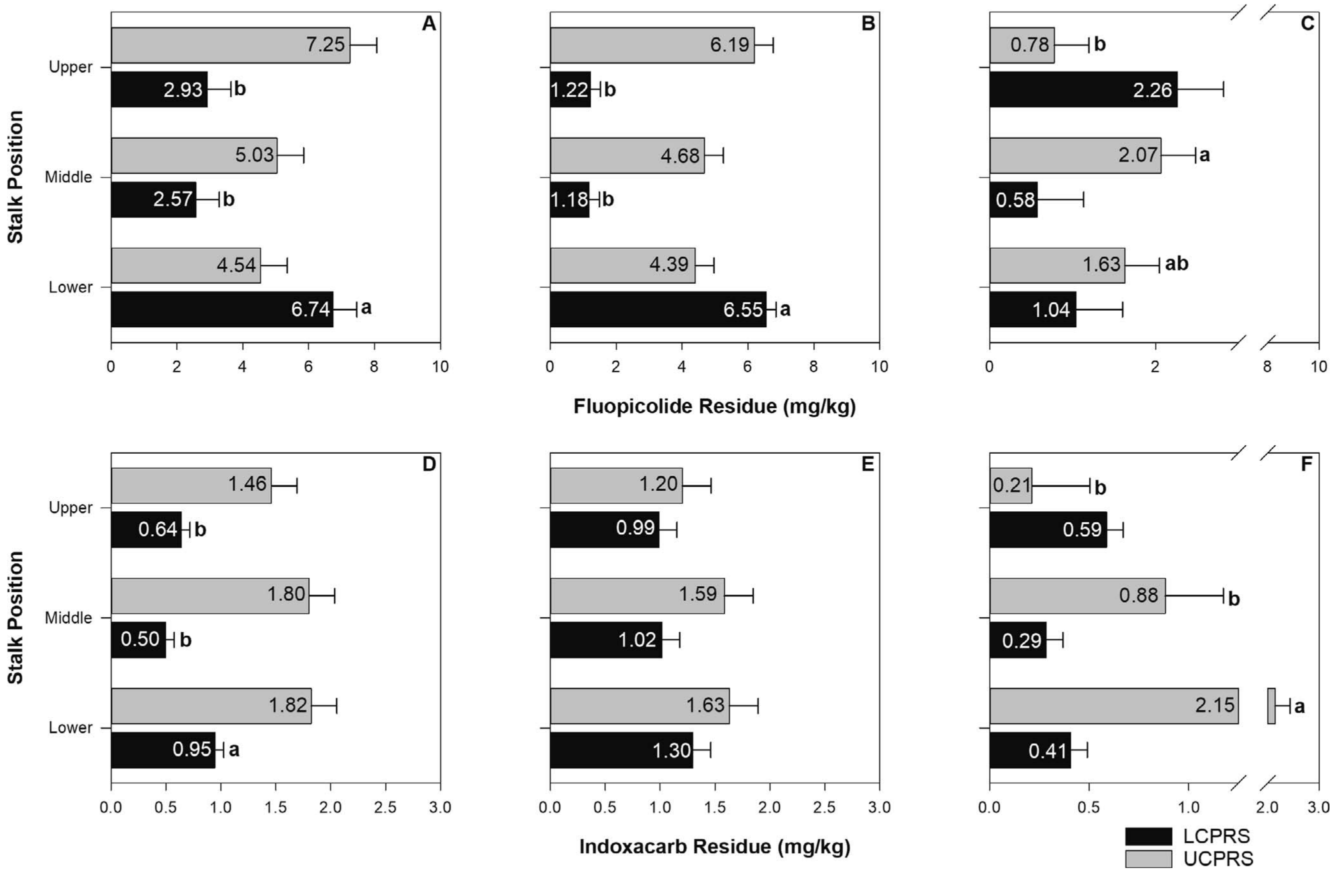

Figure 1. Cured leaf residues after applications of fluopicolide (A–C) and indoxacarb (D–F) in 2016 (A,D), 2017 (B,E), and 2018 (C,F) in the LCPRS and UCPRS. Limits of quantification for fluopicolide and indoxacarb were 0.08 and 0.09 mg/kg, respectively. Stalk positions are comprised of the following market groups: lower, lug and cutter; middle, leaf; upper, tip.

Oxathiapiprolin was detected in lower-stalk positions samples collected within the LCPRS-2017 environment (0.10 mg/kg), but not in middle- and upper-stalk positions. The application program evaluated (soil-applied only) would be very unlikely to result in residue issues that would be of concern to industry. However, further research is warranted to quantify residues that may result from transplant water or foliar applications of oxathiapiprolin.

CONCLUSION

On the basis of the observations made in this study, the residues of fluopicolide, indoxacarb, and oxathiapiprolin appear to be relatively minimal. Likewise, it is highly probable that their inclusion in a modern IPM system that is designed to both minimize and rotate pesticide applications would be widely accepted by Cooperative Extension, allied industry, and commercial producers. Although it is difficult to surmise the ultimate impact of each chemical to cured leaf residues, it is the opinion of the authors that those evaluated should be considered for use in flue-cured tobacco production.

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