Beiträge zur Tabakforschung · Band 8 · Heft 4 · Dezember 1975 DOI: 10.2478/cttr-2013-0386

Residue Levels of Fatty Compounds and Surfactants as Suckering Agents on Tobacco*

by T. C. Tso and H. Chu

Beltsville Agriculture Research Center, Northeastern Region, Agricultural Research Service, U.S. Department of Agriculture, Beltsville, Maryland, U.S.A.

and

D. W. DeJong

Tobacco Research Station, Southern Region, Agricultural Research Service, U.S. Department of Agriculture, Oxford, North Carolina, U.S.A.

INTRODUCTION

Decapitation (topping) at onset of flowering is a standard practice in the production of tobacco. Subsequently, axillary buds will grow into branches, known as suckers, because of the removal of apical dominance. The operation for the removal of these suckers is called "suckering". Materials used to inhibit the growth of axillary buds into suckers are termed as "suckering agents". The majority of the suckering agents are either lost or decomposed during the period of tobacco growth and curing, but some may remain in or on the cured leaf. This paper reports the residues of fatty ester and alcohol used as suckering agents in the field which remained in the cured leaves of Maryland, Burley, and Bright experimental tobaccos.

Many lower alkyl esters and alcohols showed various degrees of effectiveness for sucker inhibition (4). The most effective ones are saturated, 8 to 12 carbon straight chain esters and alcohols, especially those with 10 carbons (2, 3). The commonly used ester for field application is methyl caprate, and the commonly used alcohol is a mixture of 1-octanol and 1-decanol. The surfactant for ester is polyoxyethylene [20] sorbitan monolaurate (Tween 20)**, and that for alcohol is polyoxyethylene [20] sorbitan monooleate (Tween 80). Since fatty compounds are naturally occurring products in tobacco, labeled materials were used as tracers in this recovery study. ^{14}C lauric acid derivatives were used as they were readily available. The Tween surfactants with ^{14}C-labeling were supplied to us as a courtesy of ICI United States, Inc. (formerly the Atlas Chemical Industries, Inc.).

* Received for publication: 5th May, 1975.

** Mention of a trademark or proprietary product does not constitute a guarantee or warranty of the product by the U. S. Department of Agriculture, and does not imply its approval to the exclusion of other products that may also be suitable.

MATERIALS AND METHODS

Tobacco Plants: Three types of tobacco (*Nicotiana tabacum* L.) were used in this study, including cv. Maryland Catterton, Burley 21, and N.C. 95. The first two represent Maryland and Burley types and were grown and air-cured at Beltsville, Maryland, and the last one represents Bright type tobacco, grown and flue-cured at Oxford, North Carolina.

These plants were field-grown under regular culture practices and cured according to type. For sucker chemical tests of Maryland and Burley types, three plants were used for each treatment. Each plant received the chemicals once and was harvested two weeks after treatment. The air-cured leaves from three plants within each treatment were combined, and grouped into three composite samples according to top, middle, and bottom stalk positions. For Bright type tobacco, five plants were used for each treatment. Each plant received the chemicals twice, the second application was applied two weeks after the first. This type of tobacco was harvested by leaf priming and then was flue-cured. The first priming was made one week after the first treatment, the second priming was three weeks after first treatment (or one week after second treatment), and the third or last priming was five weeks after the first treatment (or three weeks after second treatment). Leaves from five plants of same priming within each treatment were combined into one composite sample.

Suckering Materials and Field Treatments: Chemicals used included the following: Methyl caprate, a mixture of 1-octanol and 1-decanol (approximately 45—55), methyl laurate, lauryl alcohol, lauric acid-1-^{14}C methyl ester, lauryl-alcohol-1-^{14}C, Tween 20, Tween 20-^{14}C (either ^{14}C-1-fatty acid, or ^{14}C-U-ethylene oxide), Tween 80, and Tween 80-^{14}C (either ^{14}C-1-fatty acid, or ^{12}C-U-ethylene oxide).

Table 1. Description of materials and dosage used for each plant.

Treatment code	Materials and combination	^{14}C-activity (CPM)	
		Maryland & Burley types	Bright type
1	830 mg methyl caprate + 320 mg Tween 20	–	–
2	636 mg mixture of 1-octanol and 1-decanol + 480 mg Tween 80	–	–
3	960 mg methyl laurate + 320 mg Tween 20	–	–
4	750 mg lauryl alcohol + 480 mg Tween 80	–	–
5	830 mg methyl caprate + 320 mg Tween 20 (^{14}C-1-fatty acid)	5.73×10^7	1.26×10^8
6	830 mg methyl caprate + 320 mg Tween 20 (^{14}C-U-ethylene oxide)	1.16×10^8	1.37×10^8
7	636 mg mixture of 1-octanol and 1-decanol + 480 mg Tween 80 (^{14}C-1-fatty acid)	6.60×10^7	1.16×10^8
8	636 mg mixture of 1-octanol and 1-decanol + 480 mg Tween 80 (^{14}C-U-ethylene oxide)	6.80×10^7	1.30×10^8
9	960 mg methyl laurate (^{14}C-1-lauric acid methyl ester) + 320 mg Tween 20	5.30×10^7	8.40×10^7
10	750 mg lauryl alcohol (^{14}C-1-lauryl alcohol) + 480 mg Tween 80	5.66×10^7	1.40×10^8

The exact combination of these active materials and surfactants, the rate of application, and the total level of ^{14}C-activity are shown in Table 1, together with the assigned code for each treatment.

Residue Determination: The combined cured leaf samples were ground and well mixed. A 10 g subsample from each treatment was extracted with 100 ml 70% ethanol in a Waring Blender for 10 minutes. Following filtration and concentration, an aliquot representing 100 mg of original tobacco sample was used for ^{14}C-counting in a toluene cocktail. Data obtained from treatments codes 1, 2, 3, and 4 were used for ^{14}C background correction of corresponding treatments.

Residue data are calculated based on ^{14}C-recovery.

RESULTS

Total yield of cured leaf from each treatment is listed in Table 2. These composite samples represented materials of three plants from Maryland and Burley types, and five plants from the Bright type. The ^{14}C-activity of composite sample from each type and the percentage of ^{14}C-recovery are listed in Tables 3, 4, and 5 for Maryland, Burley, and Bright tobaccos, respectively. Generally, the average recovery of ^{14}C-activity was low. The ^{14}C-labeled ethylene oxide moiety of Tween compounds appeared to be more stable than ^{14}C-labeled fatty acid moiety of the same compounds and thus resulted in an apparently higher ^{14}C-recovery of the former treatments.

The ^{14}C-recovery for Maryland and Burley types was the highest in top leaves where most of the chemical sprays were directly applied. As expected, the percentage of recovery was gradually reduced toward middle portion of the plant, and the lowest recovery was obtained for the bottom leaves. However, the highest ^{14}C-recovery for Bright type tobacco was usually in the second priming or at the middle position. This result may have reflected the effect of the second chemical

Table 2. Yield of composite samples from each treatment according to stalk positions or primings.

Treatment code*	Maryland Catterton				Burley 21				N. C. 95			
	Bottom g	Middle g	Top g	Total g	Bottom g	Middle g	Top g	Total g	1st priming g	2nd priming g	3rd priming g	Total g
1	49	46	86	181	84	72	85	241	202	239	173	614
2	83	76	79	238	86	84	108	278	247	250	345	842
3	103	97	97	297	109	110	83	302	188	255	257	700
4	98	95	82	275	126	123	119	368	225	237	295	759
5	66	47	72	185	105	72	100	277	163	283	252	698
6	69	74	76	219	97	80	168	345	160	187	195	542
7	62	93	88	243	92	73	116	281	201	257	304	762
8	111	100	135	346	114	119	159	392	171	290	367	828
9	107	114	98	319	128	117	120	365	148	215	214	577
10	100	86	131	317	97	85	92	274	178	233	296	707

* See Table 1.

Table 3. ^{14}C-activity of composite Maryland tobacco samples.

Treatment code*	Bottom			Middle			Top			Average recovery (%)
	100 mg	Total CPM	Recovery (%)	100 mg sample CPM	Total CPM	Recovery (%)	100 mg sample CPM	Total CPM	Recovery (%)	
1	18.2	8.9×10^2	–	17.4	8.0×10^2	–	29	2.49×10^3	–	–
2	19.3	1.60×10^3	–	21.9	1.66×10^3	–	16.3	1.28×10^3	–	–
3	21.9	2.25×10^3	–	23.8	2.30×10^3	–	23.3	2.26×10^3	–	–
4	27.0	2.65×10^3	–	27.2	2.58×10^3	–	20.8	1.70×10^3	–	–
5	133.8	8.83×10^3	.005	174.2	8.18×10^3	.004	336.7	2.42×10^4	.012	.007
6	422.1	2.01×10^4	.008	1124.2	8.31×10^4	.024	2552.9	1.94×10^5	.054	.028
7	196.7	1.22×10^4	.005	283.6	2.63×10^4	.012	315.5	2.77×10^4	.013	.010
8	147.7	1.64×10^4	.007	347.7	3.47×10^4	.016	518.8	6.86×10^4	.033	.018
9	72.2	7.72×10^3	.003	166.8	1.90×10^4	.010	600.3	5.88×10^4	.035	.016
10	99.6	9.96×10^3	.004	235.8	2.02×10^4	.010	607.4	7.65×10^4	.044	.019

Table 4. ^{14}C-activity of composite Burley tobacco samples.

Treatment code*	Bottom			Middle			Top			Average recovery (%)
	100 mg sample CPM	Total CPM	Recovery (%)	100 mg sample CPM	Total CPM	Recovery (%)	100 mg sample CPM	Total CPM	Recovery (%)	
1	20.3	1.70×10^3	–	21.9	1.57×10^3	–	16.1	1.36×10^3	–	–
2	22.5	1.93×10^3	–	20.0	1.68×10^3	–	16.9	1.82×10^3	–	–
3	22.4	2.44×10^3	–	3.4	3.74×10^2	–	14.8	1.22×10^3	–	–
4	23.5	2.96×10^3	–	19.1	2.35×10^3	–	16.3	1.93×10^3	–	–
5	43.1	4.52×10^3	.002	80.7	5.81×10^3	.002	177.1	1.77×10^4	.009	.004
6	152.4	1.48×10^4	.004	220.0	1.89×10^4	.005	940.3	1.57×10^5	.044	.017
7	51.1	4.70×10^3	.001	148.2	1.08×10^4	.005	347.4	4.02×10^4	.020	.003
8	75.8	8.64×10^3	.003	137.4	1.63×10^4	.007	384.1	6.10×10^4	.029	.013
9	74.5	9.63×10^3	.004	153.8	1.79×10^4	.009	721.8	8.66×10^4	.054	.022
10	102.5	9.94×10^3	.004	207.4	1.76×10^4	.009	469.7	4.32×10^4	.024	.012

Table 5. ^{14}C-activity of composite Bright tobacco samples.

Treatment code*	1st priming			2nd priming			3rd priming			Average recovery (%)
	100 mg sample CPM	Total CPM	Recovery (%)	100 mg sample CPM	Total CPM	Recovery (%)	100 mg sample CPM	Total CPM	Recovery (%)	
1	10.0	2.02×10^3	–	7.9	1.88×10^3	–	10.0	1.73×10^3	–	–
2	11.9	2.93×10^3	–	3.6	9.00×10^2	–	53.5	1.84×10^4	–	–
3	13.2	2.48×10^3	–	1.5	3.82×10^2	–	11.9	3.05×10^3	–	–
4	12.8	2.43×10^3	–	5.0	1.18×10^3	–	11.7	3.45×10^3	–	–
5	68.0	1.11×10^4	0.001	860.8	2.43×10^5	0.038	798.1	2.01×10^5	0.031	0.023
6	102.7	1.64×10^4	0.002	7785.7	1.45×10^6	0.211	7020.0	1.36×10^6	0.198	0.137
7	65.0	1.30×10^4	0.002	1685.6	4.33×10^5	0.074	869.7	2.64×10^5	0.042	0.039
8	79.5	1.35×10^4	0.002	1427.1	4.13×10^5	0.063	1603.4	5.88×10^5	0.087	0.050
9	48.3	7.14×10^3	0.001	181.3	3.89×10^4	0.009	366.7	7.84×10^4	0.018	0.009
10	87.3	1.55×10^4	0.002	477.5	1.11×10^5	0.016	964.7	2.85×10^5	0.040	0.019

* See Table 1.

Table 6. Calculated residual levels of test materials in tobacco leaf.

Treatment code*	^{14}C-labeled test material	Maryland Catterton		Burley 21		N. C. 95	
		Recovery of material (mg)	Residue on leaf (ppm)	Recovery of material (mg)	Residue on leaf (ppm)	Recovery of material (mg)	Residue on leaf (ppm)
5	Tween 20 ^{14}C-1-fatty acid	0.067	0.363	0.038	0.138	0.368	0.527
6	Tween 20 ^{14}C-U-ethylene oxide	0.269	1.227	0.163	0.473	2.192	4.044
7	Tween 80 ^{14}C-1-fatty acid	0.144	0.592	0.115	0.409	0.936	1.228
8	Tween 80 ^{14}C-U-ethylene oxide	0.259	0.748	0.187	0.477	1.200	1.449
9	^{14}C-1-lauric acid methyl ester	0.461	1.445	0.633	1.734	0.432	0.748
10	^{14}C-1-lauryl alcohol	0.427	1.348	0.270	0.985	0.712	1.007

* See Table 1.

treatment which was applied only one week before this priming. The average residue levels remaining on tobacco leaf were calculated, as shown in Table 6. The calculation was based on percent of ^{14}C-recovery from each treatment of each tobacco type. Lauric acid methyl ester residues were 1.45, 1.73, and 0.75 ppm, and lauryl alcohol residues were 1.35, 0.99, and 1.01 ppm for Maryland, Burley, and Bright tobacco, respectively. Calculated residue levels for the Tween materials varied widely depending on position of ^{14}C-labeling; range was between 0.14 and 4.04 ppm. The general average for the residue level of Tween compounds was approximately 0.5 ppm based on fatty acid moiety, and 1.4 ppm based on ethylene oxide moiety.

DISCUSSION AND CONCLUSION

A separate study on the fate of fatty compounds and surfactants applied on tobacco (1) revealed that there was interconversion among methyl laurate, lauryl alcohol, and lauric acid during the 16 and 144 hour sampling of fresh tobacco materials. Since results reported here were based on recovery of ^{14}C-activity which was labeled at the 1-position to carbonyl or alcoholic hydroxyl groups, the calculated fatty residues may, therefore, include the summation of acid, alcohol, and ester resulting from interconversion of the applied material. It was also found that all the Tween materials remaining on the tobacco were hydrolyzed *in situ* (1). The calculated residual data from Tweens reported here may either reflect fatty ester (laurate or oleate), or polyethoxylated polyol, depending on whether the labeling was at fatty acid or ethylene oxide moiety, respectively. The maximum calculated recovery of Tween material observed in these tests was 4 ppm; hydrolyzed fatty materials originated from Tweens would be only a small fraction of the Tweens.

In one of our preliminary tests involving Maryland and Burley tobacco types with which we used ^{14}C-labeled methyl laurate and lauryl alcohol, we found an average of 4.8 ppm residue. The present study showed an average residue of only 1.6 ppm fatty compound and approximately 1.0 ppm Tween residue. The combined total is about 2.6 ppm residue level which is much lower than earlier findings.

The naturally occurring fatty acid derivatives in cured leaf tobacco are around 7,000 ppm (4). The total lipid fraction in leaf tobacco is approximately ten times greater than the level of fatty compounds. It is apparent that the residue level of fatty compounds used as suckering agent, in the range reported in this paper, would not affect leaf quality or usability.

SUMMARY

Fatty compounds including lauryl alcohol and methyl laurate and Tween 20 surfactant (polyoxyethylene [20] sorbitan monolaurate) and Tween 80 surfactant (polyoxyethylene [20] sorbitan monooleate) with ^{14}C-labeling at various positions were used as suckering agents for Maryland, Burley, and Bright tobacco types (*Nicotiana tabacum* L.) and their residues on the tobacco determined. An average residue of 1.61 ppm of fatty compounds and 1.0 ppm of surfactants were found. The combined total of 2.6 ppm residue due to these suckering agents is far below an earlier preliminary test of 4.8 ppm of residue in comparison with 7,000 ppm naturally occurring fatty compounds in tobacco.

ZUSAMMENFASSUNG

Fettartige Verbindungen wie Laurylalkohol und Methyllaurat sowie die oberflächenaktiven Substanzen Tween 20 (Polyoxyäthylen[20]sorbitan-monolaurat) und Tween 80 (Polyoxyäthylen[20]sorbitan-monooleat) mit ^{14}C-Markierung in verschiedenen Positionen wurden als Mittel zur Kontrolle des Geizenwachstums bei Maryland-, Burley- und Bright-Tabaken (*Nicotiana tabacum* L.) benutzt und ihre Rückstände im Tabak untersucht. Der durchschnittliche Rückstandsgehalt belief sich auf 1,61 ppm bei den fettartigen Verbindungen und auf 1,0 ppm bei den oberflächenaktiven Substanzen. Der Gesamtwert von 2,6 ppm für Rückstände dieser Wachstumsregler liegt weit unter dem Ergebnis eines früheren Vorver-

suchs mit 4,8 ppm im Vergleich zu dem natürlichen Vorkommen von fettartigen Verbindungen im Tabak in Höhe von 7000 ppm.

RESUME

On a employé comme agents pour l'ébourgeonnement de tabac Maryland, Burley et Bright (*Nicotiana tabacum* L.) les composés gras suivants: alcool laurylique, laurate de méthyle, Tween 20 surfactant (polyoxyéthylène[20]monolaurate de sorbitan) et Tween 80 surfactant (polyoxyéthylène[20]mono-oléate de sorbitan) marqués au carbone 14 à différentes positions. On a déterminé leur résidu dans le tabac. Des résidus moyens de 1,61 ppm de composés gras, et 1,0 ppm de surfactants ont été retrouvés. Le résidu total combiné de 2,6 ppm dû aux agents d'ébourgeonnement en question est de beaucoup inférieur à 4,8 ppm trouvé dans des tests préliminaires, surtout si l'on compare au 7.000 ppm des corps gras se trouvant naturellement dans le tabac.

REFERENCES

1. Tso, T. C., and H. Chu: The fate of fatty compounds and surfactants used as sucker control agents on field tobacco; 1974, in preparation.
2. Steffens, G. L., T. C. Tso, and D. W. Spaulding: Fatty alcohol inhibition of tobacco axillary and terminal bud growth; Agriculture and Food Chemistry 15 (1967) 972–5.
3. Tso, T. C.: Plant growth inhibition of some fatty acids and their analogues; Nature 202 (1964) 511–12.
4. Tso, T. C.: Physiology and biochemistry of tobacco plants; Dowden, Hutchinson and Ross, Strousburg, Pa., 1972, pp. 129–139 and pp. 272–281.

Acknowledgment

We thank the ICI United States, Inc. (formerly Atlas Chemical Industries, Inc.) for providing labeled test compounds, and Frank Sharp and Mary E. Engelhaupt for their technical assistance.

The authors' address:

Beltsville Agriculture Research Center, Northeastern Region, Agricultural Research Service,
U.S. Dept. of Agriculture, Beltsville, Maryland, 20705.