Pyrolytic Evaluation of Close-Grown Tobaccos*

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INTRODUCTION

The purpose of this study was to evaluate two close-grown tobaccos by a method recently developed in this laboratory (1) for pyrolytically screening new tobacco products or varieties for selected pyrolysis and corresponding cigarette smoke components. Close-grown tobacco is tobacco that has been planted two to three times as dense as normal tobacco planting with the objective of mechanically harvesting the tobacco at one time. Thus, the total plant would be used in cigarette manufacture. Not only are the labor savings attractive, but there has also been evidence that smoke condensate from close-grown tobacco cigarettes is not as toxic in animal tests (2, 3). However, the exact smoke composition and the reasons for the reduction of activity for this condensate have not been established. The pyrolytic data would help to predict the distribution of significant compounds [phenols, nicotine, fatty acids, sterols, polynuclear aromatic hydrocarbons (PAH) and aliphatic hydrocarbons] in the smoke from the tobacco samples. An indication of the possible biological activity of smoke condensate, based on yields of PAH and phenols, would be obtained. By pyrolyzing a small quantity of the tobacco sample and by analyzing the pyrolyzate, one could avoid the costly and time-consuming process of growing a large quantity of tobacco, as required in the making and smoking of cigarettes, and the cigarette smoke analyses.

Although pyrolysis products are not an exact representation of mainstream smoke distribution and composition, pyrolysis approximates smoke formation and is useful in evaluating non-cigarette tobacco samples. We have developed pyrolysis conditions that simulate smoking conditions better than before and produce pyrolysis PAH in the same distribution as smoke PAH. We feel that this pyrolytic method is useful for examining tobacco varieties or genotypes produced under different conditions of growing, harvesting and curing. The small plots of tobacco, that are grown under different experimental agronomic conditions, do not yield sufficient tobacco for cigarette manufacture. Hence, pyrolysis data can provide useful information in guiding plant breeders in their experiments.

Tobacco that shows desirable characteristics of quality, safety, and gives good pyrolytic data would then be grown on a larger scale, made into cigarettes, and further evaluated.

Schlottzauer et al. (4) pyrolyzed tobacco extracts and showed that solanesol is one of the major precursors of smoke PAH and that the sterol esters and sterols are also important PAH precursors. Appleton et al. (5) postulated that triterpenoid material is related to the production of methylated PAH in cigarette smoke. The levels of nicotine and phenolic materials (6) have been long associated with the organoleptic properties of cigarette smoke and these materials have also been related to the total biological activity of smoke (7). This study was undertaken to evaluate pyrolytically close-grown tobacco varieties and to relate pyrolytic products with leaf compounds, with special emphasis on the levels of leaf lipids, which are known precursors of significant smoke PAH. The close-grown tobaccos that we evaluated were Coker 139 (1975) and Speight G-28 (1975). A common variety of flue-cured tobacco, NC 2326 (1976), was used as a reference material and a South Carolina tobacco from 1971 was included for comparison between aged and recent flue-cured varieties.

MATERIALS AND METHODS

Sample Preparation

The NC 2326 flue-cured tobacco and the two close-grown tobaccos, Coker 139 and Speight G-28, were provided by Dr. James F. Chaplin of the United States Department of Agriculture Tobacco Research Laboratory at Oxford, North Carolina. The South Carolina flue-cured tobacco (SC 1971) was purchased commercially. All samples were ground in a Wiley mill with a No. 4 coarse mesh sieve. The NC 2326 and SC 1971 samples consisted of leaf lamina only, while the close-grown tobaccos consisted of whole plants. The Coker 139 sample contained 34.1 % leaf, 64.7 % stalk and 1.2 % flowers. The Speight G-28 sample contained 35.5 % leaf, 59.3 % stalk and 5.2 % flowers. All samples were humidified at 60 % relative humidity for at least 48 hours. A sample

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Figure 1. Extraction and chromatographic separation of leaf lipid compounds.

of the humidified tobacco was used for pyrolysis and dry weight determination.

Separation of Tobacco Lipids

The lipid materials were analyzed (Fig. 1) in tobacco samples by methods previously reported (8). Ground tobacco material (about 1 g) was hydrolyzed for 2 hours by refluxing with 40 ml of 2 N KOH in 85 % ethanol. The hydrolyzates were acidified to pH 2 in an ice bath with conc. HCl and 25 ml of a saturated KCl solution were added. This mixture was filtered and the flask and filter paper were washed with 50 ml of 1:1 benzene:85 % ethanol. The lipids were extracted from the combined solutions with hexane (3 × 50 ml, 1 × 25 ml). The combined hexane extract was taken to dryness on a rotary evaporator and appropriate amounts of internal standards were added. Docosane (0.7 mg/50 mg of hexane extract) was added as an internal standard for the neophytadiene and total hydrocarbon analysis, 1-hexacosanol (0.3 mg/50 mg of hexane extract) for the terpene and fatty alcohol analysis, and nervonic acid (3.0 mg/50 mg of hexane extract) for the solanesol, sterol and fatty acid analysis. The residue containing the lipids was then redissolved and brought to a volume of 10 ml of hexane. A 30–40 mg aliquot of this material was placed on a 5 g Unisil* silicic acid (100–200 mesh, Clarkson Chemical Co., Williamsport, Pa.) column. The column (250 mm × 9 mm inside diameter, equipped with a sintered glass disc and tapered stopcock) was eluted with 100 ml of

hexane to remove the hydrocarbon and neophytadiene fraction, followed by 1900 ml of 25 % benzene in hexane to remove the phytol, paraffinic alcohols and triterpene fraction, and finally with 400 ml of 25 % ether in benzene to remove the fatty acids, sterols and solanesol fraction. The recovery for this total procedure was determined to be greater than 95 % by the use of ¹⁴C-labeled compounds added to tobacco samples prior to hydrolysis. The percent recovery for these labeled compounds was in all cases greater than 95 % (8).

Gas Chromatography (GC) of Lipids

A Hewlett-Packard Model 5750 gas chromatograph with flame ionization detectors, coupled to an Autolab System IV integrator, was used in the hydrocarbon analyses. All calculations were based on unitary detector response values. The hexane eluate, containing the aliphatic hydrocarbons, neophytadiene and docosane, was taken to dryness and redissolved in 100 µl of isooctane. The sample was analyzed on a 2.44 m × 3.2 mm stainless steel GC column of 5 % Dexsil 300 GC on 100/120 mesh Chromosorb W-AW. The GC oven temperature was programmed from 100–350 °C at 4°/min, and the He flow was 50 ml/min.

The benzene-hexane eluate, containing phytol, fatty alcohols, triterpenes and the internal standard 1-hexacosanol, was reduced in volume to 0.5 ml. One-half of this material was transferred to a tapered test tube and evaporated to dryness with a stream of nitrogen. Silylating reagents, 25 µl Trisil Z and 25 µl BSA (Pierce Chemical Co.), were added to the residue and the resulting solution was heated for 30 min at 76 °C. The trimethylsilyl (TMS) derivatives were then analyzed on a 2.44 m × 2 mm glass column of 3 % SP-2250 on 100/120 mesh Supelcoport. Column temperature was programmed at 4°/min from 90–325 °C. The He flow was 30 ml/min. A Hewlett-Packard Model 5830 reporting gas chromatograph was used in these analyses. Component peak areas were corrected for differences in detector response.

The ether-benzene eluate was reduced in volume and 1/3 was taken to dryness in a tapered test tube. Dimethylformamide (25 µl) and BSA (25 µl) were added to the residue and the resulting solution was heated at 76 °C for 15 min. The TMS derivatives of solanesol, fatty acids, sterols, and the internal standard, nervonic acid, were analyzed on a 45 cm × 2 mm glass column of 5 % Dexsil 300 on 100/120 mesh Chromosorb W-AW, programmed from 100–325 °C at 4°/min. The analyses were performed on a Hewlett-Packard Model 5830 reporting gas chromatograph. Peak areas of components were corrected for difference in detector response.

Pyrolysis

Pyrolyses were performed in a moving oven pyrolysis unit, as previously described by Higman et al. (1). Thirty grams of humidified tobacco was distributed uniformly over a 60 cm section of a horizontally positioned 2 cm inside diameter Vycor pyrolysis tube. The oven temperature was set at 700 °C and monitored with a thermo-

* Reference to a company or product name does not imply approval or recommendation by the United States Department of Agriculture.

couple. The nitrogen flow rate through the pyrolysis system was set at 200 ml/min with a bubble-type flowmeter. Oven movement was continuous at a rate of 2 in./min. The trapping system consisted of a series of traps at ambient temperature. The first trap was a 3-liter expansion flask. This was followed by three finger traps each containing 125 ml of a mixture of 50% benzene and 50% ethyl ether. A gas scrubbing trap, containing 125 ml of the same solvent mixture, completed the system. All solvents used in these experiments were redistilled in glass from reagent grades. The condensable pyrolyzate was washed from the traps and the cooled pyrolysis tube with alternate 100 ml portions of methanol and 50:50 benzene-ether until no additional colored material was observed to elute. The resulting solution was reduced in volume to 1 liter by rotary vacuum evaporation and divided for subsequent analyses.

Analyses of Pyrolyzate Components

The polynuclear aromatic hydrocarbons (PAH) in a portion (70%) of the pyrolyzate were analyzed according to our method (9) (Fig. 2). The pyrolyzate was partitioned between water and benzene:methanol:ethyl ether (2:1:2, v/v/v). The water-solubles were discarded and the primarily neutral materials, from the organic phase, were chromatographed on a silicic acid column and eluted with petroleum ether to yield the hydrocarbon waxes. A solvent mixture of benzene in petroleum ether (3:1) subsequently removed a fraction containing the PAH material. This PAH-containing material was

refined by gel filtration chromatography on Bio-Beads SX-12 to obtain two pure PAH-containing fractions, analyzable by GC. GC analyses were performed on a Hewlett-Packard 5830 gas chromatograph, using a 4.57 m $\times$ 0.32 cm stainless steel column containing 5% Dexsil 300 on 100/120 mesh Chromosorb W-AW. Oven temperature was programmed from 90–325 °C at 2°/min and the helium flow was 50 ml/min at 90 °C. Component peak areas were corrected for differences in detector response.

The levels of phenols, nicotine and free fatty acids were determined directly on the pyrolyzate by modifications of a reported method (10). An aliquot (10%) of the total pyrolyzate was evaporated to about 1 ml and then diluted with ethyl acetate to a final volume of 2.00 ml. A 50 μ l aliquot of this solution was treated with 50 μ l of BSA, containing 22.45 μ g of 1-hexacosanol. The resulting TMS derivatives, containing the free nicotine, were analyzed on a 3.05 m $\times$ 0.64 cm glass column of 6% OV-17 on Chromosorb G-HP. The helium flow was 30 ml/min and oven temperature was programmed from 80 to 280 °C at 2°/min. The upper temperature was maintained until all components of interest had eluted. The samples were analyzed on a Varian model 2700 gas chromatograph, coupled to an Autolab System IV integrator. Component peak areas were corrected for differences in detector response.

Gas Chromatography – Mass Spectrometry (GC-MS)

The identity of pyrolysis components in the pyrolyzate was confirmed by GC retention times and GC-MS with a Hewlett-Packard model 5710A gas chromatograph coupled via a Ryhage jet separator to a DuPont 21-492 mass spectrometer. GC conditions for the GC-MS analyses were identical with those employed for GC quantitation to obtain similar chromatograms.

Figure 2. Chromatographic and gel filtration separation procedure for polynuclear aromatic hydrocarbons (PAH) of pyrolyzates.

RESULTS AND DISCUSSION

The levels of tobacco lipids will be discussed first, followed by the analytical data on the phenols and polynuclear aromatic hydrocarbons (PAH) of the pyrolyzate. The tobacco extraction and lipid fractionation are detailed in Fig. 1. The quantitative data for all of the lipids are given in Table 1. The neophytadiene and the aliphatic hydrocarbon data were obtained from GC chromatograms similar to the chromatogram shown in Fig. 3. The levels of neophytadiene and total hydrocarbon waxes (C₂₅–C₃₄) were considerably lower for the close-grown tobaccos. The terpene and fatty alcohol data were obtained from GC chromatograms like that shown in Fig. 4. The close-grown varieties, Coker 139 and Speight G-28, contained higher levels of phytol than the reference NC 2326 tobacco. The levels of docosanol, β -amyrin and cycloartenol were substantially lower in the close-grown varieties. This indicated that lower levels of products, derived from the terpene fraction, should be expected in the pyrolyzate.

Figure 3. Gas chromatogram of leaf hydrocarbon - neophytadiene fraction.
(n: normal, i: iso, a: anteiso)

Table 1. Tobacco lipid levels: Absolute values and relative percentages (In parentheses).

	NC 2326	SC 1971	Coker 139	Speight G-28
Hydrocarbons (mg/g)				
Neophytadiene	1.74 (100)	1.34 (77)	0.06 (3)	0.07 (4)
Hydrocarbons C ₂₅ -C ₃₄	1.54 (100)	1.70 (110)	0.87 (57)	0.93 (60)
Terpenes and alcohols (μg/g)				
Phytol	131.0 (100)	162.0 (123)	149.0 (113)	192.0 (146)
Docosanol	99.3 (100)	123.0 (124)	62.1 (62)	61.0 (61)
β-Amyrin	231.0 (100)	242.0 (104)	47.0 (20)	57.0 (24)
Cycloartenol	239.0 (100)	277.0 (115)	82.7 (34)	111.5 (47)
Fatty acids (mg/g)				
C ₁₄	0.17 (100)	0.20 (117)	0.04 (23)	0.05 (29)
C ₁₆	1.96 (100)	3.12 (159)	1.34 (68)	1.74 (88)
C ₁₈ (with 1, 2 or 3 double bonds)	4.80 (100)	7.93 (165)	2.40 (50)	4.36 (90)
C ₁₈	0.62 (100)	0.97 (156)	0.46 (74)	0.54 (87)
Total	7.55 (100)	12.22 (157)	4.24 (54)	6.69 (86)
Solanesol and sterols (mg/g)				
Solanesol	19.90 (100)	22.40 (112)	0.88 (4.4)	1.17 (5.8)
Cholesterol	0.17 (100)	0.21 (123)	0.09 (52)	0.10 (58)
Campesterol	0.90 (100)	0.98 (108)	0.41 (45)	0.54 (50)
Stigmasterol				
Sitosterol				
Total Sterols	1.67 (100)	1.90 (113)	0.73 (43)	0.91 (54)

Figure 4. Gas chromatogram of terpenes - fatty alcohols fraction of tobacco leaf.

Figure 5. Gas chromatogram of fatty acids - sterols - solanesol fraction of tobacco.

The values for the major fatty acids, obtained from the tobacco hydrolyzates, were calculated from chromatograms similar to the example in Fig. 5. The aged South Carolina flue-cured tobacco sample gave very high values compared to the more recent NC 2326 variety, which gave higher levels of fatty acids than the close-grown varieties. The yield of NC 2326 total fatty acids was double those for the close-grown samples. Lower levels of pyrolysis products, derived from leaf fatty acids, would be expected for the close-grown products. The

analytical results for the C₄₅ terpenoid alcohol, solanesol, and for the major sterols of leaf were highest for the aged 1971 flue-cured tobacco. The reference sample, NC 2326, gave values which were almost double the values obtained for the close-grown materials. The solanesol content in leaf has been related to the PAH levels found in pyrolyzates (4).

The yields of condensable pyrolyzate and ash for the four samples are shown in Table 2. The data are averages of replicate analyses. The data for NC 2326 and the

Table 2. Sample and pyrolyzate yield data *.

	NC 2326	SC 1971	Coker 139	Speight G-28
Weight of sample pyrolyzed (g)	30.00	30.00	30.00	30.00
Dry weight (g)	26.73	26.84	27.53	27.52
% moisture	10.88	10.48	8.23	8.27
Weight of pyrolyzate (g)	4.62	5.50	4.54	4.63
Weight of ash (g)	10.59	9.97	10.41	10.47
% ash	35.31	33.24	34.70	34.91
% ash (dry weight)	39.62	37.11	37.81	38.05
% pyrolyzate	15.41	18.33	15.17	14.24
% pyrolyzate (dry weight)	17.30	20.45	16.55	16.28

* All data are averages of three analyses.

Table 3. Polycyclic aromatic hydrocarbon contents of pyrolyzate.

	NC 2326		SC 1971		Coker 139		Speight G-28	
	µg/g	rel. %	µg/g	rel. %	µg/g	rel. %	µg/g	rel. %
Fluoranthene	1.94	100	1.38	71	1.78	92	1.80	91
Methylfluoranthenes	3.99	100	3.41	85	3.87	99	3.58	90
Pyrene	2.21	100	2.02	91	2.02	91	2.04	92
Methylpyrenes	4.61	100	5.08	110	4.46	97	4.44	96
Benz[a]anthracene/chrysene/triphenylene	1.01	100	0.89	88	1.04	103	0.98	97
Benzofluoranthenes	0.73	100	0.59	81	0.60	82	0.72	99
Benzopyrenes	0.43	100	0.36	84	0.51	118	0.54	125

two close-grown tobaccos were relatively similar. The 1971 South Carolina flue-cured tobacco yielded relatively more pyrolyzate. However, the yields of pyrolyzate were much greater than the amounts of cigarette smoke condensate that would be obtained from the equivalent weights on cigarettes smoked on a modern smoking machine. This is due to the fact that the pyrolyzate represents the sidestream as well as the mainstream smoke.

From the tobacco lipid data one would have predicted that the pyrolyzates from these close-grown materials should have significantly lower levels of PAH materials and that the higher cellulose content of the close-grown tobaccos would lead to higher phenol levels (11). However, the observed PAH levels for the close-grown samples were higher than expected. This was mainly due to the non-uniformity of the close-grown tobacco samples and probably also due to some lipids or other PAH precursors which may have been present in the samples but were not determined. Carotenes are potent PAH precursors, but there is no GC method for them. Any solanesol or lipids that are tied up as phospholipids would also not have been determined. This problem still remains to be investigated. The values for selected PAH levels in the pyrolyzates are given in Table 3 and the analytical procedure is outlined in Fig. 2. The reference tobacco, NC 2326, gave only slightly higher values for most of the PAH compounds than the close-grown tobaccos, while the biologically significant benzopyrenes were somewhat higher in the close-grown samples. The

PAH data were obtained under conditions, developed in our laboratory, that produced pyrolytic PAH profiles similar to those found in cigarette smoke condensate (1). The good reproducibility of the PAH data for the NC 2326 and SC 1971 tobaccos (Table 4) proved conclusively that both the pyrolytic procedure and the complex PAH analytical method produced reliable results. For these leaf samples the analytical data for individual PAH varied only by about 3 %. In previous work on Kentucky Reference 1R1 cigarette tobacco (13), it was also conclusively shown that PAH profiles of cigarette smoke condensate were reproduced by 700 °C pyrolyzate profiles. However, the PAH data for the close-grown samples varied considerably from run to run. The standard deviations were large and the data were difficult to interpret. We attributed the lack of precision to the non-homogeneity of the close-grown samples. The leaf, stalk and flower components were initially separated and then recombined for pyrolysis; also, the stalks were more difficult to grind than tobacco leaves and did not produce uniform particles. The use of shredded leaf samples or shredded sheet, made from a whole plant, would give more meaningful results.

The values for phenolics, nicotine and free fatty acids determined for the whole pyrolyzate are given in Table 5 and a corresponding chromatogram is shown in Fig. 6. The 1971 South Carolina flue-cured tobacco gave similar or higher phenol values than the reference tobacco. Compared to NC 2326 tobacco, the Coker 139 and Speight G-28 samples gave significantly higher values

Table 4. Reproducibility of polycyclic aromatic hydrocarbon analyses on consecutive pyrolytic runs for flue-cured leaf samples.

	NC 2326				SC 1971		
	Run 1	Run 2	Run 3	Average	Run 1	Run 2	Average
	(µg/g pyrolyzed)						
Fluoranthene	1.94	1.90	1.98	1.94	1.44	1.31	1.38
Methylfluoranthenes	4.14	3.90	3.92	3.99	3.47	3.35	3.41
Pyrene	2.32	2.07	2.24	2.21	2.01	2.04	2.02
Methylpyrenes	4.68	4.39	4.77	4.61	5.02	5.15	5.08
Benz[a]anthracene/chrysene/triphenylene	1.11	0.97	0.94	1.01	0.87	0.91	0.89
Benzofluoranthenes	0.79	0.71	0.69	0.73	0.51	0.61	0.59
Benzopyrenes	0.40	0.43	0.45	0.43	0.36	0.37	0.36

Figure 6. Gas chromatogram of total pyrolyzate of Speight G-28 close-grown tobacco.
(Identities of numbered peaks given in Table 5; I.S. is internal standard.)

Table 5. Yields of selected pyrolyzate compounds.

GC peak (Fig. 6)	Compound	NC 2326		SC 1971		Coker 139		Speight G-28	
		mg/g	rel. %	mg/g	rel. %	mg/g	rel. %	mg/g	rel. %
1	Phenol	1.91	100	2.30	120	1.98	102	2.49	128
2	o-Cresol	1.42	100	1.40	98	1.31	91	1.88	128
3, 4	m-Cresol/p-cresol*		100		110		175		200
5	2,4-Xylenol	1.44	100	1.24	83	1.42	98	2.03	138
6	2,6-Xylenol/p-ethylphenol	0.98	100	0.97	99	0.79	81	1.20	123
7	Guaiacol		100		143		586		395
8	Catechol	1.18	100	2.40	203	2.98	243	2.87	243
9	Resorcinol/hydroquinone	1.05	100	5.35	467	3.28	315	3.62	345
10	Nicotine	20.80	100	24.20	116	6.40	30	9.03	39
11	Palmitic acid*		100		228		148		136
12	C ₁₈ acid*		100		307		178		209

* No relative response factors calculated.

for most phenolic material. The levels of guaiacol, catechol, and resorcinol/hydroquinone were much higher in the close-grown materials. This is a measure of the higher stem (cellulose, carbohydrate) contents of the close-grown samples (11). The production of phenols from cellulose and carbohydrates has been documented (12). The levels of pyrolyzate nicotine were substantially lower in the close-grown materials, about one-third of the reference tobacco. This is reasonable as stems contain less nicotine than lamina. The levels of free fatty acids were higher in the close-grown pyrolyzates, even though the values for the total leaf fatty acids were lower. The higher phenols and fatty acid levels in pyrolyzates of the close-grown tobaccos should have a deleterious effect on the flavor of cigarette smoke. The reduced level of nicotine may be a desirable feature of these close-grown tobaccos. Based on the levels of the specific pyrolysis compounds examined, the close-grown tobaccos do not appear to represent a less harmful smoking material than the reference NC 2326 tobacco, but may possibly be suitable for re-

constituted tobacco sheet formulations. The high phenol yields may be important in affecting smoke flavor, while the low nicotine contents may be desirable in formulating low-nicotine smoking products.

SUMMARY

A pyrolytic method for evaluating the smoke properties of tobacco materials for their potential to produce biologically significant smoke phenols and PAH has been applied to close-grown tobacco, in comparison to conventionally grown tobacco. Analyses were performed on the lipids of tobacco samples to attempt correlations with amounts of polynuclear aromatic hydrocarbons in pyrolyzates. Relative to a standard flue-cured tobacco, the close-grown tobacco samples contained lower nicotine, neophytadiene and hydrocarbon levels, and lower sterols, solanesol and fatty acid levels. Close-grown tobacco pyrolyzates contained equivalent PAH levels and higher

levels of phenols. Based on the pyrolysis data, the close-grown tobaccos do not offer a more desirable smoking product than conventionally grown flue-cured tobaccos. However, the lower levels of nicotine and higher amounts of smoke phenols may possibly make the close-grown materials suitable for inclusion into low-nicotine tobacco sheet products.

ZUSAMMENFASSUNG

Unter Anwendung eines Pyrolyseverfahrens, mit dem sich an Tabakproben prüfen lässt, inwieweit in deren Rauch biologisch wirksame Phenole und polycyclische aromatische Kohlenwasserstoffe (PAH) auftreten könnten, wurden Tabakpflanzen, die aus sehr eng gepflanzten Kulturen geerntet worden waren, untersucht und mit auf herkömmliche Weise gezogenem Tabak verglichen. Es wurde versucht, den in Tabakproben gemessenen Lipidgehalt mit der Menge der in den Pyrolyseprodukten enthaltenen polycyclischen aromatischen Kohlenwasserstoffen in Beziehung zu setzen. Im Vergleich zu herkömmlich gezogenem „flue-cured“-Tabak hatten die Tabakproben, die aus sehr eng gepflanzten Kulturen stammten, einen geringeren Gehalt an Nikotin, Neophytadien und Kohlenwasserstoffen sowie an Sterolen, Solanesol und Fettsäuren. Die Pyrolysate des mit engem Pflanzenabstand gezogenen Tabaks enthielten gleich viel polycyclische aromatische Kohlenwasserstoffe und vermehrt Phenole. Unter Zugrundelegung dieser Pyrolysedaten ergeben eng gepflanzte Tabake kein weniger schädliches Rauchprodukt als in herkömmlicher Art gezogene „flue-cured“-Tabake. Sie könnten sich jedoch durch ihren geringeren Nikotin- und ihren höheren Phenolgehalt im Rauch möglicherweise für die Herstellung von Folien mit niedrigem Nikotingehalt eignen.

RÉSUMÉ

Une méthode d'évaluation de matériel tabagique par pyrolyse — en particulier son potentiel de produire dans la fumée des phénols biologiquement actifs et des hydrocarbures aromatiques polycycliques — a été appliquée au tabac «close-grown» (planté à distance courte et récolté en plante entière), en comparaison avec un tabac cultivé conventionnellement. Des analyses ont été effectuées sur les lipides d'échantillons de tabac pour tenter de mettre en évidence des corrélations avec la concentration d'hydrocarbures aromatiques polynucléaires dans les pyrolysats. Les échantillons du tabac «close-grown» contenaient moins de nicotine, de néophytadiène, d'hydrocarbures, de stérols, de solanesol et d'acides gras que le tabac «flue-cured» normal. Dans les pyrolysats, le tabac «close-grown» contenait autant d'hydrocarbures aromatiques polynucléaires

et davantage de phénols que le tabac normal. Sur la base des données de la pyrolyse, les tabacs «close-grown» ne donnent pas un produit plus désirable que le tabac «flue-cured» cultivé de manière traditionnelle. Néanmoins, sa teneur en nicotine plus basse et les quantités de phénols plus élevées dans la fumée pourraient permettre l'incorporation des produits du tabac «close-grown» dans des tabacs reconstitués à basse teneur en nicotine.

REFERENCES

1. Higman, E. B., R. F. Severson, R. F. Arrendale and O. T. Chortyk: *J. Agr. Food Chem.* 25 (1977) 1201.
2. Zilkey, B. F.: *Proceedings, 3rd World Conf. Smoking and Health* (1975); U.S. Department of Health, Education and Welfare Publ. No. (NIH) 76-1221, p. 57.
3. Tso, T. C., G. B. Gori and D. Hoffmann: *Proceedings, 3rd World Conf. Smoking and Health* (1975); U.S. Department of Health, Education and Welfare Publ. No. (NIH) 76-1221, p. 35.
4. Schlotzhauer, W. S., R. F. Severson, O. T. Chortyk, R. F. Arrendale and H. C. Higman: *J. Agr. Food Chem.* 24 (1976) 992.
5. Appleton, R. A., and C. R. Enzell: *Phytochem.* 10 (1971) 447.
6. Kallianos, A. G.: *Rec. Adv. Tob. Sci.* 2 (1976) 61.
7. Hoffmann, D., I. Schmeltz, S. S. Hecht, E. L. Wynder: *Proceedings, 3rd World Conf. Smoking and Health* (1975); U.S. Department of Health, Education and Welfare Publ. No. (NIH) 76-1221, p. 125.
8. Severson, R. F., J. J. Ellington, R. F. Arrendale, M. E. Snook and O. T. Chortyk: *J. Chromatog.* 160 (1978) 155.
9. Severson, R. F., M. E. Snook, R. F. Arrendale and O. T. Chortyk: *Anal. Chem.* 48 (1976) 1866.
10. Guerin, M. R., and G. O. Olerich: *Tob. Sci.* 20 (1976) 17.
11. Schlotzhauer, W. S., O. T. Chortyk, H. C. Higman and I. Schmeltz: *Tob. Sci.* 11 (1967) 31.
12. Chortyk, O. T., and W. S. Schlotzhauer: *Beitr. Tabakforsch.* 7 (1973) 165.
13. Severson, R. F., W. S. Schlotzhauer, R. F. Arrendale, M. E. Snook and H. C. Higman: *Beitr. Tabakforsch.* 9 (1977) 23.

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