X-Ray Study of Cigarette Structure*

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INTRODUCTION

Tobacco and tobacco products have been the subject of scientific investigations for many decades (1,2,3,4,5,6) but it is safe to say that the intensity of effort in the last decade exceeds that in earlier times (7,8,9,10,11,12). This intensified activity has ranged from tar and nicotine analyses to complicated chemical, biological and clinical studies. However, relatively little has been published dealing with the physical or structural aspects of the cigarette, although these may be an important factor to the combustion process. Those few studies which have been published, deal mainly with the complicated measurements involving filling power or tobacco-packing densities rather than the structure of the cigarette itself (5,13,14).

During the past several years, studies at the AMF Technical Center have been focused upon the structural aspects of reconstituted sheets and their effects upon the resultant smoke chemistry and biology. Results of these studies, some of which were reported at the 5th International Tobacco Scientific Congress held in Hamburg, Germany, in 1970 (15), have revealed unique properties for low-density reconstituted sheet materials. Reduced tobacco density, obtained with reconstituted sheet technology, is an effective means of modifying the physical properties of cigarettes, the smoke yield and its composition (16). These results have served to intensify our interests in the structural characteristics of the cigarette itself.

This study was initiated to develop an objective method of analyzing the internal structure of a cigarette and to establish the nature of tobacco shred arrangements therein. A preliminary study suggested several promising methods, all providing some form of either visual or photographic presentation. The three reported here are:

1. macro-photography of the undisturbed contents of the cigarette after removal of the cigarette paper,
2. longitudinal serial cross-sections of the cigarette, and
3. x-radiography of the whole undisturbed cigarette.

EXPERIMENTAL METHODS

Macro-photographic Method: A technique is utilized which permits exposure of the tobacco cylinder and its shred configuration with minimal disturbance. Using a dropper, clear lacquer (Kodak film lacquer) was dispensed into an up-ended cigarette until it was permeated. It was then immediately placed horizontally onto a 1" wide strip of double-coated adhesive tape

and allowed to dry for no longer than 2 minutes. The paper was then slit with a razor from end to end and was carefully peeled away symmetrically on both sides of the inner cigarette cylinder which was weakly bonded by the moist lacquer. The paper was then pushed down onto the unused portion of the double-coated adhesive. In a few minutes, the samples became sufficiently self-supporting to withstand the handling required for study and photography.

Figure 1 illustrates the results obtained by this technique. The photograph, showing five commercial cigarettes (different manufacturers), does not appear to demonstrate any easily visible differences. Since it is probable that these five manufacturers are using different shred mixtures and different makers, it was decided to explore the differences, if any, between cigarettes made from a single shred mixture using different machines. These cigarettes are shown in Figure 2. Again, no discernible differences are apparent.

These studies indicate a random shred arrangement. Therefore, to determine the discrimination of the method, it was applied to cigarettes with known extremes in shred orientation. Because of ease of manipulation, reconstituted sheet tobacco was shredded yielding strands approximately 1/32 of an inch by about 2 feet. By careful alignment on an AMF Chico Maker, two extreme shred configurations were obtained: one was a straight, parallel alignment, the other, a long twisted alignment.

Figure 3 shows the results of these studies. Since it is possible to differentiate these arrangements, we believe it would not be difficult to distinguish systematic surface differences among the shred arrangements in commercially available cigarettes if such differences existed. Since this method, revealing only surface structure, failed to show differences in shred configuration, we probed deeper by cross-sectioning cigarettes.

Longitudinal Serial Cross-Sections: To make sections, the undisturbed whole cigarette was imbedded in a clear epoxy which had suitable cutting characteristics. The cigarette was totally immersed in a clear epoxy mixture (Stycast 1266, Emerson and Cuming) in a test-tube. After the air bubbles stopped rising, the tube and its contents were placed under mild vacuum and deaeration was carefully completed. The mixture was then poured, cigarette and all, into a rectangular mold and allowed to harden. Samples were then sectioned longitudinally using a sledge type microtome. Optimum thickness of the sections was found to be 150 microns (0.006 in.). These were dry-mounted between two glass slides, placed directly into a photo-enlarger and direct positive prints were made by projection.

* Presented at the CORESTA (General Assembly)/26th TCRC Joint Conference held in Williamsburg, Va., USA, in October 1972.

Figure 4 shows longitudinal sections of the central portion of five commercially available cigarettes (different manufacturers). One would be hard-pressed to conclude that real differences exist between these sections. Since each cigarette is depicted by only one central cross-section, serial cross-sections of one cigarette were examined to see if any structure could be observed. A photo of these sections, comprising a total thickness of 0.054 in., is shown in Figure 5. It is obvious that similar patterns are observable as one goes from one section to the next indicating structural consistency but it is difficult to visualize a three-dimensional structure. It is interesting to note that imperfections in rod formation appear to be visible. This will be further verified later.

Figure 6 is an enlargement by transmission of the inscribed area shown in Figure 5. The shred structure still seems random within the framework of rod variations. Extremes in structural differences between parallel and twisted shreds similar to those shown in the macro-photographic studies are shown in Figures 7 and 8. For these, the shred cigarettes were remade by careful hand rolling. The differences are less than expected.

Although these two photographic methods for describing cigarette structure have some merit, they were still not completely satisfactory. Interpretation of the data is difficult, even with the cross-sectioning method, and the preparative techniques are involved, time consuming and demanding.

X-Radiography: Because of the limitations imposed by the two described procedures, x-radiography was explored. Suitable commercial equipment is readily available at a reasonable cost. Using soft x-rays and a small focal spot, it is possible to obtain relatively high resolution radiographs of low-density materials such as tobacco. For these studies, a Philips Radifluor 120 was used and operated between 20 and 30 KV at 3 milliamperes. Good resolution is possible since the focal spot is small — about $\frac{1}{8}$ mm diameter. Finally, and most important, application of this technique would require no sample preparation and is non-destructive.

Again, the studies started with commercially available cigarettes. The figures presented are negatives where blackness denotes no absorption, i.e. the absence of matter. In Figure 9, x-radiographs of five commercially available cigarettes (different manufacturers) are presented. Note the sharp contrast and remember now that what you are viewing is a two-dimensional radiograph of the entire thickness of the cigarette, rather than a two-dimensional view of a cross-section. It should be thought of as a shadowgraph of all the tobacco in a cigarette. Again little apparent structural difference among the cigarettes is noted. However, it is interesting to point out that this appears to verify variations in rod formation discussed earlier. Because shreds and makers were probably different, the one shred — three maker experiment was repeated. Figure 10 shows radiographs of three cigarettes made with the same shred mixture on three different makers; and again, no

structural differences are discernible, but there appear to be differences in rod uniformity. Oriented shreds can be easily recognized by this technique as Figure 11 shows. Oriented (a and c) and twisted (b and d) shreds are shown in hand-made cigarettes. The very light lines in b and c were produced by inserting a soft-twisted (b) and straight (c) wire into the respective cigarettes.

Encouraged by these radiographs, but still feeling some limitations existed in the visualization of tobacco shred and configurations, stereo radiography was attempted. This could permit visual analysis of shred configuration throughout the entire thickness of the cigarette. The stereo radiographs were made by moving the subject $2\frac{1}{2}$ inches laterally in one plane, making separate exposures in each position. In order to assist the reader in viewing these stereo radiographs, stereo viewing glasses such as those made by the Abrams Instrument Co. of Lansing, Michigan, and available through most scientific supply houses, are recommended.

Figure 12 is a cigarette containing a twisted wire to assist visualization of the stereo x-radiograph. There is no doubt that it is more difficult to visualize the 3-D radiograph, both because of sharpness limitations in the x-ray image and because it represents a very unfamiliar object that the reader probably has never really seen.

Figure 13 is a stereo radiograph of five commercially available cigarettes (different manufacturers). The stereo technique adds a new dimension to the radiograph, permitting visual analysis of shred configuration throughout the thickness of the cigarette. However, one must conclude that there are no easily perceptible differences in shred configuration among these commercial samples, but once again it is felt that variations in rod formation are visible.

Figure 14 is a stereo radiograph of two cigarettes made with a single shred mixture on different makers.

DISCUSSION

In a paper by Halter et al. (17), investigations of the physical and combustion properties of low-density tobacco products were described. These investigations were stimulated by the multiplicity of new processes for producing tobacco products with reduced shred density, including freeze-dried tobacco and foamed reconstituted sheets. These cigarettes were all made on an AMF Chico Standard Model CCM, but the shreds themselves varied in density. All the experimental and control tobacco samples were prepared from a single lot of bright tobacco supplied by Dr. W. Johnson of North Carolina State University. The bulk of the control tobacco was conventionally cured, but a portion was submitted to a short-term heat-treatment following the yellowing phase of curing. Both the conventional and heat-treated, flue-cured tobacco types were converted by Dr. Johnson into freeze-dried samples. The conventional flue-cured control was also subsequently converted into foamed and non-foamed tobacco sheets using Microflake processes. In addition, the utility of low-density technology for possible tobacco substitutes was explored by making foamed and non-foamed reconstituted Microflake sheet

products from Eastman Kodak oxycellulose. Stereo radiographs of cigarettes made on the same machine but containing shred mixtures of varying densities and structure were prepared.

Figure 15 is a stereo radiograph of the natural-shred cigarette and the cigarette made with the same shred after it had been freeze-dried. Structural differences can be readily seen. Differences in optical density represent differences in density.

Figure 16 shows a stereo radiograph of the natural, the heat-treated, and the freeze-dried heat-treated shred cigarette. Again, the structural dissimilarity of the low-density materials can be easily seen.

Figure 17 shows three cigarettes prepared from one lot of tobacco: 1. from natural shreds, 2. from a flat-sheet reconstituted tobacco, and 3. from a reconstituted sheet made by a foam process.

Figure 18 is a stereo radiograph of cigarettes made with reconstituted oxycellulose produced by a conventional Microflake process and by a foam Microflake process.

SUMMARY

From an investigation of a variety of methods for visualization of the shred structure in a cigarette, it has been shown that two optical and one x-radiographic method produce useful information. It is concluded that the x-radiographic method, especially by the use of stereo views, provides the most information; it is a convenient and easily utilized method.

Examination of a limited number of commercial cigarettes did not show any recognizable structural differences beyond fluctuations in rod density. Novel structures utilizing low-density materials can be readily distinguished.

ZUSAMMENFASSUNG

Die Untersuchung mehrerer Verfahren, durch die die Tabakstruktur in einer Cigarette sichtbar gemacht werden kann, zeigte, daß zwei optische Methoden und eine röntgenographische Methode nützliche Informationen vermitteln. Die Autoren kommen zu dem Schluß, daß mit der Röntgenanalyse — einer bequemen und leicht durchführbaren Methode — besonders unter Verwendung von Stereoaufnahmen die besten Ergebnisse erzielt werden.

Abgesehen von Schwankungen in der Dichte des Tabakstranges konnten bei der Untersuchung einer begrenzten Anzahl handelsüblicher Cigaretten keinerlei Strukturunterschiede festgestellt werden. Ungewöhnliche Strukturen bei der Verwendung von Material geringerer Dichte können leicht festgestellt werden.

RESUME

Une étude de différentes méthodes de visualisation de la structure du tabac coupé dans une cigarette a montré que deux méthodes optiques et une méthode radiographique donnent des informations intéressantes. Il a été conclu que la méthode radiographique aux rayons X, spécialement dans les vues stéréo, donne le plus d'informations; c'est une méthode agréable et facile à utiliser.

L'examen d'un nombre limité de cigarettes commerciales n'a pas permis de mettre en évidence des différences de structure, mises à part les fluctuations de la densité du boudin de tabac. On peut distinguer aisément des structures nouvelles et extraordinaires se produisant par l'utilisation de matériaux de basse densité.

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