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To: .Roger Comes Date: June 4, 1987

From: .Vicki Baliga

Subject: .Status Report of Structural and Chemical Analyses of Low Sidestream Cigarette Papers

Low sidestream cigarette papers were examined using light microscopy, scanning electron microscopy, quantitative energy dispersive x-ray spectroscopy and atomic adsorption. Information obtained on the papers using the above techniques included the following:

1. Structural characteristics
   a. Paper cross sectional thickness
   b. Relative fiber structure
   c. Relative tightness of mesh

2. Quantitative determinations of Na, Mg, K, and Ca
   a. From bulk samples
   b. From both sides of the paper

3. Distribution of Mg, K, and Ca on both sides of the paper

These techniques were defined in a previous memo (1). A more complete report will follow.

Results:

Cross sectional thickness measurements correlated well with grams per unit area measurements. The heavier papers were thicker than the lighter weight papers with the heavier weight papers ranging in thickness from 43 u to 55 u. Both the lighter weight papers were 27-u thick (Table 1).

The relative fiber structure of the papers consisted of cylindrical and flattened fibers. These two fiber types were incorporated into the papers at different concentration ratios. The relative fiber structure correlated poorly with the grenier porosity value (Table 1).

Table 1. STRUCTURAL ANALYSIS OF LOW SIDESTREAM CIGARETTE PAPERS

	FIBER-FLAX STRUCTURE	WEAVE	PAPER THICKNESS MICRONS (STD)	WEIGHT G/M^2	GR. POR.	$Mg(OH)_2$ PERCENT	$CaCO_3$ PERCENT
P7BE	medium	tight	53.2 (5)	45	50	0	40
P4LP	course	tight	42.7 (4)	45	30	12	25
P6SW	FINE	tight	51.4 (5)	45	42	35	5
P6SU	course	medium	43.8 (5)	45	35	40	0
P6NG	course	LOOSE	27.2 (2)	25	17	0	25
P6DD	medium	tight	55.2 (4)	45	28	0	25
P7BF	course	LOOSE	27.0 (2)	25	32	0	27

Quantitative determinations for Na, Mg, K, and Ca were obtained from the bulk samples as well as from both sides of the paper. The concentrations of the above elements in the bulk samples (2) were relatively close to the expected values of those supplied by Ecusta and Kimberly Clark except for the Na values (Table 2).

Table 2. ANALYTICAL DATA ON BULK SAMPLE
PERCENT DRY WEIGHT

	P7BE	P4LP	P6SW	P6SU	P6NG	P6DD	P7BF
% NA	0.056	0.22	0.4	0.29	0.12	0.025	0.089
% MG	0.12	4.75	14.7	15.6	0.065	0.12	0.15
% K	ND	1.7	0.92	1.0	<0.25	<0.25	<0.25
% CA	19.2	13.1	2.08	1.2	12.6	15.2	12.4

Elemental determinations for Na, Mg, K, and Ca were obtained from both sides of the paper. Chemicals existing as a particulate form during the paper making process such as $CaCO_3$ and $Mg(OH)_2$ (3) were more concentrated on the outer side of the paper (Table 3).

Table 3. SUMMARY OF ELEMENTAL ANALYSES BY EDS - Na, Mg, K, AND Ca
PERCENT DRY WEIGHT

PAPER CODE	Na	Mg	K	Ca
	inner/outer	inner/outer	inner/outer	inner/outer
P7BE	0.1 / 0.0	0.1 / 0.2	0.0 / 0.0	13.3 / 22.5
P4LP	0.9 / 0.5	7.6 / 7.1	2.4 / 1.7	7.9 / 16.1
P6SW	1.0 / 1.2	15.4 / 19.2	0.1 / 1.4	1.5 / 3.1
P6SU	1.3 / 1.4	17.4 / 20.2	1.6 / 1.4	1.0 / 1.6
P6NG	0.2 / 0.3	0.1 / 0.1	0.2 / 0.0	9.9 / 21.9
P6DD	0.0 / 0.2	0.1 / 0.1	0.5 / 0.2	10.6 / 16.8
P7BF	0.3 / 0.2	0.2 / 0.3	0.1 / 0.1	9.7 / 16.2

The distributions of Mg, K, and Ca were examined to relate how the chemical components interact with the fiber matrix. $CaCO_3$ was distributed among the flax fibers in a particulate form. It did not coat the fibers (Photo 1). $Mg(OH)_2$ was also distributed among the flax fibers and it seemed to coat the fibers somewhat (Photo 2). The burn additive potassium acetate was evenly distributed on the fiber and nonfiber matrix. Potassium coated the fibers uniformly (Photo 3).

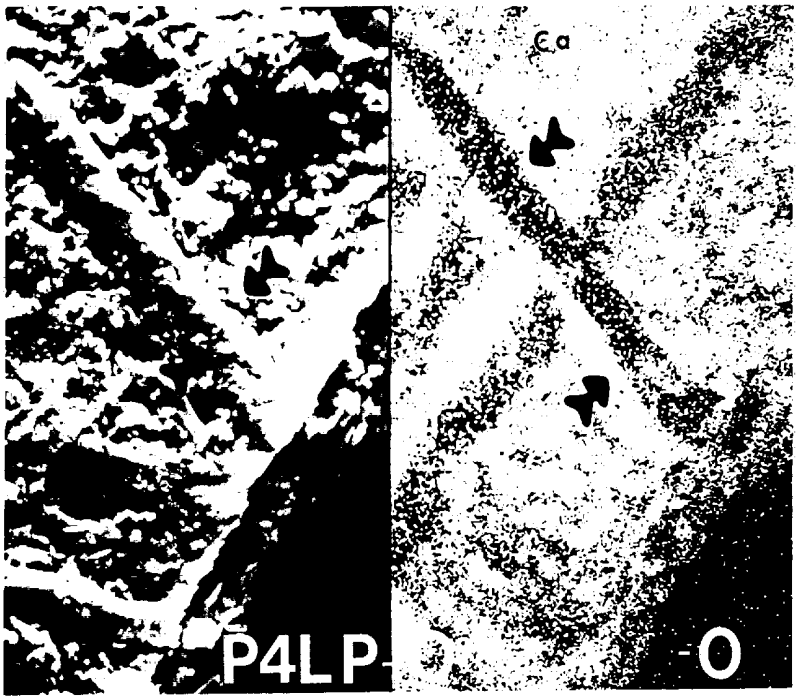

Photo 1
$CaC0_3$ distributed as particulates among fibers. X-ray dot map of **Ca**, Mag 500 X

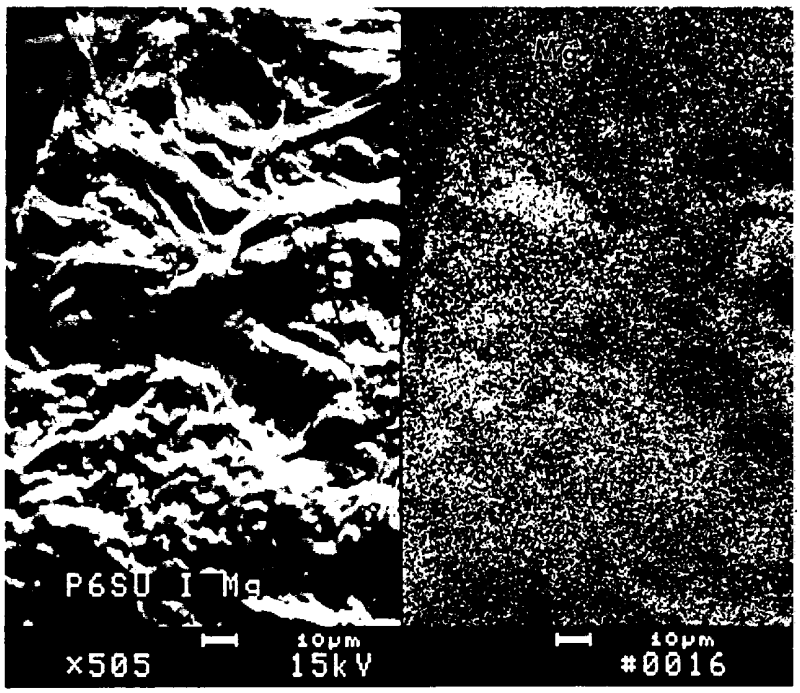

Photo 2
Mg of $Mg(OH)_2$ flax fibers with some coating effect. X-ray dot map of **Mg**, Mag 505 X

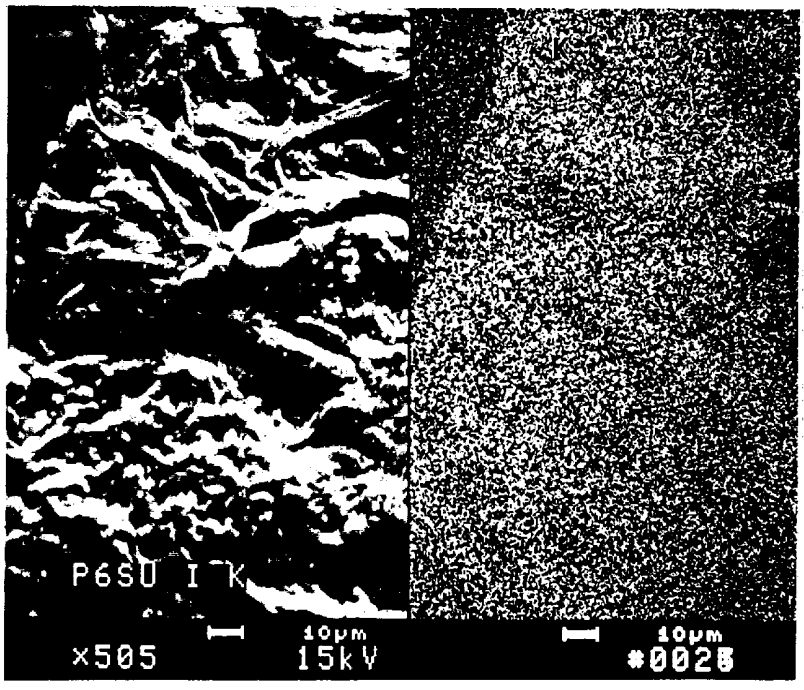

Photo 3
K evenly distributed throughout paper matrix, fibers coated. X-ray dot map of **K**, Mag. 505 X

Conclusions:

Structural and chemical data gained by microscopic analyses can help in the identification of changes in size and structure of fibers used in paper construction. Analytical microscopy can determine differences in quantities of chemicals left on the paper during the paper making process and determine how they interact with the fiber matrix of the paper.

Plans and Recommendations:

Plans for further microscopic examination include the following:

1. Complete the cross sectional examination of the papers and fibers to determine the interaction of Mg, K, and Ca with the outer and interior surfaces of the fibers.
2. From cigarettes made with $Mg(OH)_2$ papers, determine the structural changes of $Mg(OH)_2$ as a function of relative position to the char line of the burning cigarette.

Recommendations for correlative studies include the following:

1. Survey papers made with known particle sizes of $CaCO_3$ and / or $Mg(OH)_2$.
2. Examine papers made with known ratios of refined to nonrefined fibers.

References:

1. Baliga, Vicki. Structural and chemical analysis of cigarette papers. Memo to R. Comes; 1987 April 4.

2. Smith, Leon. Elemental analyses for Na, Mg, K, and Ca. General Analytical/LABSAM Report, Request Code # P87051; 1987 May 5.

3. Jarvis, Jerry. Ecusta, Pisgah Forest, North Carolina; personal communication.

4. Baliga, Vicki. Notebook # 8412; pp. 58 to 72a.

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