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RECONSTITUTED TOBACCO PRODUCT

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This invention relates to an improved tobacco product. More particularly, the invention relates to a desirable tobacco product, which product utilizes materials which were, prior to the present invention, found to be of little commercial value.

It has long been known that bright tobacco leaf, even without the addition of any flavourants, provides a good smoke when employed in tobacco products. It has a naturally sweet taste that is desirable in a smoking tobacco. It has been generally accepted as a good quality tobacco and, therefore, is in demand both for use alone and for use in tobacco blends.

More bright tobacco than any other is produced by tobacco growers in the United States and in many parts of the world. Therefore, bright tobacco stems and other portions of bright tobacco, such as bright tobacco scraps and dust, have constituted a major portion of the tobacco products which could not be completely effectively utilized by tobacco manufacturers. While there are methods known for preparing reconstituted tobacco from such bright tobacco stems, no such method has been found to be completely satisfactory, and there has been no known way of making bright tobacco stems, scraps and the like, into a product which has the same desirable smoking qualities as bright tobacco leaf.

The present invention provides a tobacco product which can have substantially the same desirable smoking qualities as bright tobacco leaf. The present invention also permits the manufacture of a tobacco product in such a way that many of the less desirable elements ordinarily delivered into tobacco smoke are decreased or completely eliminated. Thus, the invention involves a "tailor-made" smoking product that contains essentially only those ingredients which result in good burning characteristics and which produce the satisfaction expected from smoking the best grades of tobacco.

The invention contemplates preparing a tobacco product from bright tobacco stems, by treating the bright tobacco stems to remove the calcium and magnesium cross-links from the pectinaceous material contained therein, without deleteriously affecting the fibrous nature of the cellulose and hemi-cellulose in the tobacco being treated.

The invention involves producing from bright tobacco stems or other tobacco sources, a product substantially free of magnesium and calcium cross-links. Said cross-link free product is preferably pressed and ground, at which time it contains substantially all of the cellulose, hemi-cellulose and pectinaceous material which had been originally present in the stems or other tobacco parts being treated. As will be discussed more fully below, the resulting product or stem pulp is then utilized by forming a slurry of the same with water and introducing into the slurry, organic and inorganic constituents to incorporate the desired tobacco characteristics to the slurry. The resulting slurry is then formed by a conventional casting technique or by other techniques, into a reconstituted product which can be incorporated as the sole tobacco constituent or as a partial tobacco constituent, in a smoking product, for example, a cigarette, a cigar or a pipe tobacco.

One method for removing the calcium and magnesium cross-links from the pectinaceous materials found in the

tobacco is to wash the tobacco parts, which may be whole stems, shredded or chopped stems, tobacco fines or scraps, or the like, with an aqueous treating solution having a pH of from 0.5 to 3.0, and, preferably, from 1.0 to 2.5.

Preferably, the tobacco parts are first washed with water. To duplicate a bright tobacco product, the tobacco parts should, preferably, be bright tobacco parts. Other tobacco, however, such as burley tobacco, may be similarly treated and similarly employed.

The aqueous treating solution may be made suitably acidic by the addition of an inorganic acid, such as hydrochloric acid, to the water to bring the pH to the desired level. However, the water is preferably passed through an acidic-type ion exchange resin, such as an acid-form sulfate resin. If an acid form sulfate resin is used for such a purpose, it may be necessary to supply small amounts of a chloride, nitrate or similar ion, in order to provide an initial acidity to the water which can be maintained by the acidity of the resin. Other means for bringing the pH to the desired level include the use of any organic or inorganic acid with which a pH of 3 or lower can be obtained. These acids include sulfuric acid, nitric acid, orthophosphoric acid, pyrophosphoric acid and the like. The acid treatment is continued until the removal of the desired amount of calcium and/or magnesium has been accomplished. This may take from about 10 minutes to 50 hours, depending on the acidity of the solution, the temperature and mechanical capabilities of the apparatus being used. The acid water in the ion exchange column should generally contact the tobacco parts for a period of from about 1 hour to about 8 hours, and, preferably, from about 3 to 4 hours, at a temperature of from about 25° C. to 50° C., and, preferably, 25° C. to 35° C. The water after treating the tobacco can then be recycled to contact more tobacco.

The treated stems may then be pressed, or otherwise treated to remove the treating solution, air dried or dried in an oven or the like, and ground to substantially uniform size, generally from about —50 to —200 mesh in size and, preferably, from about —80 to —120 mesh in size, to form a pulp.

The resulting pulp may then be admixed with from 10 to 25 parts of water per part of pulp, at a temperature of about 0° C. to about 60° C., preferably from about 20° C. to about 40° C., and the resulting mixture brought to a pH of from about 5 to about 10, preferably from about 7.5 to about 8.6 by the addition of aqueous potassium hydroxide or the like. Then the following ingredients, which are essential to a good product, are added in the following amounts:

	Percent
(1) Calcium, added as a calcium salt, or as calcium hydroxide or the like, for example $Ca(OH)_2$, $CaCl_2$ or $CaHPO_4$, to give the following percentage of calcium (elemental) in the final product	2.0–3
(2) Potassium, added as a potassium salt, or as potassium hydroxide or the like, for example, KOH K_2SO_4, K_2HPO_4 or KCl, to give the following percentages of potassium (elemental) in the final product	0.19–5.0
(3) Malic acid or a salt thereof for example, calcium malate	5.0–20.0
(4) Citric acid or a salt thereof, for example, calcium citrate	0.75–10.0
(5) A humectant, for example, glycerine, triethylene glycol, butylene glycol or propylene glycol	1.0–6.0
(6) A sugar, for example, invert sugar, dextrose, fructose, or sucrose	5.0–20.0

Then, the following ingredients may, if desired, be added to give:

	Percent
(7) Magnesium, added as a magnesium salt, or as magnesium hydroxide or the like, for example, $Mg(OH)_2$, $MgSO_4$, or $MgCl_2$, to give the following percentage of magnesium (elemental)	0.3–1.4
(8) Nicotine, for example, free nicotine, or as a salt, such as the malate, citrate or hydrochloride	1.0–3.0
(9) A phosphate (P_2O_5), for example $CaHPO_4$ or K_2HPO_4	0.8–1.0

Other ingredients may then be added to modify the taste or smoking qualities of the product.

The choice of organic and inorganic constituents to be added to the slurry involves, basically, adding to the slurry the desirable constituents (or their equivalent) which are found in the naturally occuring tobacco leaf.

The following examples are illustrative:

Example 1

The constituents found by chemical analyses to be present in the filler of an all-bright no-additive cigarette were added to a base material. The base was 3.92 pounds of ground fiber which had been obtained from bright tobacco stems which had been decalcified by recycling slightly acidic waters over the washed bright tobacco stems and through a column of Dowex acid-form sulfate resin. The ground fibers were dispersed in 75.30 pounds of distilled water and the mixture was brought to a pH of 8.2 by the addition of 1.36 pounds of 17% aqueous potassium hydroxide. This mixture may be called the base mixture or Group I of the ingredients.

Malic acid (0.51 pound), calcium hydroxide (0.13 pound) and nicotine (0.75 pound of 20% aqueous solution) were added to 4.6 pounds of distilled water. This mixture, which may be referred to as Group II of the ingredients, was then poured into the base mixture (Group I).

The following groups of materials were also separately mixed and then added to the base mixture, in the order given below.

Group III:	Pounds
Magnesium sulfate heptahydrate	0.20
Potassium chloride	0.30
Potassium sulfate	0.02
Dibasic potassium phosphate	0.14
Citric acid monohydrate	0.07
Malic acid	0.06
Water	6.74
Group IV:	
Calcium chloride	0.14
Water	1.70
Group V:	
Nulomoline—manufactured by Nulomoline Division of Sucrest Corporation (85% invert sugar	1.25
Water	2.74
Group VI:	
Glycerin	0.14
Group VII:	
Ammonium hydroxide (concentrated)	0.14

This preparation, which made 100 pounds of slurry with a solids content of 7%, and a pH of 5.2, was passed through a homogenizer and then cast on an endless steel casting belt. The cast sheet was partially dried with electric heaters, removed from the belt when it had about a 20% moisture content, and dried further to about 15% moisture. The resulting sheet weighed about 8.8 g./sq. ft. It was shredded without difficulty into cigarette filler and cigarettes were made from it in a conventional manner. The sheet and the shredded filler handled very well during the various processing steps.

The resistance-to-draw (RTD) of the resulting cigarettes was determined. RTD is defined as the pressure drop across a cigarette, expressed in inches of water, when air flows through the cigarette at a velocity of 1050 ml./min. To determine this pressure difference, one end of the cigarette was inserted into a specially designed tube through which air was drawn. The pressure difference between the open and enclosed ends of the cigarette was measured. The RTD was 2–2.4 inches of water.

The cigarettes weighed 1.14 grams each. Ninety-nine cigarettes were made from four ounces of filler.

The cigarettes were smoked on a constant volume smoking machine which takes a 35 ml. puff of two seconds duration once in 60 seconds. The smoke was trapped on a Cambridge filter pad which retains particles larger than 0.3 micron. The filter assembly was weighed before and after smoking four cigarettes to determine the amount of total particulate matter (TPM) collected. The TPM was 10.2 mg./cigt.

The puff count was determined by smoking four cigarettes, counting the number of puffs it took for the char line to reach the 30 mm. mark on the cigarettes, and then by taking the advantage of the number of puffs as the count for the individual cigarettes. The puff count was 5.5.

Steam distillation of the filler from the cigarette removed the steam-volatile alkaloids present. Comparison of the ultraviolet absorption spectrum of the distillate with appropriate standards formed the basis for a quantitative estimate of the total alkaloid content. The nicotine, expressed as total alkaloid, was 0.84 mg./cigt.

The static burning rate, which was measured by determining the time required to burn 40 mm. of tobacco rod statically, was 6.6 minutes.

Example 2

The ingredients, as in Example 1, were combined in the same manner as in Example 1, except that 0.14 pound of citric acid monohydrate (twice the amount used in the formulated material described in Example 1) was used. The final slurry was adjusted to pH 5.2 by adding 17% aqueous potassium hydroxide as required. A sheet was cast, dried and shredded very successfully. Cigarettes prepared from this filler had much the same physical and burning characteristics as the material formulated in Example 1, as is shown in the accompanying table.

A panel of five expert smokers compared these cigarettes with the cigarettes prepared in Example 1. They found the test cigarette to be less harsh than the cigarette of Example 1, to have more total flavor, and to be preferred over the cigarettes of Example 1 by four of the panelists (one had no preference).

Example 3

Ingredients as specified for Example 1 were used in this Example, except that citric acid was omitted. The pH of the slurry was consequently high, 8.2. Procedures for homogenizing, casting, drying and shredding were unchanged from Example 1. Cigarette characteristics were similar to those of Example 1. Analysis of the sheet (see the tables which follow) showed absence of citric acid as well as a lower malic acid level. A panel of five expert smokers did not find these cigarettes significantly different from the cigarettes of Example 1.

Smoke from cigarettes of Examples 2 and 3 (citric acid augmented and omitted, respectively) was collected for analysis. After elimination of particulate matter, carbon dioxide and water, the remaining gases were analyzed by mass spectrometry. A significant difference was noted in the delivery of the cigarette prepared in Example 2 of 57% more acetonitrile, while the following components

were lower in Example 2 than Example 3 by the following differences:

	Percent
Methyl furan	18
Methyl chloride	21
Acetaldehyde	39
Acetylene	20

Example 4

Ground tobacco fibers (3.92 pounds) from bright tobacco stems were washed with water which was recycled through a column of Dowex acid-form sulfate resin and were dispersed in 75.92 pounds of distilled water. This mixture was brought to a pH of 8.2 adding 0.14 pound of concentrated aqueous ammonium hydroxide, 0.75 pound of 20% aqueous nicotine, and 0.20 pound of calcium hydroxide slurried in 1.00 pound of distilled water.

The following groups of ingredients were added to the resulting mixture in the sequence shown below (weights are in pounds):

Group I:	
Water (distilled in all cases)	1.80
Malic acid	0.44
Calcium hydroxide	0.18
Nicotine	0.75
Group II:	
Magnesium sulfate heptahydrate	0.25
Water	1.20
Group III:	
Dicalcium phosphate	0.11
Malic acid	0.13
Citric acid	0.07
Water	6.60
Group IV:	
Calcium chloride	0.16
Water	2.00
Group V:	
Nulomoline (85% invert sugar)	1.25
Water	3.05
Group VI:	
Calcium hydroxide	0.04
Water	0.25
Group VII:	
Glycerin	0.14
Water	0.05

The resulting slurry weighed 100 pounds and had a pH of 6.0. It was processed into sheet and shredded as before. The sheet had a high calcium content and very low potassium. Cigarettes failed to continue burning under static conditions, and this altered certain smoking characteristics as shown in the following tables (more puffs, higher total delivery).

Example 5

Ingredients and mode of combining them were the same as in Example 1, except that no invert surgar was added. Sheet was cast, dried and shredded as before. Low sugar meant a high ash content in the sheet, and the change in the sheet produced a light, fast-burning cigarette with consequent low puff count and low delivery of both nicotine and particulates.

The results of the various evaluations performed in Examples 1–5 are set forth below in Tables I and II.

TABLE I.—SHEET ANALYSIS

Ex. No.	Calcium, Percent	Potassium, Percent	Malic Acid, Percent	Citric Acid, Percent	Total Ash, Percent	Total Alkaloid, Percent	Reducing Sugar, Percent
1	2.05	4.33	11.74	1.00	15.28	2.14	11.63
2	2.30	5.05	9.87	1.84	17.34	2.27	11.94
3	1.95	3.44	4.13	<0.01	16.51	2.24	12.50
4	3.20	0.19	9.25	0.84	14.95	2.24	11.87
5	2.48	5.53	9.26	0.88	21.22	2.38	<1.7

TABLE II.—CIGARETTE AND SMOKE

Ex.	Cig. wt., g.	RTD,[1] in. of water	Puff Count, No. puff/cig. 30 mm. Butt	Static Burning, Min., 40 mm. Butt	Nicotine Delivery, 30 mm. Butt, mg./cig.	TPM[2] Delivery, 30 mm. Butt, mg./cig.
1	1.140	2.0–2.4	5.5	6.6	0.84	10.2
2	1.012	2.0–2.4	5.3	7.1	0.82	10.8
3	1.096	2.0–2.4	5.6	7.5	1.00	13.7
4	1.248	2.0–2.4	11.5	(3)	1.08	19.0
5	0.628	2.0–2.4	3.0	4.1	0.49	5.8

[1] Resistance-to-draw.
[2] Total particulate matter.
[3] Would not burn statically.

As used throughout this specification, all parts and percentages are by weight.

We claim:

1. A smoking tobacco product comprising from 5.0 to 90.0% by weight of a fibrous tobacco material containing tobacco pectins having substantially no calcium and magnesium cross-links; a calcium compound selected from the group consisting of calcium hydroxide, calcium chloride and calcium acid phosphate in an amount sufficient to provide from 2.0 to 3.0% by weight of elemental calcium; a potassium compound selected from the group consisting of potassium hydroxide, potassium sulfate, potassium acid phosphate and potassium chloride in an amount sufficient to provide from 0.19 to 5.0% by weight of elemental potassium; from 5.0 to 20.0% of malic acid or a salt thereof; from 0.75 to 10.0% of citric acid or a salt thereof; from 1.0 to 6.0% of a humectant and from 5.0 to 20.0% of a sugar.

2. The smoking tobacco product of claim 1 wherein the humectant is selected from a group consisting of glycerine, triethylene glycol, butylene glycol and propylene glycol.

3. A smoking tobacco product comprising from 5.0 to 90.0% by weight of the fibrous tobacco material containing tobacco pectins having substantially no calcium and magnesium cross-links; a calcium compound selected from the group consisting of calcium hydroxide, calcium chloride and calcium acid phosphate in sufficient amount to provide from 2.0 to 3.0% by weight of elemental calcium; a potassium compound selected from the group con-

sisting of potassium hydroxide, potassium sulfate, potassium acid phosphate and potassium chloride in sufficient amount to provide from 0.19 to 5.0% by weight of elemental potassium; from 5.0 to 20.0% of malic acid or a salt thereof; from 0.75 to 10.0% by weight of citric acid or a salt thereof; from 1.0 to 6.0% by weight of a humectant selected from the group consisting of glycerine, triethylene glycol, butylene glycol, propylene glycol; from 5.0 to 20.0% by weight of a sugar selected from the group consisting of invert sugar, dextrose, fructose or sucrose; a magnesium compound in an amount sufficient to provide from 0.3 to 1.4% by weight of elemental magnesium; from 1.0 to 3.0% by weight of free nicotine or of a nicotine salt, and from 0.8 to 1.0% of a phosphate.

4. A smoking tobacco product comprising from 5.0 to 90.0% by weight of a fibrous tobacco material containing tobacco pectins having substantially no calcium and magnesium cross-links, a calcium compound selected from the group consisting of calcium hydroxide, calcium chloride and calcium acid phosphate in an amount sufficient to provide from 2.0 to 3.0% by weight of elemental calcium; a potassium compound selected from the group consisting of potassium hydroxide, potassium sulfate, potassium acid phosphate and potassium chloride, in an amount sufficient to provide from 0.1 to 5.0% by weight of elemental potassium; from 5.0 to 20.0% by weight of malic acid or a salt thereof, from 0.75 to 10.0% by weight of citric acid or salt thereof, from 1.0 to 6.0% by weight of a humectant selected from the group consisting of glycerine, triethylene glycol, butylene glycol and propylene glycol, and from 5.0 to 20.0% by weight of a sugar selected from the group consisting of invert sugar, dextrose, fructose, and sucrose.

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