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[54] **RECONSTITUTED TOBACCO SHEET AND METHOD OF MAKING SAME**
2 Claims, No Drawings

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[56] **References Cited**
UNITED STATES PATENTS

802,487	10/1905	Wimmer	131/143
1,294,310	2/1919	Sayre et al.	131/143
2,293,954	8/1942	Tiger et al.	131/143UX
999,986	8/1911	Giordano	131/17
3,297,039	1/1967	Osborne et al.	131/140X
3,420,241	1/1969	Hind et al.	131/140

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ABSTRACT: Herein is disclosed a process for the treatment of tobacco in the manufacture of reconstituted tobacco products. In the method described a water-tobacco slurry is produced and thereafter at least a portion of the polyphenol constituent of the extract is precipitated or neutralized by treatment with sodium sulphate to the extent of one to five parts per hundred parts of tobacco, by weight.

RECONSTITUTED TOBACCO SHEET AND METHOD OF MAKING SAME

The present invention relates to a method for the preparation of reconstituted tobacco sheet and more particularly to an improved process for preparing light-colored reconstituted tobacco sheet, despite the use of heat as a processing aid at any stage of the process.

The commercial method for the manufacture of reconstituted tobacco sheet requires the application of heat to the material at some stage in the processing, *i.e.* either in the preparative stages and/or in the drying. It is known that the color of the finished tobacco product darkens because of the heating; the level of darkening being a function of temperature and heating time. As a rule, it is desirable to produce a light-colored tobacco sheet and various steps are taken to achieve this goal, for example the addition of dyes or coloring chemicals, the addition of color-masking agents "whiteners" such as titanium dioxide, control of temperature and heating time. These steps are never completely satisfactory because of the limitations under which they can be applied. In the case of the addition of dying, coloring and masking chemicals the introduction of extraneous material can be undesirable, especially if excessive amounts are needed to produce the desired results. Excessive use of dyes can produce "leaching" problems while excessive use of "whiteners" can result in sheets which are greyish in appearance or lack proper physical structure. In the case of controlled temperature and heating time, undesirable limitations are put upon the manufacturing procedures. The invention revealed herein makes it possible to produce a light-colored, reconstituted tobacco sheet without the resort to dyes or masking agents for color preservation and without any undue limitations on the temperature and heating time used in the process. This is achieved by selectively removing or "neutralizing" the specific agents responsible for the darkening of the tobacco on heating.

In the preparation of additive reconstituted tobacco, tobacco such as stems, field scrap cuttings, whole leaf and the like is preferably uniformly comminuted and formed into tobacco sheet suitable for later machine processing. On the other hand, in the preparation of all tobacco reconstituted sheet or in reconstituted sheets wherein a more efficient utilization of the natural gums of the tobacco is desired, the tobacco is usually ground to colloid-forming size particles by a wet process or refining. However, the large amount of energy and time required for the preparation of these colloid-forming size tobacco particles has limited its use for the preparation of reconstituted sheets.

To reduce the energy expended and time involved to comminute the tobacco particles in the wet process, it is desirable to cook the tobacco prior to refining. This cooking operation reduces the refining time and simultaneously improves the physical properties of the finished product. However, the cooking causes darkening of the tobacco material, thereby making such sheets unacceptable in a number of manufactured tobacco products.

We have now found what we believe to be the specific agents causing the darkening of the tobacco upon heating and propose as the prime object of this invention the control of color by selectively removing them or neutralizing them in situ.

The present invention also provides a solution to the long-standing problems in the processing of tobacco and results in a great reduction in energy and time required to obtain good quality reconstituted tobacco sheets of acceptable color.

A further object of the present invention is to provide a method for producing a light-colored reconstituted tobacco sheet despite the use of heat as a processing aid.

A still further object of the present invention is to remove polyphenols from the tobacco prior to exposure of the tobacco-water mixture to heat in the preparation of the reconstituted tobacco sheet without resort to dyes and color chemical additives to the tobacco.

A still further object of the present invention is to "neutralize" polyphenols in the tobacco prior to exposure of the tobacco-water mixture to heat in the preparation of reconstituted tobacco sheet.

An additional objective is to prepare a light-colored tobacco sheet without resort to dyes, coloring or masking agents.

Still another object of the present invention is to provide a method for accomplishing all of the above, which can be used in processing large quantities of tobacco resulting in a reconstituted sheet of tobacco having an acceptable color and to do so in an economical and easily controllable method.

Other objects and advantages of the present invention will be apparent from the further reading of the specification and of the appended claims.

Tobacco as defined for this invention includes any type of tobacco suitable for the manufacture of reconstituted tobacco sheet, such as stems, veins, scrap and waste tobacco, cuttings and the like, as well as whole leaf or portions thereof.

In general the present invention discloses chemical methods for precipitating or "neutralizing" the agents responsible for the darkening of the tobacco when it is exposed to heat.

It has long been commonly believed that the agents responsible for the darkening of the tobacco on heating were the carbohydrates and the amino acids, which react during the heating to form dark-colored compounds, the process being called the "Maillard" reaction. We have discovered that neither the carbohydrates nor the amino acids darken the tobacco significantly on heating, but that polyphenols contained in the tobacco, especially the basic-polyphenols, do cause a significant darkening of the tobacco upon exposure to heating. This phenomenon is also pH dependent.

A series of tests were run to isolate and determine the agents which caused tobacco darkening upon heating. The following procedure illustrates how the polyphenols were identified as the agents which cause tobacco darkening during heating. A sample of "Bright" stem dust weighing 10 gm. was mixed with 90 ml. of distilled water. The slurry was filtered on a Buchner funnel using Whatman No. 1 filter paper. The filter cake and filtrate were retained. The filter cake was subsequently mixed with 90 ml. of a predetermined liquid and the slurry transferred to a vessel, such as a mason jar, which was sealed and placed in a pressure cooker, where it was heated at 250°F. at 15 p.s.i. for 2 hours. Upon cooling, three sheets having a thickness of about 0.25—0.30 mm. were cast from the slurry on 20 cm. × 20 cm. glass plates. The sheets were dried in an oven at 110—115°C. for about 15— 20 minutes and the color of each sheet was determined in a Gardner automatic color difference meter (Model Ac-2A, Series 200). Readings were made by placing the plate over the Gardner aperture with the tobacco side facing the aperture. A number of readings were taken at different points on each plate, so as to obtain an overall average.

The test results in Table 1 were obtained with various additives in the predetermined liquid and serve to illustrate that polyphenols are the agents causing the darkening of the tobacco on exposure to heat. Water-washed Bright stem dust was used in all cases, unless otherwise indicated. The weight of the washed tobacco in each sample was 7.0 gm. dry weight and this was suspended in 90 ml. of liquid prior to heating.

The polyphenol fractions shown in Table 1 were obtained as follows:

90 ml. of a tobacco-water extract, prepared as described above, was mixed with 20 ml. of a 10 percent lead acetate solution, and the precipitate was removed by centrifugation. This precipitate comprised the acidic polyphenol fraction.

Adding more lead acetate solution to the supernatant caused no more precipitation. An alkaline reagent, such as ammonium hydroxide (NH_4OH was added to the supernatant until the pH was about 8.5—9.0. The precipitate, which was collected by centrifugation, constituted the basic-polyphenol fraction.

TABLE 1.—COLOR OF SHEETS PREPARED FROM BRIGHT STEM DUST HEATED IN SOLVENTS CONTAINING VARIOUS ADDITIVES

Materials	pH before heating	pH after heating	Rd	a+	b+
Washed tobacco plus water (positive control)	5.3	.....	24.9	3.1	21.5
Unwashed tobacco plus water	.....	3.8	16.3	.....	.....
Washed tobacco plus washings (negative control)	.....	4.0	16.4	.....	.....
Washed tobacco plus sucrose (2 gm.) plus water	.....	.....	25.4	.....	.....
Washed tobacco plus glucose (2 gm.) plus water	.....	.....	25.2	.....	.....
Washed tobacco plus fructose (2 gm.) plus water	.....	.....	24.7	.....	.....
Washed tobacco plus sucrose, fructose, glucose (2 gm. each) plus water	7.2	.....	26.8	.....	.....
Washed tobacco plus tryptone (1 gm.) (casein digest) plus water	6.3	.....	22.3	2.7	16.3
Washed tobacco plus tryptone plus sucrose (1 gm. each) plus water	6.1	.....	22.9	2.5	17.3
Washed tobacco plus tryptone plus glucose (1 gm. each) plus water	6.1	.....	22.2	3.3	17.5
Washed tobacco plus tryptone plus fructose (a gm. each) plus water	6.1	.....	22.0	2.5	17.1
Washed tobacco plus tryptone plus glucose, fructose sucrose (1 gm. each) plus water	6.2	.....	21.9	3.8	18.2
Washed tobacco plus water plus acidic polyphenols	5.2	4.5	22.6	3.5	16.3
Basic polyphenols plus washed tobacco plus water	9.3	6.3	12.4	5.8	15.0
Acidified basic polyphenols plus washed tobacco plus water	5.7	4.8	17.2	4.7	16.1
Washed tobacco plus P_b acetate sol. (10%) water	.....	.....	23.4	.....	.....

NOTE:
Rd=Brightness factor; black=0; white=100.
a+; b+=Coordidnates on a Chromaticity D_b agram.
P_b Acetate=Lead acetate.

As seen from the experimental results shown in Table 1, the basic polyphenols give a darker sheet than even the negative control (Rd readings 12.4 and 16.4, respectively). Furthermore, it is seen that while acidifying the basic-polyphenols improves the color, it is still far below the positive control (Rd readings of 17.2 and 24.9, respectively). These results also show that the sugars and amino acids do not affect the sheet color and similarly neither does the lead acetate solution used to precipitate the polyphenols.

Processes for the removal of the polyphenol fraction from tobacco by using adsorbent material are fully disclosed and described in our copending U.S. Pat. application Ser. No.639,032 filed even date herewith. The present disclosure confines itself to the precipitation and "neutralization" of polyphenols with chemical substances.

We have found that the interfering polyphenols can be inactivated "in situ" by sodium sulfite or removed from a water extract of tobacco by precipitation with chemical substances of the heavy metal salts such as lead acetate or barium acetate. While the above mentioned procedures (inactivation "in situ" or precipitation and removal) are entirely unacceptable in the manufacture of a 100 percent tobacco sheet (since they impart residuals to the tobacco materials) they are however, eminently suitable for the manufacture of non-100 percent tobacco products.

The following examples are set forth to demonstrate the method of this invention.

Ten grams of "Bright" stem dust was mixed with 90 ml. of a water solution containing various amounts of sodium sulfite. The mixtures were transferred to mason jars and treated as described in the early section of this disclosure. The obtained sheets were examined in the Gardner difference colorimeter in a manner described in an earlier part of this disclosure. The tobacco slurry compositions and the color readings obtained on the prepared sheets are shown in Table II.

TABLE II

Sample Number	Tobacco weight (gm.)	Sodium sulfite (gm.)	Water (ml.)	Rd	a+	b+
1	10.0	0.13	90.0	15.5	6.1	19.5
2	10.0	0.26	90.0	17.7	5.8	20.8
3	10.0	0.50	90.0	22.6	5.9	24.2
4 (negative control)	10.0	None	90.0	14.4	5.8	19.0
5 (positive control	(1)	None	90.0	23.6	3.5	22.3

[1] Water washed tobacco 10 gm.

It becomes apparent from the results in Table II that the effects of the polyphenols are "neutralized" by the addition of sodium sulfite to the tobacco-water slurry.

In the examples to follow, evidence is presented to show the effects of removing the basic polyphenols. Ten grams of Bright stem dust was mixed with 90 ml. of water and filtered in a Buchner funnel. The filtrate was mixed with 2 ml. of a saturated lead acetate solution, a precipitate formed. This precipitate, which comprised the acidic polyphenols, was removed by centrifugation. The supernatant was brought to pH 9.0 by the addition of NH_4OH. The precipitate, which comprised the basic polyphenols, was removed by centrifugation and the supernatant was brought to an acidic pH (4.0—5.0) by the addition of an acid such as acetic, HCl, etc. Slurries were prepared from water washed tobacco and the various polyphenolic fractions or supernatants. The slurries were treated in a manner already described. The prepared sheets were examined for their color and the results as well as the slurry compositions are presented in Table III.

TABLE III

Sample No.	Water washed tobacco (gm.)	Additive	Water (ml.)	Rd	a+	b+
1	10.0	Acidic polyphenol [1]	90.0	23.8	2.3	19.9
2	10.0	Acidified supernatant [2]		18.5	6.5	21.1
3	10.0	Acidified supernatant [2]		17.6	5.5	21.8
4	10.0	Basic polyphenol [1]	90.0	14.8	6.8	20.2
5	10.0	Basic polyphenol plus sodium sulfite (0.4 gm.).	90.0	29.4	2.0	24.7

[1] The polyphenol precipitates were obtained in a manner described in this disclosure.
[2] Volume of acidified supernatant was 90 ml.

It is apparent from the FIGS. in Table III that the acidic polyphenols do not cause any darkening of the tobacco on heating. The use of acidified supernatant from which the basic polyphenols have been removed gives a sheet much lighter than the negative control (Rd of 18.5 and 14.4 respectively). The basic polyphenolic fraction gives a sheet as dark as the negative control (Rd. 14.8 and 14.4 respectively). Very good results are achieved by "neutralizing" the basic polyphenols with sodium sulfite (Sample 5). In this case a sheet is obtained that is actually even lighter than the positive control. (Rd 29.4 and 23.6 respectively). It has therefore been demonstrated that the darkening of tobacco on heating is caused by basic polyphenols. This darkening can be avoided by adding to the tobacco a reagent, such as sodium sulfite, which "neutralizes" the effects of the darkening agents. The darkening can also be avoided by precipitating the basic polyphenols chemically and removing the precipitate.

We claim:

1. In the manufacture of reconstituted tobacco products, the process of preparing tobacco comprising the steps of treating such tobacco with water so as to extract the water-soluble components of said tobacco including the polyphenols and form an aqueous extract solution thereof, separating said

aqueous extract solution containing said polyphenols from the extracted tobacco, neutralizing the action of at least a portion of said polyphenols in said extract solution by the addition of a quantity of sodium sulfite, said sodium sulfite being employed in a range of from 1 to 5 parts per 100 parts of tobacco by weight, combining said extract solution from which polyphenols have been neutralized with said extracted tobacco so as to form a water-tobacco mixture including the extracted water soluble components of the original tobacco, except for the polyphenols neutralized therefrom.

2. A reconstituted tobacco product made by forming and subsequently drying a slurry of tobacco particles in which the tobacco is prepared in accordance with claim 1.