

[54] METHOD OF MAKING EXPANDED
RECONSTITUTED TOBACCO

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[22] Filed: Jan. 17, 1972

[21] Appl. No.: 218,488

[52] U.S. Cl. 131/140 P, 131/17

[51] Int. Cl. A24b 3/18

[58] Field of Search 131/15, 17, 140-144

[56] References Cited

UNITED STATES PATENTS

3,364,935 1/1968 Moshy et al. 131/140
3,528,434 9/1970 Halter et al. 131/140

FOREIGN PATENTS OR APPLICATIONS

955,679 4/1964 Great Britain 131/140 P

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[57] ABSTRACT

An expanded reconstituted tobacco product is produced by forming a slurry of tobacco plant parts and a binder of tobacco derived pectins in an aqueous medium into which a relatively small amount of low boiling alkanes or volatile aliphatic hydrocarbons are dispersed. An emulsifying agent may be added to obtain a better dispersion. The mixture is cast into sheet or web form and is then rapidly heated to a temperature sufficient to volatilize out substantially all of the hydrocarbons and water thereby producing a porous spongy tobacco product of increased thickness and lower density than an untreated reconstituted tobacco.

8 Claims, No Drawings

METHOD OF MAKING EXPANDED RECONSTITUTED TOBACCO

BACKGROUND OF THE INVENTION

Conventional methods of making reconstituted tobacco products involve the casting, spraying, roll-coating or the like of a slurry of finely-divided tobacco to form a tobacco sheet which is dried and then processed by methods normally used in preparing filler from reconstituted tobacco or regular tobacco leaf filler. However, the reconstituted tobacco sheet during the drying process becomes fairly dense and moreover quite flat in comparison to the typical irregular shapes of natural leaf. When such a sheet is shredded to form filler for smoking articles, the shreds are essentially flat and these tend to pack down and result in a dense filler. Thus the reconstituted sheet is said to have low filling power in that it does not possess the bulk to produce a satisfactory cigarette rod. The filling power is the ability of filler to produce a light but firm rod, and if this can be obtained with a low density product, filling power is increased. It should be pointed out that a dense filler is not only uneconomical, but it will deliver more total particulate matter (TPM) to the user.

Recognizing the problems of the usual reconstituted tobacco sheet, it was later suggested that such a sheet be prepared with a foamed structure to give it bulk and thus greater filling power. Various patents of Moshy et al., notably U.S. Pat. Nos. 3,364,935; 3,404,690; 3,404,691; and 3,410,279 describe processes in which a hydrophilic adhesive gum, capable of acting as a foaming agent, is added to a tobacco slurry. As described in U.S. Pat. No. 3,364,935, a mixture of tobacco and such hydrophilic adhesive gum foaming agent together with a foam stabilizer is thoroughly beaten into an aerated foamed mass. The foam stabilizer, of the class of surfactants and detergents, is apparently needed to prevent collapse of the foam with loss of air. The stabilized foamed tobacco mixture is then dried to obtain a shaped product of desired bulk and porosity. Should the foaming agents lack the necessary foaming effect, the patent also suggests the use of a blowing agent such as sodium bicarbonate to aid in producing the proper degree of foaming.

The difficulty with such processes is that they require the formulation of a stable foamed slurry which is generally difficult to keep intact before the expansion step without a foam stabilizer. Additionally, the presence of gum adhesive foaming agents and foaming stabilizers in the final product could easily cause the foamed tobacco sheet to possess foreign flavor notes of questionable value due to the retention of such gums as well as the foam stabilizers remaining in the final product. Residual alkali material left by the "blowing" agent could also cause undesirable effects.

A patent to Fredrickson, U.S. Pat. No. 3,524,451 describes a method of increasing filling capacity of tobacco or tobacco parts by soaking such material with an inert volatilizable organic foaming agent such as hydrocarbons or halogenated hydrocarbons to fully impregnate the tobacco. The amounts suggested by weight ratio of liquid to tobacco may be between 1-to-3 and 3-to-1. The impregnated tobacco with foaming agent is then contacted with a stream of hot gases at temperatures well above the boiling point of the organic liquid to as high as 400°F. Such a procedure presumably requires a material that stays intact when the

hot gas strikes the impregnated tobacco materials and intimately commingles therewith. Additionally, an intact cellular structure capable of absorbing and holding impregnating liquids within the tobacco cells for subsequent expansion appears to be a primary requirement. It would be expected that this procedure cannot be utilized successfully with finely divided tobacco plant parts which usually make up a reconstituted tobacco product for the reason that such finely divided materials have little of the typical intact cellular structure of tobacco. Without such cellular structure there is nothing available for cellular expansion by absorption of an organic liquid and later heating. An additional difficulty for adapting that process to reconstituted material is the fact that the impact of a hot gas stream on a comminuted reconstituted tobacco containing volatile hydrocarbons could easily reduce such a material back to its original constituents, namely, tobacco fines and dust.

A patent to Halter et al., U.S. Pat. No. 3,528,434, describes a process whereby a water-soluble thermogelling gum is added to an aqueous tobacco slurry containing dissolved air. The gum acts as an adhesive and binding agent. On heating the mixture, the thermosetting gums are stated by the patentee to gel before the dissolved air escapes to form air channels in the tobacco sheet.

It must be assumed that this process seeks to produce a porous tobacco sheet which differs from a foamed sheet produced by the escape of foaming agents. Additionally, the use and eventual retention of the thermogelling gums could easily contribute a foreign note to the smoke since it is a component part of the final product. Such gums being foreign elements and even if innocuous would be expected to reduce tobacco flavor and impact when such a product is smoked.

An object of the present invention is to provide a simplified and inexpensive process for increasing the filling capacity of reconstituted tobacco.

Another object of the present invention is to provide a process for producing an expanded, spongy reconstituted tobacco product free of non-tobacco substances producing undesired foreign notes in the tobacco.

These and other objects and advantages will become apparent from the description of the invention which follows.

SUMMARY OF THE INVENTION

The present invention relates to a spongy expanded reconstituted tobacco product and a process for producing it, in which an aqueous slurry of finely divided tobacco plant particles and a pectin binding agent derived from tobacco is commingled with a relatively low boiling volatilizable aliphatic hydrocarbon or mixture of hydrocarbons. A small amount of special emulsifier may be added to aid the dispersion. The mixture is then cast or extruded into web or sheet form and then heated to remove both water and hydrocarbons. The volatilization causes expansion of the spaces between the tobacco particles. The final dried product is a spongy tobacco material of high porosity, with substantial filling power, and may be prepared so that it contains no more than small amounts if any of non-tobacco elements, and free of residual foaming agents, foam stabilizers, non-tobacco celluloses, gums or proteins.

DESCRIPTION OF THE INVENTION

In accordance with the invention, the starting material is what is usually waste material in the form of tobacco shreds, scraps, dust and fines. The tobacco parts are slurried in an aqueous medium and then preferably refined to small particle sizes usually obtained in preparing a reconstituted tobacco. To this slurry is added a tobacco-derived binding agent either from extraneous sources or one preferably developed in situ by any of the procedures as taught by such patents as Hind et al., U.S. Pat. No. 3,353,541 or in U.S. Pat. No. 3,420,421. These patents teach the treatment of tobacco parts with specific reactants under conditions capable of releasing tobacco pectins. The latter act as excellent binding agents when combined with tobacco fines in the process of forming a reconstituted tobacco sheet or product. The following explanation will illustrate the procedure for obtaining a suitable binding agent derived from tobacco which will bond and hold tobacco fines into a coherent mass that can then be expanded by the process of this invention to provide a spongy, porous web or sheet of reconstituted tobacco.

It is known that pectin substances are present in tobacco in the form of protopectins, comprising calcium and magnesium salts of partially esterified and acetylated polymers of galacturonic acid. On treatment of the tobacco with a non-toxic reagent such as an alkali metal carbonate, or with selected chelating agents such as ethylenediamine tetraacetic acid, or with an acid such as hydrochloric or phosphoric acid, the protopectin cross-linking chains are broken and tobacco pectins are released.

A particularly advantageous reactant, combining the chain-destroying action with chelating action for removal of the alkaline earth metal cross links is diammonium hydrogen phosphate (DAP). This particular pectin releasing agent is preferred since it is not only effective in relatively small amounts but more importantly does not effect the final product in any noticeable way if the products of the pectin releasing action are retained therein. When using other tobacco pectin releasing agents, the liberated pectins may be separated from the treating solution and then added to tobacco fines as a binding agent. Alternatively, where the precipitating agent or reaction products do not affect the final product to an appreciable extent, the tobacco pectins may be released and deposited with the treated tobacco in situ without separation from the treating solution. Such procedures are fully described in both of the Hind et al patents identified previously and in both patents the advantages of using a tobacco binding agent composed of tobacco pectins is fully described.

Where an in situ reaction is carried out leading to the release of tobacco binding agents, the operation is generally carried out at relatively high temperatures. Consequently, before the addition of low boiling hydrocarbons, the next and important step is to cool the slurry of tobacco dust and binding agent to lower temperatures, namely, below 100°C. and preferably in the range of about 20° to 80°C., and more preferably in the range of about 25° to 40°C. It should be understood that whether the tobacco binding agent is prepared in situ, or whether it is obtained from other sources and added to the tobacco dust slurry, the temperature of the mixture should for good results, be adjusted to encompass the range indicated above. Experimental evi-

dence has shown that a substantially thicker and more expanded product could be made if the temperature of the slurry is held to about normal room temperature levels throughout the further additions and up to the time the tobacco slurry reaches the heating and drying stage.

To the cooled aqueous slurry of tobacco and binding agent is then added a low boiling, hydrophobic, aliphatic hydrocarbon of about five to eight carbon atoms as a foaming agent. Any straight or branched-chain hydrocarbon may be selected that falls within the boiling range of about 35° to 125°C. Such an agent may easily be removed by evaporation under relatively non-dramatic heating or drying conditions as such or in the form of a water azeotrope. Preferred hydrocarbons are the pentanes, hexanes, and heptanes or mixtures thereof, such as petroleum ether or the like. The amount of hydrocarbon added to the aqueous tobacco slurry should be kept to a minimum and used only in sufficient amount to expand the tobacco without leaving a residue. This may range from about 0.007 to no more than about 7.0 parts per 100 parts by weight of the tobacco fines. Less than this amount results in poor expansion and more than this amount tends to result in some residual material remaining in the tobacco sheet or will result in a weak and lacy tobacco sheet or such fragility as to reduce the sheet to tobacco fines. A preferred range of aliphatic hydrocarbon is from about 0.07 to about 1.5 parts of hydrocarbon per 100 parts by weight of the tobacco.

The hydrocarbons that are added to the aqueous slurry are intimately dispersed throughout the medium preferably by a high speed mixer or blender device. If relatively large particles are present the slurry may be further beaten or refined. To aid in obtaining a good dispersion or emulsion of the hydrophobic agent with the aqueous medium, a small amount of a special emulsifier may be added to the slurry of tobacco fines and hydrocarbon in an amount of about 5 percent by weight or less, and preferably from about 0.5 to 2.5 percent by weight based on the tobacco content in the slurry.

In the use of an emulsifying agent in the exercise of this invention, the emulsifying agent must be selected with care, not only for its effectiveness in dispersing the hydrophobic agent in the aqueous medium and effecting an emulsified mixture, but particularly with respect to its residual contribution to the final reconstituted tobacco product. Since the emulsifier is generally too high-boiling to volatilize out when the hydrocarbon component is removed by low temperature heating, it remains as a residual component that could, if not carefully selected as to type and amount, affect the smoking characteristics of the final foamed product by a possibly unpleasant aroma or by effecting an unpleasant "mouth coating." This expression is used in the tobacco art to refer to an orally unpleasant sensation that something is present on the inside of the smoker's mouth.

It is therefore critical with respect to the present invention, that when an emulsifier is used it should be selected to produce a well emulsified mixture so efficiently that very small amounts are necessary. Preferably the emulsifier will also impart either a desirable and delicate flavor to the tobacco, or to improve and enhance the flavor and aroma of the tobacco.

Thus, in accordance with the emulsification aspect of the invention, one emulsifier has been found that also imparts an intriguing flavor to tobacco smoke. Such an emulsifier is licorice or salts of this material, such as ammonium glycyrrhizinate. Another type of emulsifier that also adds to the tobacco flavor is a mixture of resins obtained from tobacco itself.

Licorice or glycyrrhizin salts, such as ammoniated glycyrrhizin are commercially available. Tobacco resins are obtained by known procedures, usually by extraction of tobacco with organic solvents such as hexane. These resins contain fatty acids, esters and salts that not only act as effective emulsifiers but may be added in an amount to result in enhancement of the tobacco flavor and aroma.

These flavoring emulsifying agents are effective emulsifiers in an amount of about 1 to 5 percent by weight of the tobacco slurry and generally in an amount of less than 3 percent by weight. Actually, it has been found that these agents produce effective results when used in an amount of as little as about 0.25 to 1 percent by weight based on the tobacco.

In addition to the above flavor emulsifiers there are certain innocuous or non-flavor emulsifiers that are fairly efficient emulsifying agents and effective in such small amounts that their impact in the final product is imperceptible. Thus, it has been found that stearic acid or lauric acid or lauryl ether sulfate, or the alkali or alkaline earth metal salts thereof fall within this group. Emulsifiers of this type are effective when used in an amount below 1 percent by weight of the tobacco, an effective range for satisfactory emulsification being from about 0.0002 g to about 0.2 g per 100 g of tobacco.

While the various emulsifiers mentioned above are preferably used singly if at all, they may also be used in admixture as long as the amounts do not exceed the upper limits given for the flavor type or the non-flavor type of emulsifiers.

The tobacco slurry, containing the binding agent and the low-boiling hydrocarbons is subjected to a high speed mixing and refining operation to obtain a homogeneous dispersion of the ingredients in the aqueous medium. A Cowles dissolver with a high shear impeller or a Waring blender may be used for the mixing step. Little if any foaming or entrapment of air takes place during the high speed dispersion since only very small quantities of dispersing agents are needed or used in the process. Following the high speed mixing step, the slurry is brought to below 100°C. or preferably room temperature if above this point. One then adds the volatilizable aliphatic hydrocarbon agent or agents, and preferably also the emulsifying agent. The entire mixture is again thoroughly dispersed or emulsified.

Following the effective dispersion of the hydrophobic agent throughout the slurry mixture, the dispersion mixture is then forwarded to a heating and foaming zone for forming a foamed, spongy sheet or web of reconstituted tobacco. Any conventional heating means for drying a tobacco slurry to form a reconstituted sheet may be used. One may use radiant energy heating such as that provided by infrared heating means or by using heated surfaces where the tobacco-hydrocarbon dispersion is cast or extruded onto metal plates or a moving stainless steel belt generally heated from the bottom with steam or hot gases.

The heating of the tobacco sheet containing the dispersed hydrocarbons causes the hydrocarbons or the hydrocarbon-water azeotrope to volatilize into gaseous or vapor form and in escaping, forms cavities between the interstices of the tobacco particles producing a substantially expanded product. The heating step is carried out at a temperature in the range of about 150° – 240°F. preferably from 180° – 220°F. When a moving belt is used, the speed of travel through the heating area may be 1 to 18 ft/min., and preferably 2–8 ft/minute. The dried product in the form of an expanded web or sheet may be removed using a doctor knife.

In the heating and drying operation, the temperature of drying causes the tobacco fines in conjunction with the tobacco pectin binding agent to cohere. Additionally, the low boiling hydrocarbons and water foam and volatilize off, forming cavities and expanded areas when viewed in cross-section, thereby resulting in a porous, spongy, product with a uniform color similar to natural tobacco leaf. Reconstituted tobacco sheet made in the usual manner is generally darker on top than on the belt side with the latter being mottled. A surprising characteristic of the product produced by the present method is its porosity and sponginess. In point of fact, it is so spongy and porous that a drop of water placed on its surface is absorbed as though by blotting paper.

Physical tests of the products produced by the present invention show a substantially lower density and greater thickness than that obtained in reconstituted tobacco sheet made by the usual methods. In one such test, density measurements may be carried out by weighing oven-dry material, then conditioning at 25°C. to 60 percent relative humidity for thickness measurement to avoid breakage. Density may be designated in g/sqft/10 mil thickness, although one may select any thickness from 1 to 100 mils provided all density measurements are calculated to the same thickness. In carrying out the present invention, the products produced demonstrated materially reduced densities over controls of unfoamed reconstituted tobacco tested under similar conditions.

When the products are compared to unfoamed reconstituted tobacco controls on a thickness basis, the products of the invention measured substantially thicker as well as more spongy than the controls. Moreover, in testing the products for wet strengths, the products of the invention possessed a wet strength well above 1.0 g/in/g/sq ft which permits subsequent processing of the sheet on standard equipment without difficulties.

The reconstituted foamed product, when prepared without the use of an emulsifier or when a fatty acid type of emulsifier was used, when cut or shredded to make cigarettes, required less tobacco and provided a smoke that was indistinguishable from control cigarettes prepared with ordinary reconstituted tobacco. There were no notes foreign to tobacco that could be detected in the prepared cigaret. Moreover, the product blended well with tobacco leaf filler. When the reconstituted foamed product was prepared using a flavoring type of emulsifier, the flavor of the tobacco was not only desirably enhanced but the product also possessed a substantially increased filling capacity and much lower density than ordinary reconstituted tobacco.

The following examples are illustrative of the invention. All parts as given below should be understood to be parts by weight unless otherwise designated.

EXAMPLE 1

Three hundred grams of tobacco slurry was prepared by mixing in a blender 10 parts of tobacco fines, 0.8 parts diammonium hydrogen phosphate (DAP), 1.6 parts of aqueous ammonia (30 percent NH_3) and 87.5 parts of water. The slurry was heated for one hour in a steam bath, refined for 10 minutes, and cooled to room temperature. Additional ammonia was added to maintain pH 9.0 during the heating period. After the cooling, the slurry was mixed for five minutes while 0.6 g of licorice powder and 0.15 ml heptane were added. After the mixing, the slurry was cast on stainless steel plates using a casting knife with a 50-mils setting, dried over a steam bath, and doctored off the plates. Sheet weight in grams per square foot was determined on the bone-dry material, and calculated for 10 mils thickness (a density index). Thickness of the sheet material that was equilibrated at 60 percent RH and 25°C. was measured with a model 549 micrometer, (Testing Machines, Inc. Amityville, L.I., N.Y.). The sheet was 11.95 mils thick and weighed 11.68 g/sq ft/10 mils. The sheet was spongy and porous. It had a similar color both on the plate and top sides, and resembled a dark natural tobacco leaf.

A control sheet was prepared as above but without the addition of licorice and heptane. This sheet was 8.66 mils thick and weighed 16.21 g/sq ft/10 mils. The top side of this sheet was dark, while the plate side was light in color and somewhat mottled.

EXAMPLES 2-7

In each of these six examples, 300 g of tobacco slurry was prepared as in Example 1, except the licorice amounts were varied from none to 1.5 grams and the heptane level was 0.15 ml in all cases. The casting and drying operation was carried out without delay.

The results are shown in Table 1. The color of the sheets in this series was similar for both sides and resembled natural dark tobacco leaf. The sheets were thicker and weighed less than the control.

TABLE 1

Example No.	Licorice Added g	Thickness mils	Sheet Weight g/sq ft/10 mils
Control*	—	8.66	16.21
2	—	10.73	13.29
3	1.5	12.23	11.19
4	1.2	12.10	12.93
5	0.9	12.50	11.59
6	0.15	11.64	12.56
7	0.075	11.51	12.36

*Control without heptane of licorice

It will be noted from these examples that an improved product, as compared to the control was produced even when an emulsifier was not used since the dispersion was not permitted to separate or break and lose heptane unduly. It will be noted, however, that when using the licorice emulsifier a lower density and thicker product was obtained. It will be further noted that increasing amounts of emulsifier generally caused a decreasing density and a thicker product.

EXAMPLE 8

The tobacco slurry was prepared as in Example 1 except 1.20 g. ammoniated glycyrrhizin and 0.60 ml heptane were added to 800 g slurry, and the slurry was cast at 35 mils instead of 50 mils. A control was also prepared.

	Sheet Thickness mils	Sheet Weight g/sq ft/10 mils
Control	7.0	13.0
Treated	8.5	11.3

EXAMPLE 9

The tobacco slurry was prepared as in Example 1, and maintained at 80°C. One hundred grams of the slurry was mixed for five minutes after adding 3.0 g of tobacco resins (hexane solubles). After the mixing, additional 200 g of slurry was added, and blended. When the mixture was cooled to 50°C., 0.60 ml heptane was added with additional blending for five minutes, then cast on stainless steel plates using a casting knife with a 50 mils setting. The process was completed to yield the treated sheets. Control sheets were also prepared containing the added tobacco resins, but no heptane.

	Sheet Thickness mils	Sheet Weight g/sq ft/10 mils
Control	8.4	18.2
Treated	14.4	11.1

The treated sheets were lighter and more uniform in color than the controls.

EXAMPLE 10

Three hundred grams of tobacco slurry was prepared as in Example 1 and cooled to room temperature. To the slurry were added 0.15 g stearic acid and 0.30 ml heptane, then mixed for five minutes. It was cast on stainless steel plates using a casting knife set at 50 mils and processed to yield the treated product. A control product was also prepared without stearic acid or heptane.

	Sheet Thickness mils	Sheet Weight g/sq ft/10 mils
Control	8.63	15.85
Treated	11.87	11.25

EXAMPLE 11

A tobacco slurry was prepared using a generally similar procedure to that described in U.S. Pat. No. 3,353,541 or U.S. Pat. No. 3,420,241 for obtaining tobacco pectins. Thus, to 80 parts of water at 195°C., 16 part of tobacco fines, 1.3 parts of DAP, and 2.6 parts of aqueous NH_3 (30% NH_3) were added. The mixture was stirred for one hour and subsequently refined in a disk type refiner. Additionally NH_3 was added to maintain a pH of 9.0. The slurry was cooled to room temperature, three hundred grams of the slurry was transferred into 200 ml water in which was dispersed 0.1 g calcium searate and 5 ml heptane, thereby providing about 0.2 g of calcium stearate and about 7 g of heptane per 100 g of tobacco dust. The mixture was stirred until it appeared homogeneous, usually about 5-10 minutes, cast on stainless steel plates using a casting knife with a 50 mils setting, dried over a steam bath, and doctored off the plates. The sheet was 14.0 mils thick and weighed 9.0 g/sq ft/10 mils. The color was similar on both sides, and the sheet had good physical

characteristics as to wet tensile strength, texture and porosity.

A control without calcium stearate or heptane was prepared as above. This sheet was 9.0 mils thick and weighed 16.3 g/sq ft/10 mils. It was a dense sheet with the top side much darker than the steel plate side.

The reconstituted tobacco sheets were shredded and prepared into hand made cigars. When smoked, the smoke of the experimental and control cigars appeared the same. There was no evidence of any mouth coating from either cigar.

EXAMPLES 12-14

In Example 12, 50 g of the above slurry as prepared in Example 11 with calcium stearate and heptane was dispersed in a blender containing 200 ml of water. To this was added 300 g of the original concentrated tobacco slurry without additional calcium stearate or heptane, and mixed until homogeneous, usually 5-10 minutes. This mixture contained approximately 0.01 g calcium stearate and 0.5 ml heptane, thereby providing approximately 0.02 g of calcium stearate per 100 g. of tobacco dust. The tobacco sheets were prepared as in Example 11. Results are shown in Table 2.

In Example 13, 50 g of slurry as prepared in Example 12 was dispersed in a blender containing 200 ml water. To this was added 300 g of the original concentrated tobacco slurry and treated as in Example 12. The final mixture contained approximately 0.001 g calcium stearate (0.002 g/100 g tobacco dust) and 0.05 ml heptane. Results are shown in Table 2.

In Example 14, 50 g of slurry as prepared in Example 13 was dispersed in a blender containing 200 ml water. To this 300 g of the original concentrated tobacco slurry was added and processed as in Example 12. The final mixture contained approximately 0.001 g calcium stearate (0.0002 g/100 g tobacco) and 0.005 ml heptane (0.0068 g/100 g tobacco). Results are shown in Table 2.

TABLE 2

Example No.	Calcium Sterate g	Heptane ml	Thickness mils	Weight g/sq ft/10 mils
Control*	—	—	9.0	16.3
12	0.01	0.5	11.0	11.3
13	0.001	0.05	10.0	11.2
14	0.0001	0.005	9.5	15.5

*No calcium stearate or heptane

EXAMPLE 15

Twenty-one hundred grams of tobacco slurry was prepared as in Example 1 and allowed to cool to room temperature. Three hundred grams of the prepared slurry was mixed in a blender for five minutes, then cast on stainless steel plates using a casting knife set at 50 mils. The process was carried to completion to yield the control tobacco sheet. To another 300 g portion of the slurry was added 0.15 g sodium lauryl ether sulfate and 1.5 ml heptane and mixed for five minutes. This tobacco slurry was cast and processed to yield the experimental tobacco sheet.

	Thickness mils	Weight g/sq ft/10 mils	Wet Tensile Coefficient g/in/g/sq ft
Control	7.4	19.0	7.8
Experimental	12.8	11.6	1.9

The experimental sheet was light in color and similar on both sides, whereas the control sheet was dark on

the top and mottled on the plate side. The wet tensile coefficient is sufficient for processing the sheet into cigarettes using machinery that is current in the cigaret industry today.

EXAMPLES 16-20

The procedure in Example 15 was repeated using 300 g portions of the prepared slurry to each of which 0.15 g sodium lauryl ether sulfate and varied levels of hydrocarbon foaming agent were added. The heptane levels ranged from 0.30 ml to 0.003 ml. The mixtures were blended, cast and dried to yield the experimental tobacco sheets.

TABLE 3

Example No.	Heptane Added ml	Thickness mils	Weight g/sq ft/10 mils	Wet Tensile Coefficient g/in/g/sq ft
16	0.30	13.5	11.0	5.3
17	0.15	12.5	11.1	5.5
18	0.03	10.3	13.8	6.2
19	0.015	8.8	15.3	5.8
20	0.003	8.3	16.0	7.3

EXAMPLE 21

The tobacco slurry was prepared as in Example 1 and cooled to room temperature. To 300 g of the slurry was added 0.30 g sodium lauryl ether sulfate and 0.15 ml petroleum ether (density 0.64). After mixing in a blender for 10 minutes it was cast at 50 mils on stainless steel plates, dried over a steam bath and doctored off the plates. Control sheets without the lauryl ether sulfate or petroleum ether were also prepared.

	Sheet Thickness mils	Sheet Weight g/sq ft/10 mils
Control	10.2	14.6
Treated	16.2	10.2

EXAMPLE 22

Eight hundred grams of tobacco slurry was prepared as in Example 1. After cooling the slurry, 0.80 g sodium lauryl ether sulfate and 0.30 heptane were added and mixed for 5 minutes in a blender. After mixing, the slurry was passed at 6000 psi through a Manton-Gaulin homogenizer Model 15A8BASMD (Manton-Gaulin Mfg. Co., Inc. Averett, Mass.) and immediately cast on stainless steel plates using a casting knife with a 35-mils setting. These sheets were then dried over a steam bath and doctored off the plates. A control was also prepared in the same manner as above, but no lauryl ether sulfate or heptane were added to the slurry.

	Thickness mils	Weight g/sq ft/10 mils
Control	5.26	19.65
Experimental	11.51	9.38

The experimental product was uniform and light in color whereas the control was dark on the top and mottled on the plate side.

The reconstituted films were shredded and hand made cigars prepared. The smoke flavor of the experimental cigaret was indistinguishable from that of the control. The additives in the experimental had no apparent effect on smoke flavor characteristics nor was mouth coating noted.

EXAMPLES 23-26

Temperature Variation

Four hundred grams of tobacco slurry was prepared by mixing in a blender 10 parts tobacco fines, 0.8 parts

DAP, 0.1 parts triethylene glycol, three parts of aqueous ammonia (30 percent NH₃) and 86.1 parts water. The slurry was heated for one hour on a steam bath, refined for ten minutes (pH maintained at 9.5), and cooled to 80°C. To this mixture 0.2 g sodium lauryl ether sulfate and 0.2 ml heptane were added and mixed for five minutes. The slurry was then cast on stainless steel plates using a casting knife with a 35-mils setting, dried over a steam bath and doctored off the plates. A control was prepared as above but without the lauryl ether sulfate and heptane. Sheet thickness and weight were determined and the percentage increase in thickness and decrease in weight were calculated. Results are shown in Table 4.

The above procedure was repeated except the slurries were cooled to 60°C., 40°C., and 20°C. at the time of addition of lauryl ether sulfate and heptane just prior to casting. Controls were also prepared for each corresponding temperature.

Experimental films were uniform in color with both sides similar. The control films were dark on the top side and mottled on the plate side.

TABLE 4

Example Number	Casting Temp. °C.	ness mils	Weight g/sq ft/10 mils	Thickness Increase Above Control %	Weight Decrease Below Control %
23	80	8.10	11.28	55.8	29.6
Control*	80	5.20	16.02		
24	60	9.77	8.90	101.0	49.2
Control*	60	4.86	17.53		
25	40	12.83	6.66	168.4	61.4
Control*	40	4.78	17.24		
26	20	12.63	6.64	166.5	60.9
Control*	20	4.74	17.00		

*No added lauryl ether sulfate or heptane.

The results of this group of examples (23-26) indicate that temperatures of the slurry after blending with binding agent and before the addition of hydrocarbons for foaming should be well below 100°C. and temperature adjustment to a lower level should be carried out and maintained until the combined slurry mixture

passes to the heating and drying stage.

The invention that is claimed is:

1. The method of making a reconstituted and expanded tobacco product comprising the steps of:

- Forming a slurry of tobacco plant particles and a binder of tobacco derived pectins,
- Intimately dispersing an aliphatic hydrocarbon of five to eight carbon atoms or mixture of such hydrocarbons in an amount of about 0.007 to about 7.0 parts per 100 parts of tobacco by weight into the tobacco slurry,
- forming the slurry into web or sheet form,
- Heating said tobacco slurry while in said web or sheet to a temperature of between 150° and 240°F and for a time sufficient to vaporize off water and all of said hydrocarbons and then,
- drying the web or sheet, said heating and drying steps effecting a bonding of the tobacco particles and the formation of cavities or expanded areas in and on the dried product.

2. The process of claim 1 in which the hydrocarbon is selected from the group consisting of heptane and petroleum ether.

3. The process of claim 1, wherein at step (c) a flavoring emulsifier is added in an amount between about 1 and 5 parts per 100 parts of tobacco fines by weight.

4. The process of claim 3, in which the emulsifier is tobacco resins.

5. The process of claim 3, in which the emulsifier is licorice or a salt of glycyrrhizin.

6. The process of claim 1, wherein at step (c) a non-flavoring emulsifier is added in an amount between about 0.0002 and 0.2 parts per 100 parts of tobacco fines by weight.

7. The process of claim 6, in which the non-flavoring emulsifier is stearic acid or an alkaline salt thereof.

8. The process of claim 6, in which the non-flavoring emulsifier is lauryl ether sulfate or an alkaline salt thereof.

* * * * *

UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 3,746,012 Dated July 17, 1973

Inventor(s) Edward John Deszyck

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 2, line 57, insert an -- a -- between "of" and "special".

Column 3, lines 10, 48-49, 54 and 63-64, "in situ" should be -- in situ --.

Column 7, Table I, in footnote, "of" should be -- or --.

Column 9, line 25, "surry" should be -- slurry --.

Column 9, Table 2, in second column and in footnote, "sterate" should be -- stearate --.

Column 10, line 57, "fills" should be -- films --.

Column 11, Table 4, 3rd column heading, "ness
mils"
should be -- Thickness --.
mils

Column 11, line 39, "1002C." should be -- 100°C. --.

Claim 3, line 1, "step (c)" should be -- step (b) --.

Claim 6, line 1, "step (c)" should be -- step (b) --.

Signed and sealed this 16th day of July 1974.

(SEAL)
Attest:

McCOY M. GIBSON, JR.
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents