March 17, 1964 R. J. OSBORNE 3,125,098

TOBACCO SHEET MANUFACTURE

Filed Sept. 23, 1960 2 Sheets-Sheet 1

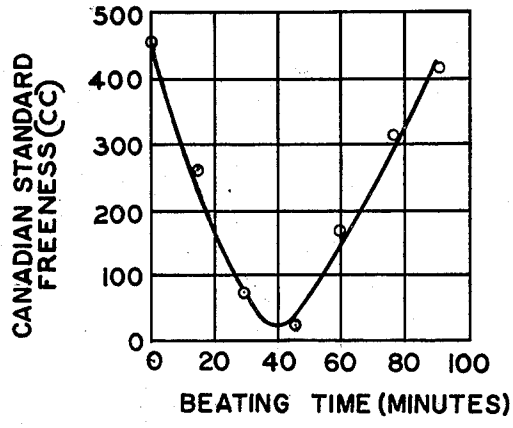



Fig. 1

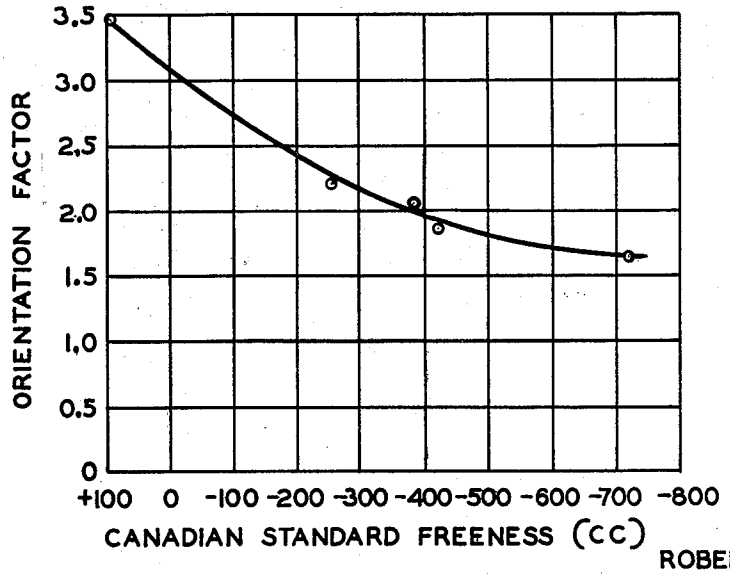



Fig. 2

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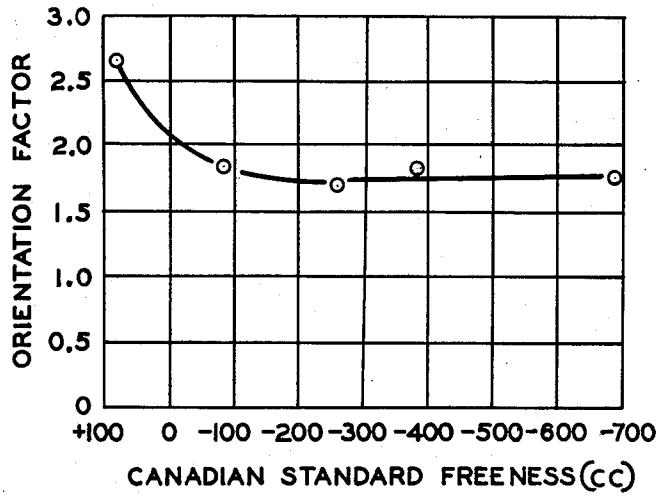



FIG.3

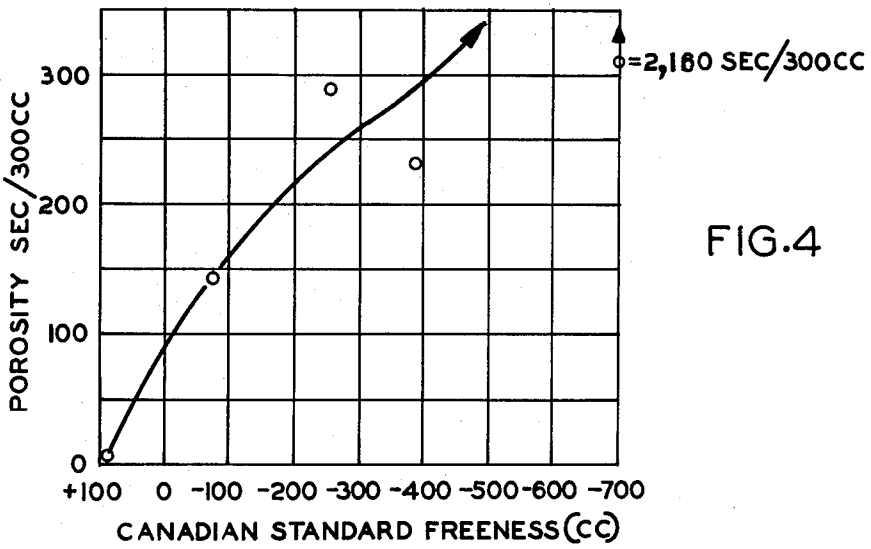



FIG.4

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United States Patent Office 3,125,098 Patented Mar. 17, 1964



3,125,098

# TOBACCO SHEET MANUFACTURE

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Filed Sept. 23, 1960, Ser. No. 58,093
24 Claims. (Cl. 131—17)

This invention relates to reconstituted tobacco sheet products and more particularly to a method for making an improved self-sustaining tobacco sheet suitable as a wrapper material from aqueous tobacco dispersion and to the product produced thereby.

In the formation of tobacco sheet by casting a tobacco dispersion into films and evaporating moisture therefrom various formulations have been proposed. In making tobacco sheet to be used as a wrapper for cigars, it is important that the leaf, web or sheet have adequate strength and resiliency to permit wrinkle free application on the ends of the cigar. Although sheets formed from reconstituted tobacco formulations of the prior art, including formulations containing fibrous material obtained by dehydrating a paper pulp to which the present invention is more particularly concerned, have been employed as binder material, no formulation known heretofore has had satisfactory physical properties making it attractive for wrapper material.

In general the following practical considerations are important in tobacco sheet manufacture. It is highly desirable that a tobacco sheet material have sufficient tensile strength and dimensional stability to withstand vigorous manipulation in the course of manufacturing and so be self-supporting, particularly in wrapping cigars where it is essential that the leaf resist shear and wrinkling when applied to a contoured surface. The sheet must also hold together well in smoking products such as cigars, cigarettes, or pipe tobacco. Preferably, it should have at least the tensile strength of natural leaf tobacco. Moreover, the tobacco sheet material should be flexible; it should resist disintegration by moisture and have wet strength so as not to gum up upon blending, casing or similar treatment as well as when used ultimately in smoking or chewing.

It is an object of this invention to provide a self-supporting tobacco sheet material of excellent strength which is easily worked into a smoking product and which has substantially the natural characteristics of tobacco including taste, color and aroma.

A further object of this invention is to provide a method of forming a self-supporting tobacco wrapper sheet material which has exceptional physical properties including unexpected tensile strength, excellent wrinkle resistance and good wet strength.

It is a further object of the invention to provide novel tobacco strip material, such as sheets, continuous webs, and the like wherein fine tobacco particles and/or dust are formed into a slurry or otherwise intimately blended into a web of film forming material containing highly refined fibers which form an integral part of the product produced and wherein said film forming material and fibers have no deleterious effect upon the product when smoked.

Other objects and advantages of the invention become apparent as the more detailed description of the invention progresses.

Fibrous materials, e.g. paper pulp, have been used heretofore to reinforce reconstituted tobacco sheet consisting essentially of ground tobacco and an adhesive in addition to the fiber. The use of the fiber permits a reduction in the amount of adhesive required to produce a structurally sound sheet and thus affords a reduction in the cost of the product as well as permitting an increase in the tobacco content of the sheet.



Suitable organic fibers which may be employed are the cellulosic materials derived from wood such as cellulose pulp in the form of cigarette paper pulp, glassine paper pulp, vegetable fibers or kenaf (ambary). Mineral fibers are also suitable such as asbestos or glass fibers. The fibrous material is generally suspended with the adhesive film forming agents in water.

The fibrous material and also the selected adhesive film forming agents are selected to form a composition which when burned in the smoking article do not adversely affect the blandness, flavors, aroma or burning qualities of the tobacco.

The cellulosic pulps which have been heretofore preferably utilized in compositions for making tobacco sheet have frequently been of the type used in the paper industry to produce glassine paper. Glassine paper is a thin hard and almost transparent paper made from chemical wood pulp which is considered highly beaten based on standards for pulp used in paper making. The pulps used for glassine paper are generally those obtained from softwoods and are prepared by the sulfite cooking process. Glassine type pulp is long fibered and easily hydrated. The pulp is well beaten and well hydrated; i.e., the fibers have imbibed a considerable quantity of water. Pulp used for glassine paper receives the largest amount of mechanical treatment of any pulp stock used for other purposes including for example writing paper or newsprint. A glassine pulp is generally produced by extended beating in a beater where the mechanical action is such as to bruise and crush the fiber, with only a minor amount of cutting of the fiber. This action forms numerous fibrils, i.e. microscopic and sub-microscopic hair-like filaments extending from the surface of the parent fiber, and as a consequence increases the surface area of the fiber with a resultant increase in absorbed water. A pulp of this type is a slow draining stock; i.e., if the pulp is allowed to drain through a screen, the fiber mat which first forms on the screen so restricts the flow that extended times are required for the remainder of the water to drain. Drainage rate is often employed to characterize such pulps.

A measure of the drainage rate, i.e. of the refinement state of the pulp, is obtained by means of a "freeness" test in which measurement of the flow rate through a pad of fibers formed on a standard screen are determined. "Freeness" may also be described as the extent to which the pulp has not been hydrated by beating. Two of the most common instruments for the measurement of freeness are the "Canadian Standard Freeness Tester" and the "Schopper-Riegler Freeness Tester." In both of these instruments, the quantity which is determined is the volume of water (expressed in cc.) which overflows from a receiver containing an orifice outlet at the bottom. In the present specification, Canadian Standard Freeness measurements are employed. In the testing instrument, high drainage rates into the receiver from the screen result in large overflow rates and hence a high freeness. Conversely, low drainage rates result in a low overflow and hence a low freeness. Glassine pulps, which are slow pulps, will normally have a Canadian Standard Freeness of about 80–100 cc.

A glassine type pulp when incorporated into a cast tobacco sheet formulation used for binder on cigars, provides longitudinal strengths of approximately 810 gm./in. and transverse strength of about 300 gm./in. for a sheet which weighs 5 gms./sq. ft. The ratio of the two strengths is known as the "orientation factor." With the use of glassine type pulp in the tobacco formulation this value is about 2.7 to 3.5. It is believed that orientation results from alignment of the fibers by viscous shear forces in the casting operation. A high orientation factor such as that obtained with glassine pulp is less than desirable

since the sheet must be made stronger in the longitudinal direction than necessary to compensate for the lower strength in the transverse direction. This means that either a thicker sheet must be made or the adhesive must be increased at the expense of tobacco content. In addition, improved conformance to contoured surfaces is obtained if the physical properties of the sheet are isotropic; i.e. uniform in all directions. By reducing the orientation factor according to the present invention, this ideal is approached.

Other characteristics of tobacco sheet employing glassine type pulp are also somewhat deficient. The elongation of the tobacco sheet, formed with glassine pulp, at break is about 5% and poor conformance to the cigar shape and/or fracture of the sheet in the cigar molding operation occurs. The resistance to flow of air through the sheet is determined on an instrument such as the Gurley Densitometer which measures the rate of air flow through a unit area under a standard pressure. The units used are time/(unit volume or air) (unit area) and the units stated in the present specification are seconds/(300 cc. of air) (1 square inch). A high reading indicates large resistance to air flow, which is most desirable for a wrapper from the viewpoint of ease of draw on the cigar. A typical sheet described above which uses glassine type pulp will have a porosity reading of 3 to 15 sec./300 cc. On the other hand, a porosity reading of 60 to 120 sec./300 cc. would be much more attractive.

Although attempts have been made to produce tobacco sheet of increased strength using pulps heretofore available for other purposes, no results are known which have been practically advantageous. Attempts have been made, for example, to substitute pulps (other than glassine) which have been shortened by cutting and therefore should have a reduced orientation factor. In so doing, it has been possible to reduce the orientation factor, i.e. ratio of longitudinal strength to transverse strength, but this is done at the expense of the longitudinal strength, rather than as desired by increasing the transverse strength. Other fibers which do not have an "inverted" Canadian Standard Freeness, such as those from tobacco stems, have a similar effect; they fail to produce suitable physical properties.

I have found that if pulps including glassine pulp are subjected to additional beating beyond the heretofore considered advanced stage even for glassine type paper, surprising results are obtained. Although the fiber is shortened as the beating is continued past the generally recognized maximum beating, the longitudinal strength of the dried tobacco sheet made with this pulp surprisingly does not decrease as would be expected. Even more striking is the fact that the transverse strength of the sheet increases as the time of beating of the pulp is increased. This unexpected result is attended by other desirable improvements in elongation and resistance to air flow. The advantages of the invention are produced by incorporating into the sheet forming formulation a fibrous pulp which has been refined so that it has an "inverted" Canadian Standard Freeness. The lower limit of refinement may be above an inverted Canadian freeness of 1000, but preferably pulp having an inverted Canadian Standard Freeness in the range between 50 and 900 is employed.

The shortening of pulp fiber size in the beating process is evidenced in the freeness test. The curve of freeness vs. beating time is of negative slope, i.e., freeness decreases with time of beating during the normal beating cycle. However, as the beating is continued beyond the normal limits, the freeness begins to increase with time. In this range of freeness, fines are noted in the overflow stream and it is evident that solid matter is coming through the screen, rather than forming a mat on it. As beating is continued, the number of fines increases and more and more of the material passes through the screen.



The freeness in this range is described herein as "inverted," a term which is applied to the Canadian freeness value of a pulp, which, if subject to additional heating, would show an increase instead of a decrease in freeness. An example of such a refining curve is shown plotted in FIG. 1 of the drawing. It may be that a pulp in the inverted area would be of little utility in paper manufacture because of the loss of fines through the Fourdinier screen. However, in the use thereof in formulation for tobacco sheet manufacture, exceptionally advantageous and unexpected results are obtained.

Because of the seemingly unorthodox result, i.e. retention of longitudinal strength with increase in transverse strength, the highly refined pulp characterized in the present invention must have an unusual characteristic to compensate for its short fiber. Microscopic examination of the fiber reveals the presence of an extremely large number of very fine fibers, or as more properly described, "fibrils." These fibrils exist as separate entities as well as being attached to and protruding from the parent fiber. The net effect of this separation is to increase the total length of fibrous material available to the system for reinforcement. Although it is not intended that the invention rely on this theory, it is probably this condition which offsets the expected effect of shortening the fiber. Because these fibrils are of much smaller diameter than the parent fiber, they are less influenced by hydrodynamic forces in the casting process so that transverse reinforcement is obtained.

The following general procedure is employed in preparing tobacco sheet material.

Finely divided tobacco is mingled with an adhesive formulation which includes fibers, to form a slurry or a suspension. The viscosity of the slurry is controlled by the relative amount of water, tobacco and adhesive used. To promote the mingling of tobacco particles and film forming agent, the slurry is agitated thoroughly until all the particles are completely wetted. Mixing may conveniently be done in a high shear mixer or a ball mill. In a ball mill, the relation of ball size and volume of the mixing chamber will depend upon several factors such as viscosity, particle size of tobacco and proportion of adhesive material solids to tobacco.

Alternatively, the tobacco particles and dry adhesive may be mixed together and added to the fiber slurry. The product of either method may be applied to a continuous substantially impervious belt using known techniques such as by casting, reverse roll coating, spraying or other conventional film forming procedures to form a continuous film which is then dried and removed in a continuous sheet. The viscous slurry can also be shaped and formed into a sheet by suitable means such as by calendering or extrusion.

Drying the wet material is a part of the sheet forming operation and is necessary in most cases to achieve substantial water resistance. The moisture content of the sheet can be adjusted by conventional tobacco treating methods. The finished dried sheet may be conveniently handled in roll form.

In the following examples softwood pulps produced by the sulfite process are employed although other pulps, e.g. pulps from softwoods and hardwoods by the kraft process, may also be employed including pulp from rag fibers or vegetable fibers such as kenaf. Parts expressed in the examples are parts by weight unless otherwise stated.

*Example 1*

A pulp was prepared by refining a sulfite pulp of Canadian Standard Freeness (CSF)=100 cc. in a JUD #1 Microfiner. This refiner is a modified Jordan engine with a tapered rotating plug equipped with a hydrating filling. In this type of filling, the bars on the plug and the shell are so designed as to provide more brushing than cutting action. In addition to the plug, there is a disc attached to the end of the plug and this disc, which

matches a stationary disc, provides additional brushing action. The pulp slurry was passed through the refiner 24 times, with the clearances between the rotating and stationary surfaces being reduced each pass. At the end of refining, the inverted freeness was 380 cc. This super beaten or highly refined pulp, having an inverted CSF of 380 cc., was used in the following Run B, and compared with a formulation employing the glassine type pulp (before additional refining) of CSF +100, Run A. The quantities shown are in parts by weight on a bone dry basis.

| Run Number | 1A | 1B |
|---|---|---|
| Adhesive (0.5/0.5 guar gum/ethyl hydroxyethyl cellulose) | 1.0 | 1.0 |
| Sulfite Pulp | [1] 1.0 | [2] 1.0 |
| Humectant (glycerine) | 0.5 | 0.5 |
| Tobacco | 7.0 | 7.0 |
| Cross Linking Agent (glyoxal) | 0.3 | 0.3 |
| Additives [3] | 0.44 | 0.44 |

[1] CSF=100 cc.
[2] Inverted CSF=380.
[3] Includes about 0.40 part diatomaceous ash whitener and about 0.04 fungicide.

The sole difference in the two formulations resides in the degree of refining of the pulp. The mixture was formed into a slurry by dissolving the gum into the pulp and adding the other ingredients with enough water to bring the solids concentration to approximately 10%. The slurry was formed into a film by casting it onto an endless steel belt and drying off the water with heated air. The dried sheets had the following physical properties after equilibrium overnight in an atmosphere at 70° F. and 80% relative humidity:

| Run Number | 1A | 1B |
|---|---|---|
| Dry Long Tensile Strength, gm./in | 635 | 747 |
| Dry Transverse Tensil Strength, gm./in | 175 | 325 |
| Wet Long Tensile Strength, gm./in | 272 | 349 |
| Wet Transverse Tensile Strength, gm./in | 42 | 152 |
| Elongation at Break, Dry, Percent | 5 | 7 |
| Sheet wt., gm./ft.$^2$ | 4.58 | 5.00 |
| Equilibrium Moisture, Percent | 19.9 | 19.9 |
| Dry Longitudinal Breaking Length, ft.* | 1,660 | 1,790 |
| Dry Transverse Breaking Length, ft.* | 458 | 780 |
| Orientation Factor | 3.53 | 2.29 |

*Standard measurement of weight of sheet which will support itself. It is independent of sheet thickness and based on strength of the sheet and sheet weight i.e. weight of sheet per unit area.

It can be seen that the transverse strength was increased markedly (70%) and the elongation substantially (40%) by the use of the more extensively beaten pulp in the sheet forming formulation.

*Example 2*

This same pulp of Example 1 having a CSF of −380 was used in a different formula, Run B, and compared with the control formulation having pulp of 100 CSF as follows:

| Run Number | 2A | 2B |
|---|---|---|
| Adhesive (locust beangum/methyl cellulose .5/.5) | 1.0 | 1.0 |
| Sulfite Pulp | [1] 1.0 | [2] 1.0 |
| Humectant (diethylene glycol) | 0.75 | 0.75 |
| Tobacco | 6.0 | 6.0 |
| Cross Linking Agent (melamine-formaldehyde resin) | 0.50 | 0.50 |
| Additives [3] | 0.74 | 0.74 |

[1] CSF=100.
[2] Inverted CSF=380.
[3] Includes about 0.35 parts pigments, 0.40 part diatomaceous ash whitener and about 0.04 fungicide.

An improvement in the transverse strength and elongation similar to that obtained in Example 1 was found:

| Run Number | 2A | 2B |
|---|---|---|
| Dry Longitudinal Breaking Length, ft | 3,180 | 2,830 |
| Dry Transverse Breaking Length, ft | 920 | 1,380 |
| Orientation Factor | 3.46 | 2.05 |
| Elongation | 4.5 | 6.3 |

The decrease in the longitudinal strength in the results of Run No. 2 is considered within the error of experimental determination.

*Example 3*

Further illustration of this desirable effect is afforded by the following example in which a formula similar to that used in Example 2 was employed. The degree of refining of pulp is shown by the designated CSF value. The tensile data, which show the effects on transverse strength are shown in the following table together with data on the control (3A) having a CSF of +100 and representing conventionally refined pulp.

| Run Number | 3A (Control) | 3B | 3C |
|---|---|---|---|
| CSF | 100 | [1] 60 | [1] 810 |
| Longitudinal Breaking Length, ft | 2,560 | 2,380 | 2,710 |
| Transverse Breaking Length, ft | 8.14 | 945 | 1,270 |
| Orientation Factor | 3.15 | 2.52 | 2.13 |
| Elongation | 5.0 | 6.5 | 7.3 |

[1] Inverted.

The film forming agent is an important structural ingredient of the sheet. Accordingly, if the film forming agent is weak or discontinuous, the sheet will crumble and disintegrate when handled in tobacco machinery. The film forming agent should be easy to handle, should be chemically stable, should be in a convenient form, and should require little special treatment to prepare it for final use. When tobacco sheet material is fed from continuous rolls into automatic cigar making equipment, its crimping and setting properties may be critically important. In addition for wrapper material, the sheet is subject to failure by wrinkles because of poor conformance to the contoured shape. Tobacco sheet material made according to this invention produces desirable crimping and setting properties and conforms very satisfactorily when used in automatic cigar making machines and other machinery for fabricating tobacco smoking articles.

Any of the various adhesive film forming agents which are known and have been used in the art may be employed. In the finished tobacco sheet the adhesive formulation may be between 0.5% and 33% by weight but a preferred range is between 1% and 20%. The viscosity of the formulation, measured on a Brookfield viscometer Model No. RVF, #6 spindle and at a spindle speed of 20 r.p.m. is generally between 500 and 5,000,000 centipoises and preferably has a viscosity range between 6,000 and 20,000 centipoises. The adhesive film forming agent or binder is selected to impart to the finished dry tobacco sheet material a high degree of moisture resistance.

The preferred film forming agents are polycaccharides, or water soluble cellulose ethers or combinations thereof. For example, mixtures of locust bean gum and ethyl hydroxy ethyl cellulose; locust bean gum and carboxymethyl cellulose; locust beam gum and methyl cellulose; guar and ethyl hydroxyethyl cellulose or water soluble salts of carboxymethylcellulose and carboxymethyl hydroxyethyl cellulose and the like are suitable. Generally the polysaccharide materials are employed in the weight ratio of 1:20 to 1:1 based on the dry weight of tobacco. Guar and locust bean gum are among the commercially available galactomannan gums. When the adhesive film forming agent is provided in the form of dry powder, particle sizes used in this invention are preferably similar in size

to the tobacco particles although they may be smaller. Cellulose glycolic acid (acid form of carboxymethylcellulose) is also a desirable film forming material since the dried cellulose glycolic acid is substantially initially water-resistant. In addition to the other film forming materials such as viscose, the polyuronides, and the like when properly formulated are other polysaccharides which can be used. Polyuronides include all uronic acid containing polysaccharides such as pectins and pectin derivatives, pectates, pectinates, pectinic acid and pectic acid forms as well as algins, algin derivatives, alginates and alginic acid forms. In particular, water insoluble pectates such as calcium and magnesium pectate are valuable adhesive.

A cross linking agent, such as glyoxal, dimethylol urea, dialdehyde starch, or melamine-formaldehyde resins may be added to improve further the water resistance of the film forming agent. If desired, a humectant such as glycerine or triethylene glycol may be added to the film forming surface. When a humectant is used, it has been found that the quantity thereof required to maintain a given degree of pliability in the final sheet varies as the ratio of fibers to film forming agent is changed. It is preferred to use a minor quantity of humectant to prevent excessive brittleness in the final film.

Natural gums have the disadvantage of not producing a coherent sheet or film when used as the sole adhesive with the rapid drying of the slurry required to maintain commercial operation. As a result, cracks form in the film during the drying operation so that small islands are formed on the belt and after drying small pieces flake off the web detracting from the uniformity desired. Drying cracks can be minimized by reducing the drying rate, but uneconomical production results.

In order to eliminate the crack formation, thermogelling gums from the class of water soluble cellulose ethers e.g. methyl cellulose, ethyl hydroxyethyl cellulose are used in conjunction with the natural gums to form a suitable adhesive film forming material. The gums function by gelling the film before significant evaporation of water takes place. The gel is firm enough so that stresses induced by shrinkage are resisted and cracks do not occur. Although the gums are effective for this phenomenon, they have shortcomings which include high price relative to the natural gums, difficulty in forming solutions and poorer reaction with the cross linking reagents used to impart wet strength to the tobacco sheet. For these reasons, it is most desirable to eliminate or at least reduce the quantity of thermogelling gum required.

I have found that the pulp highly refined according to the teaching of the invention permits the substantial minimization of needed thermogelling gum. The highly refined pulp described herein upon drying in the sheet forming composition is more effective in eliminating drying cracks than conventional pulps. This is illustrated by the following example.

*Example 4*

The slurry was made in the conventional fashion from the following formulations:

| Run Number | 4A (Control) | 4B |
|---|---|---|
| Locust Bean Gum | 0.8 | 0.8 |
| Ethyl hydroxyethyl cellulose | 0.2 | 0.2 |
| Pulp | [1] 1.0 | [2] 1.0 |
| Humectant tetraethylene glycol | 0.75 | 0.75 |
| Tobacco | 6.0 | 6.0 |
| Cross Linking Agent Glyoxal | 0.3 | 0.3 |
| Other Additives [3] | 0.44 | 0.44 |

[1] CSF=100 cc.
[2] Inverted CSF=380.
[3] Same as Example 1.

Upon drying because of insufficient ethyl hydroxyethyl cellulose thermogelling gum in the formulation, the control 4A, was badly cracked and tensile data could not be taken. However, run 4B, using the same amount of ethyl hydroxyethyl cellulose produced a solid sheet and had the following tensile properties after equilibration at 80% relative humidity:

| | |
|---|---|
| Dry longitudinal tensile strength, gm./in. | 805 |
| Dry transverse tensile strength, gm./in. | 415 |
| Wet longitudinal tensile strength, gm./in. | 485 |
| Wet transverse tensile strength, gm./in. | 220 |
| Sheet weight, gm./ft.[2] | 4.44 |

Pulp from other sources, such as those produced by the kraft or sulfate process may also be used to produce satisfactory materials. Kraft pulps, which provide the strongest papers, require somewhat longer refining times to reach a given freeness than the sulfite pulps, but at a given freeness the pulps are comparable. The following example illustrates the use of a kraft pulp.

*Example 5*

An unbleached commercially available kraft pulp (from Brightwater Paper Company) was refined at a 13 pound bedplate load in a laboratory Valley Beater from 690 cc. CSF to 385 cc. inverted CSF in 430 min. The fiber content was maintained at 1.8% of the total suspension. The refined pulp was incorporated into the formula of Example 2. The tobacco sheet had the following properties:

| | |
|---|---|
| Dry longitudinal breaking, length-ft. | 2460 |
| Dry transverse breaking, length-ft. | 1350 |
| Orientation factor | 1.82 |
| Elongation at break ----percent-- | 8 |

*Example 6*

Similar results were obtained with a bleached kraft hardwood pulp in the same formula. This pulp had been refined in a similar manner to the above pulp. The freeness of the pulp was taken from 630 cc. to 425 cc. (inverted) in 190 minutes. The following tensile properties were obtained:

| | |
|---|---|
| Dry longitudinal breaking, length-ft. | 2410 |
| Dry transverse breaking, length-ft. | 1490 |
| Orientation factor | 1.62 |
| Elongation at break ----percent-- | 7 |

*Example 7*

This example illustrates further the effect of the extent of the refining on the orientation factor. The formula of Example 2 was used in this series of runs. The pulps were from the same source and were prepared in the same manner as that used for the pulp of Example 2. The following tensile data were obtained:

| Run Number | 7A | 7B | 7C |
|---|---|---|---|
| Canadian Standard Freeness (inverted) of Pulp, cc | 262 | 410 | 725 |
| Dry Longitudinal Breaking Length, ft. | 2,410 | 2,500 | 2,290 |
| Dry Transverse Breaking Length, ft. | 1,075 | 1,330 | 1,400 |
| Orientation Factor | 2.24 | 1.88 | 1.64 |
| Elongation, Percent | 9 | 9.5 | 9.5 |

When data are combined with those of Example 2, a correlation between orientation factor and pulp freeness can be obtained. This is shown in FIGURE 2. It must be noted that absolute strengths should not be compared between these two series since variations in mixing technique or gum lot will have an influence on the result. However, the orientation factor is influenced to a much lesser degree.

The following example comprising five runs illustrates as shown in FIGS. 3 and 4 that a kraft pulp when highly refined provides advantageous effects similar to those with the sulfite pulp.

*Example 8*

A pulp from the same source as in Example 5 was refined in the JUD #1 Microfiner. Samples of pulp were

removed during the course of refining so that a set of pulps of different freeness were obtained. These pulps were incorporated into the following formula:

| | Parts by weight |
|---|---|
| Guar Gum | 0.9 |
| Ethyl hydroxy ethyl cellulose | 0.1 |
| Pulp Fiber | 1.0 |
| Tobacco | 9.75 |
| Humectant (Diethylene glycol) | 0.40 |
| Cross Linking Agent (Dialdehyde starch) | 0.50 |
| Additives [1] | 0.26 |

[1]Same as Example 1.

The effects of refining on the orientation factor and on the porosity are shown in FIGS. 3 and 4 respectively. The tensile data which were obtained are indicated below:

| Run Number | 8A | 8B | 8C | 8D | 8E |
|---|---|---|---|---|---|
| Pulp Freeness, CSF (cc.) | 90 | [1] 85 | [1] 260 | [1] 390 | [1] 700 |
| Longitudinal Breaking Length, ft. | 2,200 | 2,340 | 2,410 | 2,280 | 2,280 |
| Transverse Breaking Length, ft. | 830 | 1,240 | 1,410 | 1,270 | 1,320 |
| Porosity, sec./300 cc. | 7 | 147 | 292 | 230 | 2,160 |

[1] Inverted.

The beneficial effects of the extended refining on transverse strength and porosity are apparent from the analyses of the products.

In FIGS. 2, 3 and 4 the inverted Canadian Standard Freeness values are identified as Canadian Standard Freeness values expressed in negative quantities in order to more clearly show the variations of the "Orientation Factor" and "porosity sec./300 cc." during the course of change of the said freeness values.

In addition to use in tobacco sheet formed by casting a film from an aqueous suspension containing ground tobacco, this pulp may be used in sheet formed by the process described in the U.S. Patent No. 2,734,509 of D. F. Jurgensen, Jr. In that patent, the use of a web of film forming material containing a fibrous material is described. The highly refined pulp which is the subject of this invention is also useful in that patented process, wherein fine tobacco particles or tobacco dust are adhered to both faces of a web of film forming material containing admixed fibers which form an integral part of the composite tobacco material produced. In the procedure described in the above Jurgensen patent, a layer of tobacco dust is applied to a wetted film forming surface, there is then applied to this layer of deposited tobacco a layer of film forming material containing the pulp. The properties imparted by the highly refined pulp, according to the discovery which forms the essence of the present invention, imparts the hereinabove disclosed improvements also to webs manufactured according to the technique described in that patent.

A representative example of a dispersion which may be used in conjunction with the process of U.S. Patent 2,734,509, i.e. a dispersion to which the tobacco dust is adhered, is the following.

*Example 9*

| | Parts |
|---|---|
| Sodium carboxymethyl cellulose | 1.0 |
| Sulfite pulp | [1] 1.0 |
| Glycerine | 0.3 |
| Water | 47.7 |

[1] (320 cc.) inverted CSF.

The dispersion provided by this formulation produces relatively higher film strengths than dispersions based on tobacco stem films, for example, and thereby permits the use of a lighter web in the sheet.

Although the illustrative examples herein presented has shown the advantages of using highly refined pulp with reference to controls using conventionally refined pulp, it is to be understood that the invention herein described also provides definite advantages when the highly refined pulp of various kinds is employed in admixture, as such, or in admixture with conventionally refined pulp in suitable proportions.

The tobacco employed in forming the tobacco sheet material of the invention may be derived from either leaves of stems and need not be by-product material although one of the economic advantages of the invention is the utilization of otherwise useless fines of good quality tobacco which often crumbles from the leaves during ordinary processing.

FIG. 1 shows the effect of beating time on the CSF value, the ascending portion of the curve showing the inverted value of this variable.

The finely divided or fragmented tobacco may be prepared by grinding or by other comminuting means. For example, dust may be used. Sheet using entirely dry ground tobacco is a preferred but not a necessary form of the invention. Tobacco which is entirely dry ground is tobacco which has not undergone comminution in the presence of excess liquid, such as a wet milling. Satisfactory tobacco sheets can be made from finely divided tobacco which will pass through a 20 mesh screen and will be mostly retained by a 325 mesh screen. A preferred particle size range is between 60 and 250 mesh. Small tobacco particles appear to burn more evenly than large particles.

The preferred finished sheet has a tensile strength superior to that of natural leaf tobacco. Of course, the strength and sheet thickness may be adjusted for particular applications. A preferred sheet thickness range is between .002" and .011". Tensile strength may be, for example, about 400 grams per inch on material .003" thick. The sheet is self-supporting and coherent even after immersion in water. In characterizing properties of the wetted web, strength may be defined so that at least one linear foot of material has a strength of about 400 grams.

Tobacco sheet material prepared according to this invention has preferably a moisture content in the range between about 6% and 48%. A particularly desirable range of moisture content in cigarette tobacco is 9% to 13% and in cigar binder 16% to 22% on a dry tobacco basis and about 30–40% for wrapper.

Some inorganic fillers which may be used in powder form, for example, with tobacco sheet material are kaolin and fuller's earth. Among suitable organic fillers are various cellulosic preparations. Fillers may range from 2% to 15% by weight of the finished sheet. Various food dyes may also be used in manufacturing the tobacco sheets and foils to produce preferred color to the web.

The tobacco sheet of this invention has many useful applications and smoking articles such as cigars, cigarettes, pipe tobacco as well as chewing tobacco made in whole or in part from this sheet material, are within the contemplation of the invention. The sheet material may be fed from rolls to automatic machines, for example, cigar machines, for use as a binder or wrapper. The sheet may also be shredded for filler in pipes, cigarettes, and cigars. It may be mixed with shredded whole tobacco or used alone. It may also serve as an outside wrapper for cigars or cigarettes and has the advantage of uniformity in appearance and in physical properties as well as uniformly blended flavor.

While the invention has been described in its preferred embodiment, it will be understood that the invention is not limited to the specific details set out for purposes of illustration and that those skilled in the art may make various changes in these details within the scope of the claims without departing from the spirit of the invention.

I claim:

1. The method of forming self-supporting tobacco

sheet which comprises reducing tobacco to a finely divided state, mixing with said tobacco from about 2% to about 30%, based on the weight of tobacco, of a highly refined fibrous pulp having an inverted freeness as measured by the Canadian Standard Freeness Test, and an aqueous fluid to form a homogeneous dispersion having a solids content of at least 5%, forming a film of said dispersion on a film forming surface substantially impervious to the solids content of said dispersion and removing moisture from the film.

2. The method of forming self-supporting tobacco sheet which comprises reducing tobacco to a finely divided state, mixing with said tobacco from about 2% to about 30%, based on the weight of tobacco, of a highly refined fibrous pulp having an inverted freeness between 50 cc. and 900 cc. as measured by the Canadian Standard Freeness Test, and an aqueous fluid containing a film forming agent to form a homogeneous dispersion having a solids content of at least 5%, forming a film of said dispersion on a substantial impervious film forming surface and evaporating moisture from the film.

3. The method of forming self-supporting reconstituted tobacco sheet which comprises reducing tobacco to a finely divided state, mixing with said tobacco from about 2% to about 30%, based on the weight of tobacco, of a highly refined softwood sulfite pulp having an inverted freeness as measured by the Canadian Standard Freeness Test, and an aqueous fluid containing a film forming agent to form a homogeneous dispersion having a solids content of at least 5% forming a film of said dispersion on a substantial impervious film forming surface and evaporating moisture from the film.

4. The method of forming self-supporting reconstituted tobacco sheet which comprises reducing tobacco to a finely divided state, mixing with said tobacco from about 2% to about 30%, based on the weight of tobacco, of a highly refined hardwood kraft pulp having an inverted freeness as measured by the Canadian Standard Freeness Test, and an aqueous fluid containing a film forming agent to form a homogeneous dispersion having a solids content of at least 5%, forming a film of said dispersion on a substantial impervious film forming surface and evaporating moisture from the film.

5. The method of forming self-supporting reconstituted tobacco sheet which comprises reducing tobacco to a finely divided state, mixing with said tobacco from about 2% to about 30%, based on the weight of tobacco, of a highly refined softwood kraft pulp having an inverted freeness as measured by the Canadian Standard Freeness Test, and an aqueous fluid containing a film forming agent to form a homogeneous dispersion having a solids content of at least 5%, forming a film of said dispersion on a substantial impervious film forming surface and evaporating moisture from the film.

6. The method of forming self-supporting reconstituted tobacco sheet which comprises reducing tobacco to a finely divided state, mixing with said tobacco from about 5% to 25%, based on the weight of tobacco, of a highly refined fibrous pulp having an inverted freeness as measured by the Canadian Standard Freeness Test, and an aqueous fluid containing a film forming agent and a cross linking agent to form a homogeneous dispersion having a solids content of at least 5% forming a film of said dispersion on a substantial impervious film forming surface and evaporating moisture from the film.

7. The method of claim 6 in which the film forming agent comprises a galactomannan.

8. The method of claim 6 in which the film forming agent comprises a galactomannan in combination with a water soluble cellulose ether.

9. A self-sustaining tobacco sheet material comprising a mixture including finely divided tobacco, from about 2% to 30% by weight based on the weight of the dry tobacco, of fibrous material obtained by dehydrating a pulp having an inverted freeness as measured by the



Canadian Standard Freeness Test, and a quantity of adhesive film forming agent sufficient to maintain said sheet material in self-sustaining condition.

10. The material of claim 9 containing additionally for the adhesive film forming agent a small but effective amount of cross linking agent.

11. The tobacco sheet of claim 9 in which the adhesive film forming material comprises a galactomannan.

12. The material of claim 9 in which the adhesive film material comprises a galactomannan in combination with a water soluble cellulose ether.

13. A self-sustaining tobacco sheet material comprising a mixture including finely divided tobacco, from about 5% to 25% by weight based on the weight of the dry tobacco, of paper fiber, said fiber obtained by dehydrating paper pulp having an inverted freeness between 50 cc. and 900 cc. as measured by the Canadian Standard Freeness Test, and a quantity of adhesive film forming agent sufficient to maintain said sheet material in self-sustaining condition.

14. A self-sustaining tobacco sheet material comprising a mixture including finely divided tobacco, 5% to 25% by weight based on the weight of the dry tobacco, of fibrous material obtained by dehydrating softwood sulfite pulp having an inverted freeness between 50 cc. and 900 cc. as measured by the Canadian Standard Freeness Test, and a quantity of adhesive film forming agent sufficient to maintain said sheet material in self-sustaining condition.

15. A self-sustaining tobacco sheet material comprising a mixture including finely divided tobacco, from about 5% to 25% by weight based on the weight of the dry tobacco, of fibrous material obtained by dehydrating hardwood kraft sulfite pulp having an inverted freeness between 50 cc. and 900 cc. as measured by the Canadian Standard Freeness Test, and a quantity of adhesive film forming agent sufficient to maintain said sheet material in self-sustaining condition.

16. A self-sustaining tobacco sheet material comprising a mixture including finely divided tobacco, from about 5% to 25% by weight based on the weight of the dry tobacco, of fibrous material obtained by dehydrating softwood kraft pulp haivng an inverted freeness between 50 cc. and 900 cc. as measured by the Canadian Standard Freeness Test, and a quantity of adhesive film forming agent sufficient to maintain said sheet material in self-sustaining condition.

17. A self-sustaining tobacco sheet material comprising a mixture including finely divided tobacco, 5% to 25% by weight based on the weight of the dry tobacco, of fibrous material obtained by dehydrating a paper pulp having an inverted freeness between 50 cc. and 900 cc. as measured by the Canadian Standard Freeness Test, and a quantity of guar gum-ethyl hydroxyethyl cellulose mixture, as adhesive film forming agent, sufficient to maintain said sheet material in self-sustaining condition.

18. A self-sustaining tobacco sheet material comprising a mixture including finely divided tobacco, from about 5% to 25% by weight based on the weight of the dry tobacco, of fibrous material obtained by dehydrating a paper pulp having an inverted freeness between 50 cc. and 900 cc. as measured by the Canadian Standard Freeness Test, a quantity of guar gum-ethyl hydroxyethyl cellulose mixture, as adhesive film forming agent, sufficient to maintain said sheet material in self-sustaining condition, and a cross linking agent.

19. In the method of forming reconstituted tobacco sheet by binding finely divided tobacco particles in an aqueous dispersion with a suitable adhesive film forming agent and vaporizing the moisture therefrom, the improvement which comprises homogeneously distributing into the sheet forming aqueous dispersion from about 2% to about 30% by weight, based on the dry weight of tobacco, of paper pulp having an inverted freeness as measured by the Canadian Standard Freeness Test.

20. In the method of forming reconstituted tobacco sheet by binding finely divided tobacco particles in an aqueous dispersion wtih a suitable adhesive film forming agent and vaporizing the moisture therefrom, the improvement which comprises homogeneously distributing into the sheet forming aqueous dispersion from about 2% to about 30% by weight, based on the dry weight of tobacco, of paper pulp having an inverted freeness between 50 cc. and 900 cc. as measured by the Canadian Standard Freeness Test.

21. In the method of forming reconstituted tobacco sheet by binding finely divided tobacco particles in an aqueous dispersion with a suitable adhesive film forming agent and vaporizing the moisture therefrom, the improvement which comprises homogeneously distributing into the sheet forming aqueous dispersion from about 2% to about 30% by weight, based on the dry weight of tobacco, of paper pulp having an inverted freeness between 50 cc. and 900 cc. as measured by the Canadian Standard Freeness Test, and a cross linking agent.

22. In the method of forming reconstituted tobacco sheet by binding finely divided tobacco particles in an aqueous dispersion with a suitable adhesive film forming agent and vaporizing the moisture therefrom, the improvement which comprises homogeneously distributing into the sheet forming aqueous dispersion from about 2% to about 30% by weight, based on the dry weight of tobacco, of softwood sulfite pulp having an inverted freeness as measured by the Canadian Standard Freeness Test.

23. In the method of forming reconstituted tobacco sheet by binding finely divided tobacco particles in an aqueous dispersion with a suitable adhesive film forming agent and vaporizing the moisture therefrom, the improvement which comprises homogeneously distributing into the sheet forming aqueous dispersion from about 2% to about 30% by weight, based on the dry weight of tobacco, of hardwood kraft pulp having an inverted freeness as measured by the Canadian Standard Freeness Test.



24. In the method of forming reconstituted tobacco sheet by binding finely divided tobacco particles in an aqueous dispersion with a suitable adhesive film forming agent and vaporizing the moisture therefrom, the improvement which comprises homogeneously distributing into the sheet forming aqueous dispersion from about 2% to about 30% by weight, based on the dry weight of tobacco, of softwood kraft pulp having an inverted freeness as measured by the Canadian Standard Freeness Test.

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# UNITED STATES PATENT OFFICE

# CERTIFICATE OF CORRECTION

Patent No. 3,125,098 March 17, 1964

Robert J. Osborne

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 4, line 3, for "heating" read -- beating --; column 6, second table, column 2, line 3 thereof, for "8.14" read -- 814 --; column 10, line 10, for "of" read -- or --; column 12, line 43, for "haivng" read -- having --.

Signed and sealed this 4th day of August 1964.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents