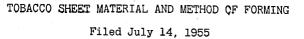
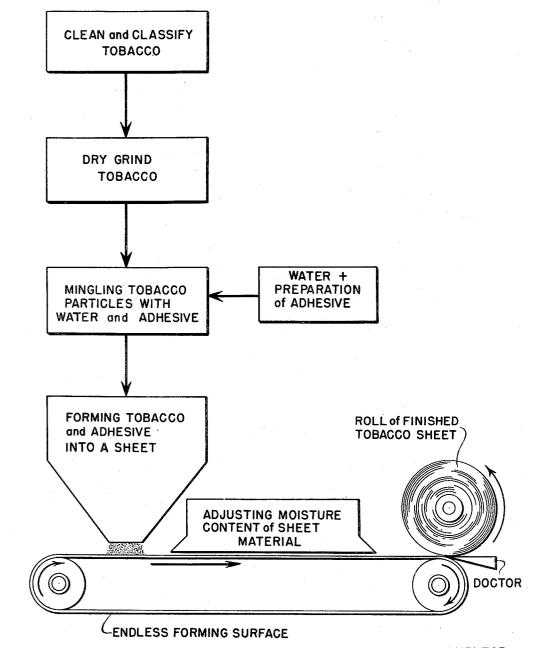
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TOBACCO SHEET MATERIAL AND METHOD OF FORMING

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application Ser. No. 477,111 filed December 22, 1954, now abandoned.

This invention relates to self supporting tobacco sheet material and to a method of forming it. This invention further relates to improved smoking articles made with 20 such a product. Particularly, the invention relates to a substantially water resistant tobacco sheet material characterized by the aroma, color, taste and burning characteristics of natural whole leaf tobacco.

Heretofore, different kinds of tobacco sheets have been 25 made by various methods. Paper making techniques have been employed in which tobacco is pulped in a large quantity of water and many of the desirable elements of natural tobacco such as flavors are leached away. Tobacco has been wet ground to a colloidal paste and then 30 cast into sheets which do not burn as well as the product of this invention. Laminated tobacco products have also been made by the application of a layer of tobacco dust to a layer of adhesive. Homogeneous tobacco sheets have been made with water soluble adhesive binders and ³⁵ homogeneous sheet materials have tobacco particles imboth wet and dry ground tobacco. Water insoluble binders which impair the smoking quality of the tobacco have been suggested.

Various materials have been proposed as film forming agents to be used in converting tobacco particles into 40forms suitable for use in manufacturing cigarettes, cigars, pipe tobacco, chewing tobacco and the like. Perhaps, the primary characteristic required of such agents is that they should not introduce any offensive odor, taste, or color into the tobacco or the tobacco smoke or alter 45 substantially the natural properties of tobacco as a smoking product.

Other considerations are also important for the practical application of these agents to tobacco sheet material. It is highly desirable that a tobacco sheet ma- 50 terial should have sufficient tensile strength and dimensional stability to withstand vigorous manipulation in the course of manufacturing and so be self supporting. It must also hold together well in smoking products such as 55 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. The tobacco should resist disintegration by moisture and have wet strength so as not to gum up upon blending, 60 casing and similar treatment as well as when used ultimately in smoking or chewing. 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.

Heretofore, no homogeneous (non-laminated) self ⁶⁵ supporting, substantially water resistant, tobacco sheet material which burns with the desirable characteristics of natural tobacco has been made. Homogeneous material is made by a suspension rather than a laminating 70 method.

Accordingly, it is an object of this invention to pro-

vide a self supporting, moisture resistant, tobacco sheet material 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, moisture resistant, tobacco sheet material which is easily worked into a smoking product and which has substantially the natural characteristics of tobacco including taste, color and 10 arema.

These and other objects and advantages of the invention are elaborated and set forth in the following description.

The advantages of the present invention are realized This application is a continuation-in-part of copending 15 by mixing with an adhesive film forming agent, which may be dispersed in water, and which is adaptable to the formation of a water resistant sheet, a quantity of finely divided tobacco, to form a viscous suspension, and forming this suspension into a dry tobacco sheet. Filler material and fibers may also be incorporated with the binder. The viscous suspension may be formed into a sheet in many different ways, such as extrusion, calendering, molding, or by applying it upon a substantially impermeable film forming surface, for example.

> The drawing illustrates schematically a typical procedure for the manufacture of tobacco sheet material according to this invention.

> In tobacco sheet material prepared according to this invention, the adhesive film forming agent serves as a matrix for dispersed tobacco particles. These particles are imbedded in the adhesive material. This material is formed from a slurry or mixture of tobacco particles and adhesive materials and shows a more homogeneous structure than laminated forms. While both laminated and bedded in adhesive agents, only the homogeneous sheets are characterized by tobacco particles both dispersed and imbedded in the adhesive matrix. For example, sheets which have only a surface coating of tobacco particles over a film or layer of adhesive which is internally free from tobacco are clearly laminated forms, whereas sheets which include tobacco particles entirely surrounded by adhesive material as well as a surface coating of tobacco are of the homogeneous type.

> The adhesive formulation may include intermingled fifibers to add strength to the tobacco sheet or optionally fibers may be omitted. Some suitable organic fibers are polysaccharides such as cellulose pulp in the form of cigarette paper pulp or glassine paper pulp. Mineral fibers are also suitable such as asbestos or glass fibers. The fibrous material is suspended with the adhesive film forming agents, preferably in a small quantity of water.

> The fibrous material and also the selected adhesive film forming agents are of a composition which when burned in the smoking article do not adversely affect the blandness, flavors, aroma or burning qualities of the to-This characteristic is described as being "combacco. patible" with tobacco.

> 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 should be between 500 and 5,000,000 centipoises. The preferred viscosity range is 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 agent is a polysaccharide and is usually water dispersible in the first step of the method of forming the sheet according to this invention. When the adhesive film forming agent is provided in the form of

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dry powder, particle sizes used in this invention are preferably similar in size to the tobacco particles and may be smaller. Cellulose glycolic acid (acid form of carboxymethyl cellulose) is a preferred film forming material since the dried cellulose glycolic acid is subsetantially initially water resistant. Hydroxyethyl carboxymethyl cellulose, viscose, and galactomannan gum dispersions as well as water insoluble algin compounds when properly formulated are other polysaccharides which can be used. Polyuronides include all uronic acid containing poly- 10 saccharides 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. Polyuronides such as pectins may also be used. In particular water insoluble pectates such as calcium 15 and magnesium pectate are valuable adhesives. Any of these materials may also be used to form laminated tobacco sheets by methods similar to those described in copending applications S. N. 124,042, S. N. 220,174 and S. N. 262,575, now respectively U. S. Patents 2,734,510 20 vention has preferably a moisture content in the range and 2,734,513 and 2,734,509, all issued February 14, 1956.

Adhesive film forming materials used in this invention may be variously distinguished as either soluble, substantially insoluble, water resistant and initially water 25 insoluble. A true solution is regarded as a complete dispersion of particles on a molecular level wherein each molecule is individually completely disassociated from other molecules. Substantially insoluble materials have such a low degree of solubility as to appear under all ordi- 30 nary conditions of tobacco use to be insoluble and these materials maintain a self supporting film coherence even when completely wetted by water, saliva or ordinary casing solutions for a practical working span of time and indeed will hold together even while completely im- 35 mersed. Water resistant materials have a wet strength between soluble materials, such as methyl cellulose, and substantially insoluble materials, such as cellulose glycolic acid. Initially water insoluble material does not form a true solution but may be applied in a gelatinous and 40dispersed condition to tobacco particles. On the other hand, initially soluble materials such as the sodium salt of carboxymethyl cellulose can be converted to ultimately insoluble forms after a tobacco sheet is formed by the application of insolubilizing agents such as acids or metal 45 salts which can convert the adhesive to an insoluble form. Initially insoluble polysaccharide adhesive material is less than freely soluble at at least one stage in the manufacture of tobacco sheet material prior to the final sheet 50 forming operation.

The film forming agent is the structural foundation of the sheet. If the film forming agent is weak or discontinuous, the sheet will crumble and disintegrate into dust when handled in tobacco machinery. When tobacco sheet material is fed from continuous rolls into 55 automatic cigar making equipment, its crimping and setting properties may be critically important. Tobacco sheet material made according to this invention produces desirable crimping and setting properties for use in auto-60 matic cigar making machines and other machinery for fabricating tobacco smoking articles.

The tobacco can be from either leaves or stems and need not be byproduct material although one of the economic advantages of the invention is the utilization of $_{65}$ otherwise useless fine quality tobacco which often crumbles from the leaves during ordinary processing.

The preferred finished sheet has a tensile strength and thickness approximately that of natural leaf tobacco. Of course, the strength and sheet thickness may be adjusted 70 for particular applications. A preferred 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. This feature may be defined 75 tions of adhesive film forming agent.

so that at least one square foot of material has this property.

The finely divided or fragmented tobacco may be prepared by grinding or by other fragmenting means. For example, dust may be used. Sheet made entirely of dry ground tobacco is a preferred 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. However, while colloidal tobacco may be used for a minor part of the blend, preferably the larger designated particle sizes are employed as these tend to give a better burning sheet.

Tobacco sheet material prepared according to this inbetween 8% and 24%. 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.

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.

Finely divided tobacco which is prepared, for example, as described above, is mingled with an adhesive formulation which may include fibers to form a slurry or a suspension. A minimal quantity of water is employed in the adhesive formulation to avoid leaching solubles from the tobacco. 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 ball mill. The exact relation of ball size and volume to total 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 (with or without fibers) may be mixed together and a slurry formed with the mixed dry particles. The product of either method may be applied to a belt, by casting or spraying for example, to form a coating film which is then dried and removed in a continuous sheet.

The viscous slurry can also be shaped and formed into a sheet by calendering, extrusion or molding, when the viscosity is very high.

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.

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 part 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. The invention is more particularly illustrated and de-

scribed by the following examples which show formula-

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Darte

EXAMPLE ONE

A preferred example of a method in accordance with the present invention of making a substantially water insoluble self supporting tobacco sheet material was carried out as follows: Tobacco was cleaned of foreign matter and comminuted to pass through an 80 mesh screen. The size reduction was conveniently carried out in a ball mill.

A preferred polysaccharide film forming agent was prepared by dissolving 2.6 pounds of sodium carboxymethyl cellulose in 174 pounds of water. To the solution 2.5 pounds of paper pulp were added and dispersed together with 0.5 pounds of glycerine and 0.375 pounds of glyoxal. The glycerine and glyoxal served respectively as a plasticizer and a cross-linking agent. A 9% HCl solution was added until the pH was between 2.6 and 2.8. This resulted in the formation of acid carboxymethyl cellulose, an active step in deliberately fixing the film forming agent in a substantially water insoluble form.

Finely divided tobacco was blended with the film forming agent to form a smooth homogeneous suspension which ranged in consistency between a syrupy viscous liquid and a firm paste. In this example, one part of dry tobacco was added to nine parts of binder dispersion.

The suspension was applied to a stainless steel belt, where it was spread out, dried, remoistened and removed as a finished sheet. The drying operation was the final step in fixing the binder in a substantially water insoluble form.

EXAMPLE TWO

Binder mixture

	raits	
Locust bean gum	. 2	
Water	. 100	3
Glycerine	. 0.6	
Glassine pulp	. 2	

The locust bean gum, a galactomannan gum derivative from legume endosperm, was sprinkled into cold water with agitation. A Cowles dissolver was found to be a 40 good agitator for this purpose, although other types of agitators also produce good results. After two hours agitation in cold water, the gum dispersion was heated to 170-200° F. with continued agitation and held above 170° F. for thirty minutes. Glycerine and glassine pulp 45were then mixed in. After cooling, 12 parts of ground tobacco dust passing through an 80 mesh screen were mixed in. The tobacco-binder suspension was then passed through a colloid mill set at low clearance, i. e. .002-.010 inches using one pass at .010 inches and a $_{50}$ second pass at .002 inch clearance. The viscosity of the final tobacco-binder suspension was 12,400 centipoises at 85° F. and the pH was 6.4. The suspension was cast on a stainless steel belt, dried and remoistened by a fine water spray to the point where a continuous sheet of to- 55 bacco could be removed from the casting belt.

EXAMPLE THREE

Binder mixture

		f
Hydroxyethyl cellulose (HEC)	3	Č
Water		
Filler paper pulp	1.5	
Glycerine		
Glyoxal		€

All materials were charged into a vessel equipped with an agitator and agitated for one hour. A Cowles dissolver is suitable for agitation purposes. Sixteen parts of finely ground cigar binder leaf scrap passing through an 80 mesh sieve were added and the tobacco-binder 70 suspension was agitated for thirty minutes. The viscosity of the tobacco-binder suspension was 6500 centipoises, and the pH was 6.0.

The tobacco binder suspension was sprayed onto a tobacco, at least a portion of which is entir moving stainless steel casting belt, dried and carefully 75 within a matrix of cellulose glycolic acid.

remoistened to permit removal of the tobacco sheet in a continuous process.

It was desirable to reduce the suspension pH to 4-5 to obtain better resistance to disintegration on soaking in water. Lower pH increases the rate of crosslinking of

the HEC by glyoxal, and thus increases water resistance. EXAMPLE FOUR

	Binder mixture
n	Parts
Ő	Sodium alginate, low viscosity (Kelgin LV) 2
	Glycerine 0.6
	Water 90
	Cigarette paper pulp fiber 2

The sodium alginate was agitated in the water until completely dissolved. To this was added a solution of 0.4 parts calcium chloride dissolved in 10 parts water. On adding the calcium chloride, lumps of gelatinous calcium alginate precipitated. As agitation continued these 20 lumps broke up and the entire mass became a uniform gel. After this gel was passed through a colloid mill at a 0.001 inch clearance it was a smooth dispersion of insoluble calcium alginate in a highly hydrated form. It is desirable to restrict the addition of calcium chloride to 25 50–90% of the stoichiometric equivalent of sodium alginate to obtain a good balance between stability of the dispersion and water resistance of the dried film. If the calcium equivalent added exceeds 80%, the dispersion tends to be lumpy and does not produce a smooth film. 30 When the added calcium equivalent is less than 50% of the sodium alginate, the dried film tends to be sensitive to water and to disintegrate on wetting. The fibers were then thoroughly mixed into the binder dispersion. Sodium pectate may be substituted conveniently for sodium alginate and propylene glycol alginate may also be used. To the binder dispersion prepared above, eight parts of ground tobacco, minus 80 mesh, were added to form a suspension. The suspension was passed through a colloid mill twice. One pass was at a clearance of 0.01 inch and the second pass was at a clearance setting of 0.002 inch. Finally, the milled tobacco-binder suspension was applied to a casting belt and treated as described above to produce continuous tobacco sheets.

There have thus been described methods of forming a novel tobacco sheet material useful in the manufacture of smoking articles. The material is resilient, substantially water insoluble, self supporting and retains the qualities of natural tobacco leaf such as aroma, flavor and color. The process is based upon the blending of finely divided tobacco and a substantially water insoluble film forming agent to form a novel tobacco sheet material.

What is claimed is:

1. Tobacco sheet material comprising finely divided tobacco, at least a portion of which is entirely dry ground, within a matrix of an initially water insoluble polysaccharide adhesive.

2. Tobacco sheet material comprising finely divided tobacco, at least a portion of which is entirely dry ground, and randomly dispersed fibers, within a matrix of sub-Parts 60 stantially water insoluble polysaccharide.

 Tobacco sheet material comprising finely divided tobacco, at least a portion of which is entirely dry ground, within a matrix of at least one adhesive material selected from the group consisting of carboxymethyl cellulose, 65 hydroxyethyl cellulose, galactomannan gums and substantially water insoluble polyuronides.

4. Tobacco sheet material comprising finely divided tobacco within a matrix of cross-linked polysaccharide adhesive.

5. Tobacco sheet material comprising finely divided tobacco within a matrix of polysaccharide adhesive and glyoxal.

6. Tobacco sheet material comprising finely divided tobacco, at least a portion of which is entirely dry ground, within a matrix of cellulose glycolic acid.

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7. Tobacco sheet material comprising finely divided tobacco, at least a portion of which is entirely dry ground, randomly dispersed non-tobacco fibers, within a matrix of cellulose glycolic acid.

8. Tobacco sheet material comprising finely divided tobacco, at least a portion of which is entirely dry ground which will pass through a 20 mesh screen, and paper pulp fiber within a matrix of cellulose glycolic acid.

9. Tobacco sheet material comprising finely divided tobacco within a matrix of glyoxal and hydroxyethyl 10 cellulose.

10. Tobacco sheet material comprising finely divided tobacco imbedded in an initially water insoluble algin compound.

11. Tobacco sheet material comprising finely divided 15 tobacco imbedded in calcium alginate.

12. Tobacco sheet material comprising finely divided tobacco imbedded in an initially water insoluble pectin derivative.

13. Tobacco sheet material comprising finely divided 20 tobacco which will pass through 20 mesh screen and cellulose fiber, within a matrix of glyoxal and hydroxy-ethyl cellulose.

14. Tobacco sheet material comprising finely divided tobacco and randomly dispersed fibers, imbedded in an ²⁵ initially water insoluble algin compound.

15. Tobacco sheet material comprising finely divided tobacco and randomly dispersed fibers, imbedded in an initially water insoluble pectic acid compound.

16. Tobacco sheet material comprising finely divided ³⁰ tobacco and randomly dispersed fibers, imbedded in an initially water insoluble polyuronide.

17. Tobacco sheet material comprising finely divided tobacco and randomly dispersed fibers, imbedded in calcium alginate.

18. Tobacco sheet material comprising finely divided tobacco in association with propylene glycol alginate.

19. The method of forming tobacco sheet material which comprises forming in a liquid a suspension of finely divided tobacco, at least a portion of which is entirely 40 dry ground, and an initially water insoluble polysaccharide adhesive, applying said suspension to a forming surface, drying said suspension to form tobacco sheet material and removing said tobacco sheet material from said forming surface. 45

20. The method of forming tobacco sheet material which comprises forming in a liquid a viscous suspension of finely divided tobacco, at least a portion of which is entirely dry ground, and an initially water insoluble polysaccharide adhesive, calendering said suspension to form a tobacco sheet and drying said tobacco sheet.

21. The method of forming tobacco sheet material which comprises forming in a liquid a viscous suspension of finely divided tobacco, at least a portion of which is entirely dry ground, and an initially water insoluble polysaccharide adhesive, extruding said suspension to form tobacco sheet material and drying said sheet material.

22. The method of forming tobacco sheet material which comprises forming in a liquid a suspension of finely divided tobacco, at least a portion of which is entirely dry ground, randomly dispersed non-tobacco fibers and an initially water insoluble polysaccharide, applying said suspension to a forming surface, drying said suspension to form tobacco sheet material and removing said tobacco sheet material from said forming surface.

23. The method of forming tobacco sheet material which comprises forming in a liquid a suspension of finely divided tobacco, at least a portion of which is entirely dry ground, randomly dispersed non-tobacco fibers and cellulose glycolic acid, applying said suspension to a forming surface, drying said suspension to form tobacco sheet material and removing said tobacco sheet material from said forming surface.

24. The method of forming tobacco sheet material which comprises adding to an aqueous suspension of cellulose glycolic acid and paper pulp fiber a quantity of finely divided tobacco, at least a portion of which is entirely dry ground, which will pass through 20 mesh screen, disposing said suspension upon an impermeable forming surface, drying said suspension to form tobacco sheet material and removing said tobacco sheet material from said forming surface.

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