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F. J. SOWA ET AL

2,485,670

METHOD FOR PRODUCING TOBACCO WEB MATERIAL

Filed June 9, 1942

FIG. 1

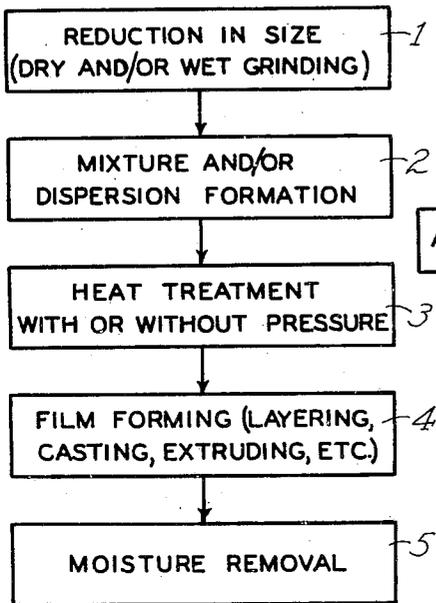


FIG. 2

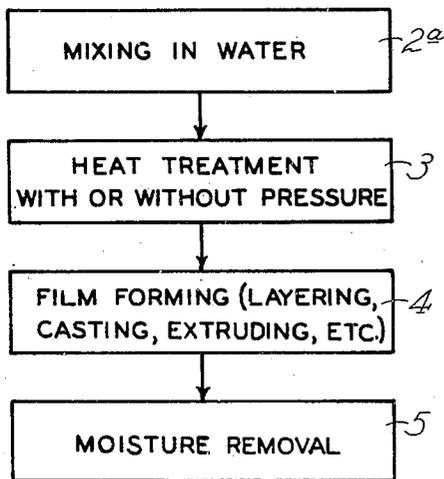


FIG. 3

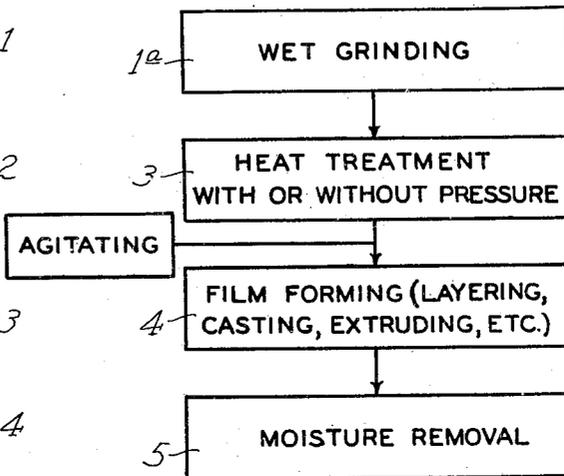


FIG. 4

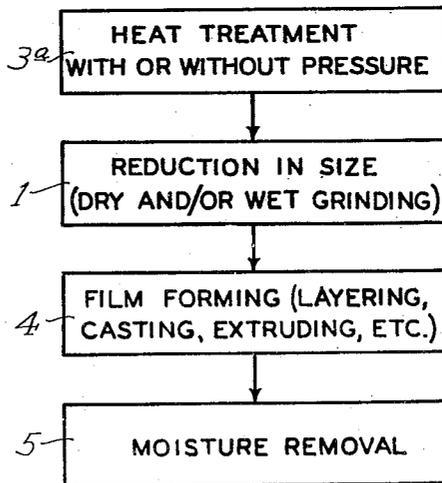
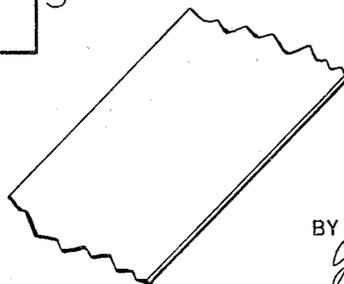


FIG. 5



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METHOD FOR PRODUCING TOBACCO WEB MATERIAL

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3 Claims. (Cl. 131-140)

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This invention relates to improved sheets, webs or films formed from natural leafy materials such as tobacco, and to the novel method of producing such sheets, webs or films.

While it is not broadly new to manufacture paper and similar sheet material having paper characteristics from tobacco or other like leafy stock, it is customary to reduce such stock to paper forming pulp by the use of chemicals according to conventional paper making technique so that the fibers of the stock will be separated from their binding material without degradation thereof and can interlock to provide the mat in the finally resulting product. These practices, however, when applied to the production of tobacco or like papers have proved unsatisfactory due to the fact that in making such paper, stems or fibers must be added and in reducing the tobacco stock in forming the necessary tobacco pulp, the processes employed destroy most of the essential and valuable characteristics of tobacco, such as color, taste and aroma, so that the resulting product is of little value as a material for smoking or in the manufacture of smoking articles.

According to our invention we make possible the formation of sheet or paper like material from natural leafy products with or without fibrous parts thereof, and retain substantially all of the desirable features and natural characteristics, such as aroma, taste and color, while at the same time the sheet material thus produced has adequate strength for processing and can be handled as if it were natural tobacco in the manufacture of smokable articles therefrom.

Our invention relates, therefore, to the formation of webs, sheets, films or filaments from natural organic leafy material, such as tobacco or combinations of one or more kinds of tobacco in the same general manner as described in Patent 2,433,877 filed by Franklin H. Wells and Frank J. Sowa, October 9, 1941 for Tobacco sheet material granted January 6, 1948, and has as its chief object, the production of improved sheets, webs, films or filaments.

It is an object of our invention to provide a method for producing sheets or webs, films or filaments from natural leafy organic material such as whole tobacco leaves, veins or stems, broken leaf fragments, or any portions thereof in desired combinations and retain in the resulting product substantially all of the desirable characteristics of tobacco while at the same time at least a part of the undesirable constituents such as tar and nicotine are reduced.

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It is an added object of our invention to produce a novel tobacco sheet material or film with or without interspersed fibers and a method for forming the same wherein tobacco, reduced to a finely divided state in a neutral fluid medium to form a slurry with or without small tobacco fibers, is subjected to heat treatment with or without pressure either before or after the tobacco has been reduced to its ultimate size, after which the slurry is formed into webs, sheets or films.

It is an additional object of our invention to produce an improved sheet material, films or filaments of tobacco which is capable of being handled or formed into smoking articles and wherein these materials have better tensile strength and more resistance to fracturing when folded than sheets or films formerly produced.

Our process contemplates the division of the tobacco into fine particles ranging in size from colloidal and larger. While we have found that best results are obtained when the tobacco is reduced to a substantially colloidal state, particles capable of passing through a sixty mesh screen under some conditions will give a satisfactory product. The fine tobacco particles are mixed preferably in a neutral aqueous medium, such as water, with or without the retention of fibrous particles so that the natural flavor and color of tobacco remain substantially unchanged in the finished product yet wherein because of the subjection of the mixture to heat or heat and pressure either before or after grinding, the aroma is improved and certain undesirable constituents of the tobacco are present in lowered quantities in the resulting product.

It is an additional object of our invention to produce thin films, webs or filaments from natural leafy organic material such as tobacco, or the like, and provide a novel method for forming the same with or without binders but wherein if binders or fillers are used the resulting tensile strength of the material produced will be increased.

Other objects of our invention will be set forth in the following description, it being understood that the above statement of objects is intended generally to explain the same without limiting it in any manner.

In the accompanying drawings which form a part of this specification, and in which like characters of reference indicate the same or like parts:

Figure 1 shows a schematic and diagrammatic illustration of a preferred method of carrying out our invention;

Figure 2 shows a similar illustration of several

related steps of the method illustrated in Figure 1;

Figure 3 shows a similar illustration of a slightly modified method;

Figure 4 shows a similar illustration of a modified method;

Figure 5 shows a sheet of the material made.

The tobacco utilized may be of any suitable kind and size, such for instance as whole leaves or portions thereof, stems or mixtures of each, or if desired, waste tobacco such as clippings, dust, chips and so forth may be used. For best results we have found that preferably the tobacco should be fairly dry, that is, contain moisture, say not in excess of five per cent, although this is not a limiting factor since obviously the tobacco can be reduced to the desired size and state if the moisture content is greater.

The sheet forming material, such as tobacco, also may be in any desired condition, as for instance, whole leaves or portions thereof, stems or mixtures of each, and since most of the natural physical characteristics of tobacco leaves as such are lost in the practice of our invention, we also contemplate the use of waste tobacco such as clippings, dust, chips, and so forth, which result from the manufacture of cigars, cigarettes and other tobacco articles.

The tobacco being processed may be reduced to proper size, as indicated at *f* or *fa* in Figures 1 and 3, respectively, either by dry grinding or wet grinding, or a combination of steps including both dry and wet grinding.

When tobacco is dry ground, a selected quantity is preferably dried to remove most of the moisture retained therein, after which it is broken up into small pieces in any suitable manner. It has been found that dry grinding proceeds faster and more satisfactorily when the moisture content is reduced below five per cent. Any suitable device may be employed for reducing the broken up dried tobacco to final size. For instance, the tobacco may be placed in a ball mill and ground to colloidal, or substantially colloidal size, or fine powder capable of passing through an eighty mesh screen. In some instances powder capable of passing through a sixty mesh screen may be used, but generally we prefer the finer sizes. This method of grinding, however, is relatively slow and the amount of time consumed varies according to the quantity of tobacco selected for grinding, the size of the mill and the balls or pebbles used therein, and the speed of operation of the mill. For optimum results the charge should not exceed the volume of voids in the mill. The amount of time required is also a function of the size and condition of the tobacco before it is placed in the mill. For instance, if the material being comminuted is small particle size at the outset, less time will be required than if the material is in the form of broken up leaves and stems.

If more rapid material grinding is preferred, a plate or disc type colloidal mill may be used, in which case a selected quantity of tobacco or other leafy material can be reduced advantageously to extremely fine size in a matter of minutes.

If the tobacco or other natural leafy material selected for formation into sheets or films, is to be wet ground as at *f* in Figures 1 and 4, and *fa* in Figure 3, a selected quantity of tobacco is preferably broken up in order to expedite its reduction, placed in a grinding device, such as a ball mill, and an aqueous medium, such as water is

added thereto to establish a suitable ratio of tobacco to water, say of 1:8 or 1:6 by weight depending upon the type of tobacco. Greater or lesser ratios can be used advantageously depending on the type of tobaccos being ground and the viscosity of the resulting slurry desired, the thinner the sheet desired the less viscous the slurry.

The mixture is ground for a period sufficient to reduce the solid material to extremely fine size, wherein some, and preferably the greater part, of the particles are in a colloidal state. It has been found that the grinding time varies over a considerable range, and that when this mixture is ground for a period ranging between six and eight hours in a 1 $\frac{1}{4}$ gallon mill, using $\frac{1}{2}$ inch balls at 45-50 revolutions per minute, advantageous results are obtained. In some instances there may remain small fibrous particles of tobacco due to the presence of veins and stems as indicated at 2 in Figure 1. This, however, is not objectionable because in most instances such minute fibrous particles appear to have a good effect upon the finished film or sheet and add to its strength.

We have also found that a good method of grinding tobacco is to wet grind whole tobacco leaves for one or more hours and then pass the grind through a screen. When such a grind has been prepared and screened, the unground portions consisting principally of stems and veins, are removed and the filtrate consisting of the better portions of the leaves can then be wet ground in a ball mill, as above described, for a suitable time as described. This forms a good slurry which can be extruded, spread out or centrifugally applied to suitable sheet forming surfaces or formed into filaments. The proportions of tobacco and water used in the above case run substantially the same as when a charge of powdered tobacco and water is introduced into a ball mill.

The material may also be subjected to both dry and wet grinding. If this type of grinding is resorted to, selected tobacco is preferably dried and then ground either in a disk type colloid mill rapidly in a few minutes or more slowly, about three hours, in a ball mill, as described hereinabove. When the material has been reduced to proper size, say capable of passing through a sixty or eighty mesh screen or smaller, it is introduced into a second grinding device, such as a ball mill or left in the first mill and mixed with a suitable quantity of fluid in order to prepare it for a wet or final grinding. Any fluid preferably neutral in nature, such as water or dilute ethyl alcohol, with which a colloidal dispersion will take place, can be used. It is important that a fluid be chosen which will have little or no deleterious effects upon the tobacco or otherwise change its normal characteristics insofar as taste, odor and color are concerned, since they are carried over into our final product and retained therein. Water forms a very satisfactory medium and is usually used in an amount suitable to form a fluid mixture. A ratio of 1:8 by weight may be used, although other suitable proportions as described hereinabove, will give the same or satisfactory results.

As in the case of dry grinding, the period of grind is dependent to the same extent upon the quantity of material being ground, as represented by the mixture of fluid, and pulverized tobacco, the capacity of the ball mill, if a ball mill is selected as the grinding mechanism, the size of balls

and speed of operation of the mill. We have found that a ball mill is a satisfactory device to use in carrying out this step of our process and that with a 1½ gallon mill containing 800 cubic centimeters of water and 100 grams of tobacco constituting the wet grind mixture or slurry, the time required to reduce the charge of powdered tobacco to fine particles or to sheet or film forming size and state ranges between one and ten hours, and that on an average of six hours has proven to be an advantageous period. If anything, the material ground for the longer period of time results in a denser sheet.

It has also been found advantageous under some conditions to make slurries having fibrous tobacco particles intermixed therewith. Such slurries are formed in any manner, such as by the addition of a quantity of fibrous tobacco particles of small size added in any required quantity dependent upon the amount of fibrous particles desired in the final sheet or film; or particles can be added by grinding together in any of the methods described above, a mixture of tobacco leaf portions and stems, and/or veins, which in the period of grinding will not be converted to colloidal size but will still retain fibrous characteristics. When this type of slurry is subjected to heat or heat at atmospheric or greater heat at elevated pressures, as described hereinafter, the finely ground and colloidal tobacco particles form a sol with the fibrous particles interspersed therethrough and when layered into films or sheets or filaments, the contained fibers add to the tensile strength of the resulting product.

A good sheet results from grinding 75 parts of dry tobacco powder with 600 parts of water in a ball mill for four hours after which time 25 parts of leafy tobacco, which contains veins, and 200 more parts of water are added to the mill making the tobacco water ratio substantially 1:8 by weight, and the whole mixture is ground for two to three hours additional depending upon the required thinness of the final product. In the second grinding phase, practically all of the leafy portions of the tobacco are converted into extremely fine or colloidal particles except the vein portions thereof which remain as very small fibrous particles which add to the tensile strength of the final product. Obviously the quantity of tobacco fibers present in a slurry produced in the above manner will vary according to the type of tobacco leaves or leafy material subjected to grinding. However, as far as the above proportions are concerned, a few fibrous particles more or less have no marked influence on the sheets or films produced from such a slurry.

If a slurry is produced by mixing together a selected quantity of ground tobacco and water in a ratio of say 1:6 or 1:8 by weight, or other proportion such as described above, an intimate suspension can be obtained by mixing the material mechanically in any known manner as desired, the particles may be dispersed throughout the liquid phase by agitating with steam, both to effect a proper dispersion and to assist in the subsequent formation of films and the like as described previously.

The mixture of tobacco and water is next subjected to heat at atmospheric pressure or to greater heat under elevated pressure as by treating the tobacco or slurry in an autoclave for a period of time sufficient to hydrate and swell the particles until substantially a hydrosol is formed, especially insofar as the finest or colloidal particles are concerned. Also, during the time of

hydration there is some hydrolysis taking place and it is from this action that it is believed that the heat accelerates the hydration of the tobacco particles and also accelerates the limited amount of hydrolysis of such materials contained in tobacco as glucosides, thereby increasing the aroma of the resulting tobacco product and greatly enhancing its desirability for use in forming smoking tobacco articles. The subjecting of the mixture to heat at atmospheric pressure or to a higher heat at elevated pressures, as indicated by numeral 3 in Figures 1, 2, and 3, is also believed to reduce somewhat the total tar and nicotine content in the smoke given off when smoking articles made from our sheet material are consumed. The resulting sheets, films or filaments also retain their general characteristic tobacco color and flavor.

Heat at atmospheric pressure or higher heat at elevated pressure treatment may be given either before the tobacco is converted into a slurry as illustrated at 3a in Figure 4 or after. If it is resorted to after the formation of the slurry, it is preferred to place a quantity of the slurry in a pressure cooker and cook it therein at a pressure of fifteen pounds per square inch for a period of three hours at 250° F. water vapor temperature. Other pressures and cooking periods can be used advantageously. For instance, if the pressure is increased then the desired result is effected in a shorter time. For instance, if sixty pounds per square inch be used, the necessary cooking will be effected in about fifteen minutes. Lower pressures are usually used and have been found to give best results. If pressure is decreased below fifteen pounds per square inch, a period of time greater than three hours is necessary. Also, if desired, the mixture can be cooked at normal atmospheric pressure by heating in a reaction flask connected to a reflux condenser for about five hours and satisfactory results are obtained. During the heat treatment of the slurries, it is advantageous to agitate the slurry or suspension as by shaking or stirring, or in any other suitable manner such as bubbling live steam into the suspension because this breaks up particles tending to conglomerate and assists in the complete hydration of the individual particles of the suspension.

The slurries treated in the above manner are formed into sheets, webs or filaments, as indicated by numeral 4, Figures 1-4 much in the manner as taught in the above referred to Wells and Sowa Patent 2,433,877, as by pouring the slurry over film forming surfaces, or spraying on a film forming surface with a spray gun, or by the use of suitable extruding mechanism, or by applying onto the interior of a rotating cylinder. The material is dried until it is in a flexible, pliable condition capable of being used in formation of binders, wrappers and as parts of cigars, and other smoking or tobacco articles. This step may be indicated generally at 5 in Figures 1-4 inclusive. It is usually best to dry the layers or sheets in a somewhat humid atmosphere since the product is somewhat more flexible than when it is dried rigid in a dry atmosphere. However, if the latter method is resorted to, humidifiers may be employed to restore the flexibility. The material produced in accordance with our invention may be bent or folded with considerable success and usually will not fracture along the line of bend, and especially is this so with respect to the films formed from slurries containing small tobacco fibers dispersed

therethrough treated with heat at atmospheric pressures or higher heat at elevated pressures. Filaments may be formed from the sheets or webs in any desired manner, such as cutting, or they may be formed by extruding the slurry through suitable conventional devices. The resulting products are dense, tough and have suitable strength, flexibility and other desirable characteristics of tobacco leaves and may be put to any similar use.

Slurries having fibrous tobacco particles intermixed therewith are also formed by addition of a quantity of fibrous tobacco particles or by grinding together in any of the methods described hereinabove a mixture of tobacco leaf portions and stems and/or veins, in which the period of grinding mentioned will not destroy the fibrous characteristics of the fibrous material although the fibers will be reduced in size and length. If the material being ground has been treated to heat at atmospheric pressure or higher heat at elevated pressures previous to being ground or if the resulting slurry is subjected to heat as described hereinabove, the finally ground and colloidal tobacco particles form a sol with the fibrous particles interspersed therewith. As described hereinabove, when this type of sol is layered into films or sheets, the contained fibers add to the strength of the resulting product. It will be appreciated that fibers may be retained in slurries if the grinding time is controlled. A good slurry results in which the leafy material is practically entirely destroyed except for minute fibrous particles of very short length, which remain because of the lack of the complete grinding of the veins or stems. When such a slurry is cast or sprayed or otherwise applied to a film forming surface, a strong product results. As mentioned above any suitable ratio of tobacco to fluid medium by weight may be used instead of 1:3 ratio described above depending upon the thickness of the final sheet or film to be formed; the less fluid medium used the thicker the sheet and vice versa using the same amount of slurry. The quantity of fibrous particles present in slurries produced, as above described, will vary according to the type of tobacco since obviously some tobaccos have more veins or stems per leaf than do others.

Peptizing agents may be added to the slurries to assist somewhat in the dispersion and filamentation of the particles prior to heat treatment. These agents are added in relatively small quantities, for instance $\frac{1}{2}$ of 1 per cent by weight. Gluconic acid, tannic acid, dilute sodium hydroxide or any other suitable material may be used advantageously.

In some instances it is advantageous to add binders, fillers and plasticizers to the tobacco slurry resulting from grinding tobacco in water in order to increase the strength of the resulting film produced before or after subjecting to heat at atmospheric pressure or greater heat at elevated pressures.

Suitable materials are methyl ether of cellulose sold under the trade-mark of "Methocel," or ethyl ether of cellulose known as "Ethocel," or gelatine or polyvinyl alcohol. These materials when used in the proper quantity, usually $\frac{1}{2}$ of 1 per cent by weight, have no objectionable effect upon the finished product insofar as handling and use in tobacco products are concerned.

While the preferred embodiments of the invention have been described, it is to be understood that the invention is not confined to the specific method steps for carrying out the method herein set forth, by way of illustration, as it is apparent that many changes and variations may be made therein, by those skilled in the art, without departing from the spirit of the invention or exceeding the scope of the appended claims.

What is claimed is:

1. The method of forming tobacco sheet material which comprises drying a selected quantity of tobacco, grinding to reduce said quantity of tobacco to small size, reducing said tobacco in water to form a slurry containing colloidal tobacco, subjecting said slurry to heat under fifteen pounds pressure per square inch at a water vapor temperature approximately 250° F. for a period ranging between three and six hours to form a hydrosol, applying said hydrosol to a film forming surface and forming a thin layer thereon, and extracting moisture from said layer to form sheet material.

2. The step in forming tobacco sheet material from a suspension of ground tobacco in a fluid medium containing colloidal tobacco which comprises subjecting said suspension to fifteen pounds pressure per square inch at a water vapor temperature not exceeding 250° F.

3. The step in forming tobacco sheet material from a colloidal dispersion of tobacco which comprises subjecting said dispersion to fifteen pounds pressure per square inch at a water vapor temperature not exceeding 250° F. for a period not exceeding six hours.

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