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TOBACCO PROCESSING AND RESULTING PRODUCT

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This invention relates to the manufacture of coherent tobacco products suitable for smoking and more particularly to such tobacco products in the form of a leaf or foil with an appearance and other physical qualities that they may be applied as the outer wrapper of cigars, cigarillos and the like in lieu of leaf tobacco ordinarily used as such wrapper.

In spite of the many processes that have been developed or suggested to convert tobacco into a continuous foil or sheet, no process has heretofore been found to yield a product that in appearance and other physical properties is a satisfactory replacement of the leaf tobacco that is used as the outer cover or wrapper of cigars of all shapes and sizes. One broad class of these prior processes treats tobacco by the techniques and with the equipment familiar to papermakers. In this class of processes, a pulp is prepared by beating or otherwise refining tobacco in water; the ultimate sheet product is fibrous in structure, is relatively stiff and springy, and in appearance and to the feel is paper-like. The acceptance of such a paper-like sheet as a cigar wrapper is limited to that type of cigar manufacturing operation in which cost reduction is considered so important that the appearance and smoking quality of the cigars produced may be sacrified.

Another broad class of prior processes involves the preparation of a binding agent in a fluid condition and the dispersion therein of finely divided or pulverized tobacco. In such case, the product is a coherent mass in which the tobacco particles are bonded or held together by a matrix of binding agent. This type of product is generally not suitable for use as cigar wrapper because of appearance and other physical limitations. Additionally, such product gives an undesirable smoke taste because of the substantial presence of binding agent.

A primary object of this invention is to convert tobacco into a continuous foil or sheet that in appearance, suppleness, elasticity and smoke taste is admirably suited for use as wrapper in the manufacture of cigars.

Another important object is to process tobacco in such manner that the tobacco develops cohesive and film-forming properties.

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

A fundamental feature of the invention involves the treatment of tobacco to develop cohesiveness from the substances naturally present therein. For this purpose, tobacco is placed in water and heated while in water in a sealed zone also containing oxygen at elevated pressure. Such processing of tobacco is basically an autoclaving operation in which the tobacco, desirably in comminuted form, is suspended in water and the aqueous suspension in an autoclave or equivalent vessel is heated while in contact with a pressurized atmosphere containing oxygen. Since oxygen is essential to the autoclaving of the aqueous suspension of tobacco, it is preferred to use a concentrated form of oxygen such as commercial-grade oxygen of at least 95% by volume purity obtained by the liquefaction and rectification of air. The partial pressure of oxygen in the autoclave, measured at room temperature (about 77° F.), is at least 70 pounds per square inch gauge (p.s.i.g.) and preferably is at least 300 p.s.i.g. Clearly, to attain such oxygen partial pressures with air, it is necessary to use air at a pressure of at least about 350 p.s.i.g. and preferably at least about 1500 p.s.i.g. Generally, the autoclaving temperature is at least 265° F. and most frequently falls in the range of about 285 to 320° F. A temperature in excess of 340° F. is rarely employed. The aqueous tobacco suspension is maintained at the desired autoclaving temperature for at least 5 minutes and more generally for a period in the range of 10 to 40 minutes.

The autoclaved aqueous tobacco suspension has sufficient cohesive property that it may be dried to a coherent form such as shreds, rods, filaments and sheets. While the tobacco may initially be in a finely divided state when charged to the autoclave, it is preferred to supply to the autoclave tobacco cut or chopped to pieces having dimensions of the order of one inch. Desirably, such chopped tobacco should have not more than 10% by weight of particles passing through a 16-mesh screen. Particularly when the autoclaved suspension or slurry contains coarse particles of tobacco, the slurry is homogenized to a substantially uniform, creamy pulp that may be spread evenly in subsequent operations involved in making a tobacco sheet or similar coherent product.

While homogenization may be performed with any known means, such as a Waring Blendor, a wet hammer mill or a Rietz disintegrator having a high-speed rotor within a stator, it is particularly advantageous to use a device which subjects the aqueous suspension to intensive shearing. Accordingly, a valve-type homogenizer operating at a pressure of the order of at least 2000 p.s.i.g. is preferred for converting the aqueous suspension into a uniform homogenized pulp. The valve-type homogenizer may optionally be operated at pressures up to 5000 p.s.i.g. and higher but in the usual case there is no technical or economic justification for operating at pressures beyond 5000 p.s.i.g. Where the tobacco subjected to autoclaving is initially in the form of large fragments or coarse particles, it is advisable to pass the autoclaved aqueous tobacco suspension through a Rietz disintegrator and then through a valve-type homogenizer.

The creamy homogenized pulp of autoclaved tobacco may be spread or cast as a film on a suitable support such as a stainless steel belt and dried to a coherent tobacco sheet. Often, this creamy pulp may have a quantity of finely divided tobacco added thereto. This further quantity of tobacco is advantageously dry-ground to particles of which more than 99% by weight pass through a 100-mesh screen and more than 25% by weight pass through a 200-mesh screen. The creamy pulp of autoclaved tobacco serves as the aqueous binding agent for the fine particles of dry-ground tobacco.

An autoclave conveniently provides the sealed zone requisite for heating an aqueous suspension of tobacco in the presence of pressurized oxygen in accordance with this invention. Advantageously, the autoclave is equipped with a moderate speed stirrer, i.e., a stirrer having a speed in the approximate range of 200 to 1500 revolutions per minute. With a stirred autoclave, the comminution of the tobacco to be treated therein may be minimized to the extent that even physically tough tobacco stems previously cut to coarse pieces of about ¼ to ½ inch in length may be used. The benefit of treating coarse particles of tobacco in the autoclave appears to lie in fiber preservation. Tobacco fiber fragments which survive the process of converting comminuted tobacco into a continuous sheet improve both the tensile strength and flexibility of that sheet. Where the tobacco entering the autoclave is in the form of coarse particles, it is usually necessary to homogenize the aqueous slurry of autoclaved tobacco to a smooth pulp which may be spread evenly in the sheet-forming operation. In such case, use of a valve-type homogenizer operating at an elevated pressure of the

order of at least 2000 p.s.i.g. is favored since homogenization is thereby achieved without complete fiber destruction or disappearance.

Where there is an opportunity to keep tobacco stems separated from tobacco laminae and both materials are to be used in making a product of this inventon, it is preferred to treat the stems in the autoclave and to prepare from the laminae the finely divided tobacco which is ultimately combined with the treated portion of the tobacco. In this manner, the components of tobacco stems which ordinarily contribute a certain harshness to the smoke of cigars and cigarettes are apparently altered because tobacco stems treated in accordance with this invention have been found by smoking experts to give a blander smoke than that from the same tobacco stems without treatment. At the same time, by withholding at least a substantial portion of the tobacco laminae from the treatment in the autoclave, the final product has the benefit of the fine aroma and smoke taste components of tobacco laminae.

As known in the tobacco art, humectants and plasticizers such as glycerol, sorbitol and various glycols are generally used in tobacco smoking products to avoid excessive drying and embrittlement of the tobacco prior to smoking. Humectants and plasticizers are desirably incorporated in the products of this invention at any point in the process after all or part of the tobacco has been treated in the autoclave. Humectants and plasticizers in an amount usually not exceeding about 10% by weight and frequently not exceeding about 5% by weight of the total tobacco content are conveniently added to the homogenized pulp of autoclaved tobacco. Humectants and plasticizers may be even added to the final product of the invention, e.g., a tobacco sheet, by applying such materials, usually in aqueous solution, as a coating or spray on the tobacco sheet.

The autoclaved tobacco pulp with or without dry-ground tobacco may be converted to a continuous tobacco sheet on a stainless steel conveyor belt equipped with a film applicator such as a reverse roll coater and with drying hoods, all as shown in U.S. Patent 2,747,583. In making tobacco sheets pursuant to this invention, it has been found particularly advantageous to heat the film or coating applied on the top side of the stainless steel belt by condensing steam on the bottom side. Such technique is illustrated in U.S. Patent 2,155,453. In this way, a very high drying speed is attained without impairment of the final product.

In most instances, the aqueous layer of autoclaved tobacco applied on the conveyor belt is dried to an adherent film that is more readily removable from the belt after reordering, i.e., conditioning with moisture. U.S. Patent 2,747,583 demonstrates reordering of the adherent film with a fine water mist from a spray nozzle which may desirably be enclosed by a humidification chamber as illustrated in U.S. Patent 2,867,220. Alternatively, a spongy roller, maintained continuously wet with water, may be disposed in rotating contact with the conveyor belt so that the dry tobacco film is moistened as it passes under the roller. As previously indicated, humectants and plasticizers like glycerol may be admixed with the water used in reordering the tobacco film.

An advantageous way of reordering the dry tobacco film is to cool the hot conveyor belt leaving the drying zone, as by spraying water against the uncoated side, and impinging steam against the tobacco film. Thus, the steam penetrates the tobacco film and condenses therein to effect the desired reordering.

The reordered tobacco film is then removed or peeled from the conveyor belt and wound up as a roll of continuous tobacco sheet as shown in U.S. Patent 2,747,583. Often, a doctor blade is used to facilitate parting of the tobacco sheet from the surface of the belt. Where the tobacco sheet is ultimately shredded for use in the manufacture of cigarettes or pipe tobacco, the sheet may be cut into pieces, such as squares with sides measuring 2 or 3 inches, as it is removed from the conveyor belt so that these pieces which are collected in suitable bins may subsequently be easily blended, prior to shredding, with tobacco leaves going into the cigarettes or pipe tobacco.

For a better understanding of the invention and its scope, illustrative embodiments are presented hereinbelow in detail. In the examples, proportions are given in parts by weight unless otherwise specified.

Example 1

Fermented Connecticut shade tobacco of 1957 was passed through a hammermill in a moist condition so as to produce a minimum of dust. To 1400 parts of hot tap water were added 75 parts of the comminuted tobacco. With this mixture occupying about 70% of the internal volume of an autoclave, substantially pure oxygen was bubbled up through the mixture for 30 seconds when the vent valve at the top of the autoclave was closed and the pressure was allowed to build up to 450 p.s.i.g. The flow of oxygen was stopped and the contents of the autoclave were stirred at about 500 r.p.m. (revolutions per minute). After about 3 minutes, oxygen was again introduced into the autoclave merely to adjust the pressure back to 450 p.s.i.g. which had dropped a little because of the solution of gaseous oxygen in the aqueous tobacco suspension. The flow of oxygen was then stopped and heat was applied so as to bring the contents of the autoclave to a temperature of 302° F. in 30 minutes. With continuous stirring, the mixture in the autoclave was maintained at the desired temperature of 302° F. for 20 minutes. A gauge pressure of 625 p.s.i.g. at 302° F. indicated that there were no leaks in the autoclave.

At the end of the 20-minute period, the mixture in the autoclave was rapidly cooled to a temperature of about 175° F. The vent valve was opened to release the gas pressure remaining in the autoclave and the aqueous tobacco suspension was transferred while still hot to a Waring Blendor where it was homogenized to a uniform creamy pulp in one minute. The extremely short homogenizing period is a significant advantage of the autoclaving treatment of this invention because in the absence of oxygen it has been found that the autoclaved tobacco requires considerably more homogenization to convert it to a creamy pulp that can be spread evenly.

The creamy pulp was spread evenly on a stainless steel band which was subsequently heated with steam condensing on the underside of the steel band in order to dry the autoclaved and homogenized tobacco coating to a coherent sheet. The dried coating on the steel surface was rehumidified and stripped as a tobacco sheet approximately 0.003 inch thick.

The tobacco sheet had a pleasing appearance and a golden brown color similar to that of some grades of wrapper leaf tobacco. The tensile strength of the tobacco sheet was 220 grams per square millimeter. When it was applied as wrapper to cigar bunches and the cigars thus made were smoked, they were found to have a very agreeable smoke taste.

Tests have shown that without oxygen in the autoclaving operation, the tobacco sheet produced has a very dark brown color making the sheet unacceptable for use as wrapper on cigars. Furthermore, the presence of oxygen in the autoclave has been shown to cause a striking reduction of nicotine in the tobacco thus treated. The tobacco used in this case had an initial nicotine content of 1.40%. After autoclaving without oxygen, the nicotine content was 1.37%. After autoclaving according to the procedure of this example, the nicotine content was only 0.45%.

A sample of shed-cured Connecticut shade tobacco of 1958 having an initial nicotine content of 0.85% contained only 0.075% after autoclaving with oxygen under the conditions specified in this example. Similarly, a nicotine value of a sample of shed-cured Maryland tobacco

was reduced from 0.96% to 0.18% by the same treatment. The nicotine content was measured on a weight basis.

Example 2

Burley tobacco stems were flaked by passage between steel rollers operating at different speeds. Seventy-five parts of this flaked stem were placed in the autoclave used in Example 1 together with 1425 parts of hot water. Oxygen was then added to this mixture under agitation to a pressure of 200 p.s.i.g. The temperature of the autoclave contents was raised to 302° F. and held there for 20 minutes. The oxygen-treated stem slurry was cooled rapidly and then passed 6 times through a valve-type homogenizer at a pressure of 3000 p.s.i.g.

Three parts of a mixture of finely ground tobacco (by-products of cigarette manufacture) per part of homogenized stem solids were admixed with the stem pulp. Glycerine was added to the extent of 3% of the dry tobacco weight and the mixture was then cast and dried on a stainless steel belt as in Example 1. The tobacco sheet after reordering was wound up as a continuous roll and subsequently cut into pieces. When 12% by weight of the sheet was mixed with a blend of cigarette filler tobaccos, shredded and the whole mixture made into cigarettes, a group of smokers could not distinguish these cigarettes from cigarettes made with the same tobacco blend containing none of the processed cigarette sheet.

This example was repeated with one variation. The autoclaved stem slurry after being rapidly cooled was decanted onto a fine mesh nylon screen (76 mesh) and allowed to drain free of liquid. The residual wet stems were then mixed with 1000 parts of water. This slurry was passed 6 times through the valve-type homogenizer at a pressure of 2000 p.s.i.g.

The tobacco sheet otherwise was made as already described. This sheet was somewhat lighter in color than the sheet first described in this example. The two sheets were equal in strength even though the modified sheet had been made with less homogenization (at 2000 p.s.i.g. instead of 3000 p.s.i.g.). The modified sheet was also found to pass the comparative cigarette smoking test.

Example 3

Seventy-five parts of shed-cured Connecticut all-purpose tobacco, hammermilled so that at least 80% by weight passed through a 16-mesh screen but not more than 20% pased through a 50-mesh screen were suspended in 1400 parts of water. Using the autoclave of Example 1, oxygen was introduced so as to attain a pressure of 450 p.s.i.g. at equilibrium. The temperature of the autoclave contents was raised during 30 minutes to 302° F. This temperature was maintained for 20 minutes and without cooling the contents of the autoclave were discharged through a 16-mesh screen into a cold receiving vessel. The oxygen pressure within the autoclave was sufficient to effect this discharge.

The treated tobacco slurry together with 12 parts of triethylene glycol was treated for 20 minutes in a high-speed shear mixer which rendered it sufficiently homogeneous for casting and drying as described in Example 1. This sheet had a tensile strength of 265 grams per square millimeter, quite sufficient for mechanical application as a binder in the manufacture of cigars. Cigars made with this sheet as a binder were of excellent smoking quality.

Example 4

Nine parts of baled-sweated Wisconsin tobacco of filler grade, wet hammermilled to pass a ¼-inch screen, were agitated together with 91 parts of water in a vessel fitted with a removable screen basket (20-mesh). After 20 minutes, the basket was lifted from the vessel, allowing the water to drain from the tobacco. The water remaining in the tank contained very fine particles of tobacco and a sludge consisting of soil and other contaminants which had adhered to the tobacco leaves. The sludge was allowed to settle for 5 minutes, whereupon the supernatant water was pumped to a hopper where it was combined with the previously drained tobacco and fed by means of a screw conveyor into a Rietz disintegrator fitted with a 0.032-inch screen.

The Rietzed slurry was adjusted to a total solid content of 7.5% by weight and placed in an agitated feed tank from where it was pumped by a metering pump at a rate of 26 to 28 parts per hour against a fluid pressure of 600 p.s.i.g. (established by a regulating valve as described below) into a continuous process autoclave consisting of the following components:

(*a*) A pressurized oxygen source supplying oxygen to the slurry line at a rate of 2.5 parts per hour at a pressure of 605 to 610 p.s i.g.

(*b*) A steam-jacketed scraped-surface heat exchanger capable of heating the oxygenated slurry to 300° F. in about 3 minutes.

(*c*) Two steam-jacketed vessels equipped with stirrers that agitated the oxygenated slurry and maintained its temperature at 300° F. for the desired holding time which in this example was 40 minutes.

(*d*) A refrigerant-jacketed scraped-surface heat exchanger which cooled the treated slurry from 300° F. to 50° F. in about 3 minutes.

(*e*) A back-pressure valve through which the slurry discharged continuously at a rate which maintained a pressure of 600 p.s.i.g. in the system.

The cool tobacco slurry which discharged freely was passed once through a valve-type homogenizer at a pressure of 5000 p.s.i.g. To 100 parts of tobacco in the homogenized slurry, were added 1 part of titanium dioxide, 6 parts of locust bean gum (in 1.5% by weight aqueous solution), 15 parts of glycerine and 0.00625 part of a water-soluble mint green dye. The blend was mixed for one hour. During the mixing, the viscosity of the mixture was reduced to 2000 centipoises at 50° F. by adding water.

The tobacco sheet was cast and dried on a stainless steel belt as in Example 1. When dry and prior to being reordered with water for take-off, a thin coating of ethylcellulose was sprayed evenly onto the tobacco sheet for the purpose of building up water resistance in the sheet. The application of this coating was made by means of a compressed air actuated spray nozzle from a 2.5% by weight ethylcellulose solution in ethanol (95%) containing 0.25% polypropylene glycol (P1200). The coating was applied to the moving sheet at such a rate that 80 to 100 milligrams of ethylcellulose were deposited per square foot of sheet. The alcohol was evaporated from the sheet by the residual heat still in the sheet and in the belt.

The sheet, a light, wrappery material in texture and color, was wound up as an endless roll of cigar wrapper. This wrapper was 0.0025 inch thick and had a tensile strength of 195 grams per square millimeter. Cigars made with this wrapper were judged to have a fine appearance and smoking quality.

Example 5

Ripe leaves of Connecticut broadleaf tobacco were primed in the field from the stalk and dehydrated by placing them in a forced-draft oven at a temperature of 130° F. for 24 hours. The weight of the leaves after drying was about 10% of the fresh weight. This dried material was hammermilled so that not more than 10% by weight passed a 16-mesh screen.

Using the autoclave of Example 1, 75 parts of the dried and hammermilled leaf were dispersed in 1000 parts of water, and oxygen was introduced to an equilibrated pressure of 350 p.s.i.g. The autoclave contents were heated in approximately 30 minutes to 302° F. and held at that temperature for 20 minutes. The autoclave was cooled, the oxygen pressure was released and the tobacco slurry withdrawn from the autoclave was treated

for 15 seconds in a Waring Blendor while still luke warm. A solution containing 7.5 parts of a commercial grade of hydroxyethylated starch dissolved in 100 parts of water by heating to 175° F. for 10 minutes was added as well as 10 parts of triethylene glycol as plasticizer and 0.56 part of titanium dioxide. After brief mixing to insure uniform distribution of all ingredients, the smooth slurry was cast, dried, coated with ethylcellulose, rehumidified and taken from the stain'ess steel belt in a manner similar to that already described in Examples 1 and 4.

The tobacco sheet had a fine golden brown appearance, making it suitable for use as a cigar wrapper. It had ample tensile strength for mechanical app'ication on the cigar machine. Cigars made with this wrapper were judged by smoking experts to have a good smoke taste.

Example 6

A blend of baled-sweated Wisconsin tobacco leaves was moistened with water to a level of about 22% by weight and passed through a chopper. The pieces of tobacco emerging from the cutter were about 1 inch in size. Eighteen parts of this chopped tobacco were added to 165 parts of water and the mixture stirred in a tank fitted with a 14-mesh screen at the bottom. After 20 minutes, the stirrer was stopped and the sludge portion allowed to settle out through the screen. A valve at the side of the tank, just above the screen was opened and the suspension of tobacco and water was run into a 20-mesh basket. The water which drained from the tobacco was combined with the sludge water which had drained through the screen in the tank. This mixture of sludge water was allowed to settle for 5 minutes; the supernatant liquid was decanted and combined with the dewatered tobacco.

The dry solid weight content of the tobacco slurry was adjusted to 7.75% by adding water to the mixture in a slurry tank. One hundred forty-six parts of this tobacco slurry were pumped into an autoclave equipped with a stirrer (about 500 revolutions per minute). The liquid charge filled approximately 70% of the autoclave. Oxygen was injected into the autoclave until the pressure was 620 p.s.i.g. when the vessel was sealed off and the contents heated to 300° F. in 21 minutes. At that temperature, the pressure was 985 p.s.i.g. and additional oxygen was introduced into the autoclave to raise the pressure to 1200 p.s.i.g. as a precaution against the loss of oxygen by leakage. The batch was held at this temperature and pressure for 40 minutes and was then cooled by circulating water through an annular jacket and through an internal coil. When the temperature of the batch had reached 180° F., the discharge valve of a dip tube in the autoclave was opened and the contents were blown out into a receiving tank. The yield of autoclaved slurry was 141 parts. Thirty-two parts (containing 2.4 parts of dry tobacco) of this slurry, 0.032 part of titanium dioxide and 8 parts of water were placed in a laboratory pulp beater (Valley Beater) with 18 pounds on the arm of the bed plate. After 1 hour of beating, the slurry temperature had decreased from 132° F. to 102° F. The beaten slurry was then cooled in a mixing vessel and 0.18 part of locust bean gum (as a 1.5% by weight aqueous solution), 0.084 part of diatomaceous earth, 0.43 part of 1,3 butylene glycol, 0.00015 part of water-soluble mint green dye (as a 0.1875% by weight aqueous solution) and 0.6 part of flue-cured tobacco powder (dry-ground to pass through a 100-mesh screen) were added. The viscosity of the mixture was adjusted to 3000 centipoises at 50° F. by the addition of water. After 30 minutes of mixing, the mixture was placed in vacuum vessel and subjected to a vacuum of 29 inches of mercury for 20 minutes.

The mixture was then cast on a stainless steel belt by spreading under a series of three doctor blades reducing the wet film thickness by stages to a final value of 0.030 inch. The film was dried in a manner identical with that described in Example 1 and sprayed with a coating of ethylcellulose plasticized with polypropylene glycol as described in Example 4.

The sheet was reordered by passing it, while still on the stainless steel belt, through a zone in which the belt was first cooled by water sprayed on the underside and then through a zone in which steam was impinged on the sheet through a series of nozzles. The cool belt condensed the steam bringing the moisture content of the sheet to 25% by weight. The reordered sheet was removed from the belt and wound up as a roll.

This tobacco sheet (0.0025 inch in thickness) had the fine golden yellow color of a desirable cigar wrapper as well as the necessary elasticity required for use as a wrapper. The tensile strength of the sheet was 325 grams per square millimeter and its elongation was 5% when tested at 75% relative humidity and 72° F. Cigars made with this wrapper and smoked by experts were characterized as having a mild aroma, a good burn and fine white ash.

Example 7

A blend of Wisconsin tobaccos was chopped and washed in a manner identical with that described in Example 6. After adjusting the total dry solids content of the slurry to 7.75% by weight by adding water, 162 parts of slurry were pumped to the stirred autoclave of Example 6. Oxygen was fed into the vessel up to a pressure of 620 p.s.i.g. and the autoclave contents were then heated in 22 minutes to 325° F. The pressure was 1125 p.s.i.g. at this temperature. Oxygen was again introduced to raise the pressure to 1200 p.s.i.g. At that pressure and at 325° F., the batch was held for 20 minutes when cooling was started. When the batch temperature was 180° F., the contents of the autoclave were passed through a Rietz disintegrator fitted with a $\frac{3}{32}$-inch screen and cooled in a jacketed tank to a temperature of 65° F. The yield of treated slurry was 157 parts.

Separately, 10 parts of cut stems from Pennsylvania tobacco which previously had been flaked as described in Example 2 were mixed with 135 parts of water. This mixture was placed in the stirred autoclave. Oxygen was introduced until the pressure was 350 p.s.i.g. when the vessel was sealed and the contents heated. The temperature was brought to 300° F. in 22 minutes and held there for 10 minutes, the pressure at this temperature being 585 p.s.i.g. The contents were then cooled to 125° F. and the pressure reduced, by venting some gas, to 75 p.s.i.g. Air at 75 p.s.i.g. was introduced while the contents of the autoclave were blown out through a dip tube in the vessel.

The stem slurry was placed in a basket centrifuge and alternately spun and washed until the effluent ran practically colorless. The original 10 parts of stem yielded 35 parts of wet "cake" having a solids content of 14% by weight.

A little over 6 parts of the wet "cake" (containing 0.88 part of dry stem solids) was added to 44 parts of water and mixture was beaten in a laboratory Valley Beater for 50 minutes using a 12 pound weight on the bedplate arm. The beaten slurry was dewatered to a dry solids content of about 9% by weight.

To 65 parts (containing 4.6 parts of tobacco) of the autoclaved Wisconsin tobacco slurry prepared as described, 5.8 parts of the dewatered beaten stems (containing 0.53 part of dry solids) and 10 parts of water were added. While the mixture was agitated, 0.116 part of titanium dioxide was added. The mixture was then passed once through a valve-type homogenizer at a pressure of 3000 p.s.i.g. To the homogenized mixture were added 0.18 part of diatomaceous earth, 27.6 parts of a 1.4% by weight aqueous solution of hydroxyethylcellulose (viscosity of 4400 centipoises in a 2% by weight solution at 20° C.), 0.775 part of triethylene glycol, 0.256 part of a 0.1875% by weight aqueous solution of a water-soluble mint green dye and 1.28 parts of dry-ground Connecticut shade tobacco passing through a 100-mesh screen.

After the mass was thoroughly mixed for 20 minutes, it was deaerated in a vacuum chamber, and cast and dried on a stainless steel belt as described in Example 6. To the dried sheet was applied a sprayed coating of 75 milligrams per square foot of ethylcellulose (100 centipoise grade, 49% ethoxyl content) and 9 milligrams per square foot of polypropylene glycol. This was accomplished by pumping the coating materials, dissolved in a mixture of 65 parts of toluene and 35 parts of methyl ethyl ketone at a concentration of 2.5% by weight based on the ethylcellulose, to a triple spray arrangement consisting of three rotating bells (each 6-inch diameter) located diagonally across and along the moving stainless steel belt at a height of 9 inches above the belt. The bells were charged with 90,000 volts of electricity and the belt was grounded. In this way the spray was atomized by the electrically charged rotating bells and attracted uniformly to the surface of the sheet. The solvent was flashed off by the residual heat in the warm belt.

The coated sheet was reordered by steam as described in Example 6, removed from the belt and wound up. The sheet had a tensile strength of 350 grams per square millimeter and an elongation of 6%. It had excellent color and texture and when applied as a wrapper to a cigar and smoked, it was judged to have a superior taste and good burn.

Example 8

Five parts of fully bleached, softwood sulphate pulp were dispersed into 95 parts of water at 120° F. in a fiber pulping tank. Fifty parts of this pulp and 50 parts of water were placed in a pilot-plant size beater (Cycle Beater) having a movable roll bar and a fixed bedplate. The pulp was beaten for a 4-hour period during which the weights on the roll bar arm were increased in stages from 20 to 70 pounds. At the end of this period, a sample of pulp removed from the beater showed a beating degree of 95 as measured in a Schopper-Riegler freeness tester. After beating, the free water was decanted from the fiber slurry. To 145 parts of autoclaved slurry containing 10.3 parts of Wisconsin tobacco blend prepared as in Example 6 except that the slurry had been passed through the Rietz disintegrator after autoclaving as described in Example 7, was added 0.31 part of titanium dioxide. After mixing, the slurry was passed once through a valve-type homogenizer at 3000 p.s.i.g.

To the homogenized slurry were added 2.7 parts of a 5.8% by weight (0.157 part of solids) slurry of cellulose fibers, prepared as just described, 0.356 part of diatomaceous earth, 54.4 parts of a 1.4% by weight aqueous solution of guar gum, 1.7 parts hydroxypropyl glycerine, 0.51 part of a 0.1875% aqueous solution of water-soluble mint green dye, and 2.54 parts of a blend of dry-ground Wisconsin tobaccos. The mass was mixed for 30 minutes, deaerated by pumping it through a perforated plate into an evacuated chamber and cast, dried, coated, reordered and wound up as described in Example 7.

The tobacco sheet had a desirable greenish yellow color suitable for a cigar wrapper. When measured at 72° F. and 75% relative humidity, the tensile strength was 305 grams per square millimeter and the elongation was 6.3%. When tested as wrapper on a cigar, the sheet was rated good in smoking quality.

Example 9

A blend of Wisconsin tobaccos was brought to 20% by weight moisture content, chopped in a guillotine cutter to pieces of about 1.5 inches in size. The cut tobacco was then passed through a thrashing machine which separated the stems from the leaf blade. The final pieces of leaf blade ranged in size from about 0.5 to 1.5 inches while the stems were mostly 1 inch long.

The blade portion was washed as described in Example 6 and fed into the stirred autoclave of that example as a slurry with a solids content of 7.75% by weight. The vessel was charged with oxygen to a pressure of 620 p.s.i.g. and the contents were then heated in 15 minutes to 280° F. at which temperature the pressure was increased to 1200 p.s.i.g. by introducing additional oxygen. After 10 minutes, the autoclave contents were cooled to 180° F. and passed through the Rietz disintegrator and further cooled to 65° F. To 50 parts of this slurry was added 0.051 part of titanium dioxide and the mixture was passed through the valve-type homogenizer once at 2000 p.s.i.g.

Similarly, a batch of tobacco stems was washed and autoclaved, but held at a temperature of 315° F. for 30 minutes at a pressure of 1200 p.s.i.g. After cooling to 180° F., this batch was passed through the Rietz disintegrator and cooled to 65° F. After adding 0.051 part of titanium dioxide to 50 parts of this stem slurry, the mixture was passed through the valve-type homogenizer three times at a pressure of 5000 p.s.i.g.

Ten parts of the homogenized stem slurry were added to 30 parts of the homogenized blade slurry and with mixing there were further added 15 parts of a 1.5% by weight aqueous solution of locust bean gum, 0.105 part of diatomaceous earth, 0.45 part of triethylene glycol, 0.106 part of water-soluble mint green dye as a 0.1875% by weight aqueous solution, and 0.56 part of a blend of dry-ground whole leaves of Wisconsin tobacco. After mixing for 30 minutes, the mass was deaerated, cast, dried, coated, reordered and wound up all as described in Example 8.

The tobacco sheet was most suitable as a cigar wrapper having fine texture and color as well as a tensile strength of 340 grams per square millimeter. Cigars made with this wrapper were found very pleasing by discriminating smokers.

The foregoing examples are illustrative of the many possible variations and modifications of the invention. The fundamental step of treating an aqueous suspension of tobacco at elevated temperature and in the presence of an elevated partial pressure of oxygen is a valuable technique in developing binding power or cohesivity in the tobacco to an extent that it becomes possible to make a wide range of tobacco sheets and like products having different physical properties while containing no binding agent from an extraneous source or only a restricted proportion thereof. Experience has shown that the presence of oxygen in the autoclaving operation is beneficial in two important respects: it facilitates the development of binding power in the treated tobacco and it prevents the darkening of the tobacco. If the aqueous tobacco suspension is autoclaved in the absence of oxygen, higher temperatures are required to develop the desired binding power and at the same time the tobacco becomes disagreeably darkened. Still another advantageous result of the use of oxygen in the autoclave is that the treated tobacco suspension requires less homogenization to prepare a smooth, creamy pulp of the type that is preferred for casting a thin continuous film on a supporting surface like a stainless steel belt. In other words, while the autoclaved tobacco of this invention may, for example, be passed once through a valve-type homogenizer operating at a pressure of 3000 p.s.i.g., the same tobacco autoclaved in the absence of oxygen would require several passes through the same homogenizer to achieve comparable smoothness and creaminess in the homogenized pulp.

As known, in a valve-type homogenizer the aqueous suspension of solid particles at the chosen elevated pressure enters a controlled microscopic clearance between the homogenizing valve and valve seat. At this point the high pressure is instantaneously changed to high velocity so that the material passing through the clearance at extremely high velocity is subjected to high shear, extreme and intense turbulence, cavitation and impact. The impact action occurs as the material leaves the clearance and impinges on the impact ring surrounding the valve clearance, Thus, in a valve-type homogenizer, the aqueous

suspension of solid particles is subjected to pressurization and intensive shearing homogenization.

The claims should not be interpreted in any restrictive sense other than that imposed by the limitations recited within the claims.

What is claimed is:

1. The improved process of manufacturing a coherent tobacco product adapted for smoking, which comprises placing tobacco in water, agitating and heating the aqueous suspension of tobacco to a temperature of at least 265° F. for at least 5 minutes while in contact with an atmosphere having an oxygen partial pressure of at least about 70 p.s.i.g. measured at room temperature, and converting the thus treated aqueous suspension of tobacco into said coherent tobacco product.

2. The process of claim 1 wherein said treated aqueous suspension of tobacco is subjected to pressurization and intensive shearing homogenization.

3. The process of claim 2 wherein said treated aqueous suspension of tobacco after having been subjected to pressurization and intensive shearing homogenization is combined with tobacco dry-ground to a powder passing through a 100-mesh screen in the conversion of said treated aqueous suspension into said coherent tobacco product.

4. The process of claim 1 wherein said treated aqueous suspension of tobacco is combined with tobacco dry-ground to a powder passing through a 100-mesh screen in the conversion of said treated aqueous suspension into said coherent tobacco product.

5. The process of claim 1 wherein the heating of the aqueous suspension of tobacco is to a temperature in the range of about 285 to 320° F., and the oxygen partial pressure is at least about 300 p.s.i.g.

6. The process of claim 5 wherein the heating of the aqueous suspension of tobacco at the temperature in said range is for a period in the rage of 10 to 40 minutes.

7. The process of claim 6 wherein said treated aqueous suspension of tobacco is subjected to pressurization and intensive shearing homogenization.

8. The process of claim 7 wherein said treated aqueous suspension of tobacco after having been subjected to pressurization and intensive shearing homogenization is combined with tobacco dry-ground to a powder passing through a 100-mesh screen in the conversion of said treated aqueous suspension into said coherent tobacco product.

9. The process of claim 5 wherein the tobacco is divided into substantially a lamina fraction and a stem fraction, said lamina fraction and said stem fraction are individually heated as an aqueous suspension, and thereafter the thus individually treated aqueous suspensions are combined in the conversion thereof into said coherent tobacco product.

10. The process of claim 9 wherein both treated aqueous suspensions are subjected to pressurization and intensive shearing homogenization.

11. The process of claim 10 wherein both treated aqueous suspensions after having been subjected to pressurization and intensive shearing homogenization are combined with tobacco dry-ground to a powder passing through a 100-mesh screen in the conversion of both treated aqueous suspensions into said coherent tobacco product.

12. The process of claim 9 wherein said treated aqueous suspension of the stem fraction is subjected to separation for the substantial elimination of the aqueous portion thereof, and thereafter the residual stem solids portion is combined with the treated aqueous suspension of the lamina fraction.

13. A coherent tobacco smoking product made of comminuted tobacco comprising at least a substantial portion of said tobacco having been heated as an aqueous suspension to a temperature of at least 265° F. for at least 5 minutes while in contact with an atmosphere having an oxygen partial pressure of at least about 70 p.s.i.g. measured at room temperature, the thus treated portion having a materially lower nicotine content than the original nicotine content of the tobacco in said treated portion.

14. The tobacco smoking product of claim 13 wherein the substantial portion of said tobacco that has been heated was originally unfermented tobacco.

15. The tobacco smoking product of claim 13 wherein a portion of the comminuted tobacco is a dry-ground powder passing through a 100-mesh screen.

16. The tobacco smoking product of claim 13 wherein said product is in the form of a thin sheet having a sprayed coating of ethylcellulose amounting to not more than about 100 milligrams per square foot of said sheet.

17. An improved aqueous binding agent for binding tobacco particles together in a continuous and coherent form, which comprises an aqueous suspension of tobacco that has been heated to a temperature in the range of about 285 to 320° F. for a period in the range of about 10 to 40 minutes while in contact with an atmosphere having an oxygen partial pressure of at least about 300 p.s.i.g. measured at room temperature.

18. The binding agent of claim 17 wherein the aqueous suspension of tobacco that has been heated has also been subjected to pressurization of at least about 2000 p.s.i.g. and intensive shearing homogenization to yield a smooth, creamy mass.

19. The binding agent of claim 17 wherein the tobacco that has been heated as an aqueous suspension comprised predominantly tobacco stems.

20. The binding agent of claim 19 wherein the aqueous suspension of tobacco that has been heated has also been subjected to pressurization of at least about 200 p.s.i.g. and intensive shearing homogenization to yield a smooth, creamy mass.

References Cited in the file of this patent

UNITED STATES PATENTS

214,391	Hornbostel	Apr. 15, 1879
2,708,441	Viglione	May 17, 1955

FOREIGN PATENTS

372,383	France	Feb. 18, 1907
1,504	Great Britain	1879

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,076,729 February 5, 1963

Paul W. Garbo

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 12, line 50, for "200 p.s.i.g." read -- 2000 p.s.i.g. --.

Signed and sealed this 27th day of August 1963.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

DAVID L. LADD
Commissioner of Patents