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UNDERLINED TEXT HAS BEEN
ADDED TO DISCLOSURE OF
APPLICATION PM-1522 (WHICH IS NON-UNDERLINED TEXT)

PROCESS AND APPARATUS FOR IMPREGNATION
AND EXPANSION OF TOBACCO

- 5 [CURRENT PLAN IS TO FILE THIS AS A STAND-ALONE
APPLICATION, MAYBE LATER AMENDING TO CROSS-REFERENCE AS
A CIP]

Background Of The Invention

This invention relates to a process for
10 expanding the volume of tobacco and an apparatus for
carrying out the process. More particularly, this
invention relates to expanding tobacco using carbon
dioxide.

The tobacco art has long recognized the
15 desirability of expanding tobacco to increase the bulk
or volume of tobacco. There have been various reasons
for expanding tobacco. One of the early purposes for
expanding tobacco involved making up the loss of weight
caused by the tobacco curing process. Another purpose
20 was to improve the smoking characteristics of
particular tobacco components, such as tobacco stems.
It has also been desired to increase the filling power
of tobacco so that a smaller amount of tobacco would be
required to produce a smoking product, such as a
25 cigarette, which would have the same firmness and yet
would deliver lower tar and nicotine than a comparable
smoking product made of non-expanded tobacco having a
more dense tobacco filler.

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Various methods have been proposed for expanding tobacco, including the impregnation of tobacco with a gas under pressure and the subsequent release of pressure, whereby the gas causes expansion of the tobacco cells to increase the volume of the treated tobacco. Other methods which have been employed or suggested have included the treatment of tobacco with various liquids, such as water or relatively volatile organic or inorganic liquids, to impregnate the tobacco with the same, after which the liquids are driven off to expand the tobacco. Additional methods which have been suggested have included the treatment of tobacco with solid materials which, when heated, decompose to produce gases which serve to expand the tobacco. Other methods include the treatment of tobacco with gas-containing liquids, such as carbon dioxide-containing water, under pressure to incorporate the gas in the tobacco and when the impregnated tobacco is heated or the ambient pressure reduced the tobacco expands. Additional techniques have been developed for expanding tobacco which involve the treatment of tobacco with gases which react to form solid chemical reaction products within the tobacco, which solid reaction products may then decompose by heat to produce gases within the tobacco which cause expansion of tobacco upon their release. More specifically:

U.S. Patent No. 1,789,435 describes a method and apparatus for expanding the volume of tobacco in order to make up the loss of volume caused in curing tobacco leaf. To accomplish this object, the cured and conditioned tobacco is contacted with a gas, which may be air, carbon dioxide or steam under pressure and the pressure is then relieved, the tobacco tends to expand. The patent states that the volume of the tobacco may,

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by that process, be increased to the extent of about 5-15%.

U.S. Patent No. 3,771,533, commonly assigned herewith, involves a treatment of tobacco with carbon dioxide and ammonia gases, whereby the tobacco is saturated with these gases and ammonium carbamate is formed in situ. The ammonium carbamate is thereafter decomposed by heat to release the gases within the tobacco cells and to cause expansion of the tobacco.

U.S. Patent No. 4,258,729, commonly assigned herewith, describes a method for expanding the volume of tobacco in which the tobacco is impregnated with gaseous carbon dioxide under conditions such that the carbon dioxide remains substantially in the gaseous state. Pre-cooling the tobacco prior to the impregnation step or cooling the tobacco bed by external means during impregnation is limited to avoid condensing the carbon dioxide to any significant degree.

U.S. Patent No. 4,235,250, commonly assigned herewith, describes a method for expanding the volume of tobacco in which the tobacco is impregnated with gaseous carbon dioxide under conditions such that the carbon dioxide remains substantially in the gaseous state. During depressurization some of the carbon dioxide is converted to a partially condensed state within the tobacco. That patent teaches that the carbon dioxide enthalpy is controlled in such a manner to minimize carbon dioxide condensation.

U.S. Patent No. Re. 32,013, commonly assigned herewith, describes a method and apparatus for expanding the volume of tobacco in which the tobacco is impregnated with liquid carbon dioxide, converting the liquid carbon dioxide to solid carbon dioxide in situ,

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and then causing the solid carbon dioxide to vaporize and expand the tobacco.

Copending and commonly-assigned U.S. Patent application 07/717,064, filed June 18, 1991, discloses
5 a process for impregnating tobacco with carbon dioxide and then expanding the tobacco. That disclosed process includes steps of contacting tobacco with gaseous carbon dioxide and controlling process conditions to cause a controlled amount of carbon dioxide to condense
10 on the tobacco. The condensed carbon dioxide is vaporized during the process, aiding in cooling the tobacco.

The present improvement invention achieves benefits not fully realized by the invention of the
15 application identified above, nor by any known prior tobacco expansion process.

Prior processes for impregnating tobacco using gaseous carbon dioxide without controlled condensation cannot achieve sufficient cooling of a
20 high bulk density tobacco bed because cooling is provided only by gas expansion. As the packing density of the tobacco bed increases, the mass of tobacco to be cooled increases and the volume or void space remaining in the tobacco bed and available for cooling gas
25 decreases. Without sufficient cooling, an acceptable pre-expansion stability of the impregnated tobacco cannot be achieved.

Typically, a loosely filled tobacco bed exhibits a tobacco bulk density gradient with a higher
30 bulk density toward the bottom due to the compressing effect of the weight of the column of tobacco. Tobacco expansion processes using gaseous carbon dioxide and loosely filled tobacco beds of relatively low bulk density result in non-uniform cooling of the tobacco

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and thus non-uniform stability and expansion of the tobacco unless special cooling measures are adopted.

As a result,
The packing density at the bottom of a deep tobacco bed will be the limiting factor in a gas-only process, because the bottom of a deep bed will have too great a packing density to be efficiently cooled by gas expansion cooling. Therefore, tobacco expansion processes using gaseous carbon dioxide are limited to relatively small or shallow tobacco beds. While such beds have been used for experimental development, they are not commercially practical.

Summary Of The Invention

The present process employing saturated carbon dioxide gas in combination with a controlled amount of liquid carbon dioxide, as described below, has been found to overcome the disadvantages of the prior art processes and provides an improved method for expanding tobacco. The moisture content of the tobacco to be expanded is carefully controlled prior to contact with the saturated carbon dioxide gas. The temperature of the tobacco is carefully controlled throughout the impregnation process. Saturated carbon dioxide gas is allowed to thoroughly impregnate the tobacco, preferably under conditions such that a controlled amount of the carbon dioxide condenses on the tobacco. After the impregnation has been completed, the elevated pressure is reduced, thereby cooling the tobacco to the desired exit temperature. Cooling of the tobacco is due to both the expansion of the carbon dioxide gas and the evaporation of the condensed liquid carbon dioxide from the tobacco. The resulting carbon dioxide-containing tobacco is then subjected to conditions of temperature and pressure, preferably rapid heating at atmospheric pressure, which result in the expansion of

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the carbon dioxide impregnant and the consequent expansion of the tobacco to provide a tobacco of lower density and increased volume.

Tobacco impregnated according to the present invention may be expanded using less energy, e.g., a significantly lower temperature gas stream may be used at a comparable residence time, than tobacco impregnated under conditions where liquid carbon dioxide is used.

In addition, the present invention affords greater control of the chemical and flavor components, e.g., reducing sugars and alkaloids, in the final tobacco product by allowing expansion to be carried out over a greater temperature range than was practical in the past.

Furthermore, impregnating and expanding tobacco according to the present invention can achieve a greater process throughput than processes using gaseous carbon dioxide under conditions that do not result in condensation of the carbon dioxide prior to venting. According to the present invention, evaporation of condensed carbon dioxide provides sufficient cooling so that even tobacco of a substantially high bulk density [or packing density] may be effectively impregnated and expanded. In order to provide a tobacco bed ^{HAVING BOTH} of a desirable high density and a more uniform density throughout the bed, the tobacco may be compressed or compacted before it is impregnated with carbon dioxide gas. Thereby, the mass throughput of the process is increased.

The process throughput ^{may be} also be increased because the cycle time of the impregnating process may be reduced according to the invention. Because a reduced quantity of gas is required according to the invention, pressurization and venting steps can be

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ADD ENVIRONMENTAL
BENEFITS

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or rapid gas vent

performed more quickly. Also, the compacted tobacco bed is less likely than a loose bed to be percolated or moved by a rapid gas fill. Additionally, less heat of compression develops because a smaller volume of gas is compressed. Additionally, the evaporation of condensed carbon dioxide removes heat of compression that does develop. Therefore, no separate post-compression cooling step is required to remove heat.

The increased process throughput due to increased mass throughput and decreased cycle time achieves greater cost economy in production, or allows capital cost savings by reducing the size of the process equipment. Furthermore, a small batch, short cycle process operates as an essentially continuous process carried out in a preferred apparatus as described below.

What is
this right?

Detailed Description Of The Invention

The present invention relates broadly to a process for expanding tobacco employing a readily available, relatively inexpensive, non-combustible and non-toxic expansion agent. More particularly, the present invention relates to the production of an expanded tobacco product of substantially reduced density and increased filling power, produced by impregnating tobacco under pressure with saturated gaseous carbon dioxide and a controlled amount of condensed liquid carbon dioxide, rapidly releasing the pressure, and then causing the tobacco to expand. Expansion may be accomplished by subjecting the impregnated tobacco to heat, radiant energy or similar energy generating conditions which will cause the carbon dioxide impregnant to rapidly expand.

To carry out the process of the present invention, one may treat either whole cured tobacco

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leaf, tobacco in cut or chopped form, or selected parts of tobacco such as tobacco stems or possibly even reconstituted tobacco. In comminuted form, the tobacco to be impregnated preferably has a particle size of
5 from about 6 mesh to about 100 mesh, more preferably the tobacco has a particle size not less than about 30 mesh. As used herein, mesh refers to United States standard sieve and those values reflect the ability of
10 more than 95% of the particles of a given size to pass through a screen of a given mesh value.

As used herein, % moisture may be considered equivalent to oven-volatiles content (OV) since not more than about 0.9% of tobacco weight is volatiles other than water. Oven volatiles determination is a
15 simple measurement of tobacco weight loss after exposure for 3 hours in a circulating air oven controlled at 212°F. The weight loss as percentage of initial weight is oven-volatiles content.

Brief Description Of The Drawings

20 The above and other objects and advantages of the invention will be apparent upon consideration of the following detailed description and representative examples, taken in conjunction with the accompanying drawings, in which like run designations refer to like
25 runs throughout, and in which:

FIG. 1 is a standard temperature-entropy diagram for carbon dioxide;

FIG. 2 is a simplified block diagram of a process for expanding tobacco incorporating one form of
30 the present invention;

FIG. 2A is a variant of FIG. 2 showing a process for compressing, impregnating and expanding tobacco according to one embodiment of the present improvement invention:

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FIG. 3 is a plot of weight percent carbon dioxide evolved from tobacco impregnated at 250 psia and -18°C versus post-impregnation time for tobacco with an OV content of about 12%, 14%, 16.2%, and 20%;

5 FIG. 4 is a plot of weight percent carbon dioxide retained in the tobacco versus post-vent time for three different OV tobaccos;

FIG. 5 is a plot of expanded tobacco equilibrium CV versus hold-time before expansion for
10 tobacco with an OV content of about 12% and about 21%;

FIG. 6 is a plot of expanded tobacco specific volume versus hold-time before expansion for tobacco with an OV content of about 12% and about 21%;

FIG. 7 is a plot of expanded tobacco
15 equilibrium CV versus expansion tower exit OV content;

FIG. 8 is a plot of percent reduction in tobacco reducing sugars versus expansion tower exit OV content;

FIG. 9 is a plot of percent reduction in
20 tobacco alkaloids versus expansion tower exit OV content;

FIG. 10 is a schematic diagram of an impregnation vessel showing the tobacco temperature at various points throughout the tobacco bed after
25 venting;

FIG. 11 is a plot of expanded tobacco specific volume versus hold-time after impregnation prior to expansion;

FIG. 12 is a plot of expanded tobacco
30 equilibrium CV versus hold-time after impregnation prior to expansion;

FIG. 13 is a plot of tobacco temperature versus tobacco OV showing the amount of pre-cooling required to achieve adequate stability (e.g., about

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1 hour post-vent hold before expansion) for tobacco impregnated at 800 psig;

FIG. 14 is a schematic top view of an embodiment of an apparatus for carrying out a short cycle impregnation process on high bulk density tobacco according to the invention:

FIG. 15 is a schematic cylindrical ^{sectional} projection of the apparatus of FIG. 14:

FIG. 16 is an enlarged vertical section through the pressure vessel of FIG. 15, taken along a plane perpendicular to the viewing direction of FIG. 15:

FIG. 17 is a plot of tobacco temperature versus distance from the top of a pressure vessel showing temperature data for a process according to the invention and for an all gas impregnation process, with tobacco bed packing densities of 5.1 and 11 lbs./cu.ft.:

FIG. 18 is a plot of tobacco temperature versus pressure in a pressure vessel during venting, showing venting progressing from the right to the left with temperature data for a process according to the invention and for an all gas impregnation process, with tobacco bed packing densities of 5.1 and 11 lbs./cu.ft. and

FIG. 19 is a plot of tobacco temperature versus time during venting, showing T_g and T_b for tobacco bed packing density of 8.6 lbs./cu.ft.

Generally, the tobacco to be treated will have an OV content of at least about 12% and less than about 21%, although tobacco having a higher or lower OV content may be successfully impregnated according to the present invention. Preferably, the tobacco to be treated will have an OV content of about 13% to about 15%. Below about 12% OV, tobacco is too easily broken, resulting in a large amount of tobacco fines. Above

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about 21% OV, excessive amounts of pre-cooling are needed to achieve acceptable stability and a very low post-vent temperature is required, resulting in a brittle tobacco which is easily broken.

5 The tobacco to be expanded will generally be placed in a pressure vessel in such a manner that it can be suitably contacted by carbon dioxide. For example, a wire mesh belt or platform may be used to support the tobacco in the vessel.

10 In a further improvement according to the present invention, tobacco with a high bulk density may be processed. In order to achieve a desirable high packing density ^{or} a more uniform density throughout a tobacco bed, or both a high bulk density and a more
15 uniform tobacco bed, the tobacco may be compacted or compressed before it is impregnated with carbon dioxide. The tobacco may be compacted before it is placed in the pressure vessel or within the pressure vessel or both, so that the resultant packing density
20 of the tobacco in the pressure vessel is essentially uniform and substantially greater than the packing density of a typical loose fill tobacco.

For a batch impregnation process, the tobacco-containing pressure vessel is preferably purged
25 with carbon dioxide gas, the purging operation generally taking from about 1 minute to about 4 minutes. In the preferred embodiment involving a high bulk density bed of tobacco, void space is minimized and purge requirements are reduced. The example
30 described in detail below with reference to FIGS. 14-XXX operates with only a 5 second purge step. The purging step may be eliminated without detriment to the final product. The benefits of purging are the removal of gases that may interfere with carbon dioxide

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recovery and the removal of foreign gases that may interfere with full penetration of the carbon dioxide.

The gaseous carbon dioxide which is employed in the process of this invention will generally be
5 obtained from a supply tank where it is maintained in saturated liquid form at a pressure of from about 400 psig to about 1050 psig. The supply tank may be fed with recompressed gaseous carbon dioxide vented from the pressure vessel. Additional carbon dioxide may be
10 obtained from a storage vessel where it is maintained in liquid form generally at a pressure of from about 215 psig to about 305 psig and temperatures of from about -20°F to about 0°F. The liquid carbon dioxide from the storage vessel may be mixed with the
15 recompressed gaseous carbon dioxide and stored in the supply tank. Alternatively, liquid carbon dioxide from the storage vessel may be preheated, for example, by suitable heating coils around the feed line, to a temperature of about 0°F to about 84°F and a pressure
20 of about 300 psig to about 1000 psig before being introduced into the pressure vessel. After the carbon dioxide is introduced into the pressure vessel, the interior of the vessel, including the tobacco to be treated, will generally be at a temperature of from
25 about 20°F to about 80°F and a pressure sufficient to maintain the carbon dioxide gas at or substantially at a saturated state.

Tobacco stability, i.e., the length of time the impregnated tobacco may be stored after
30 depressurization before the final expansion step and still be satisfactorily expanded, is dependent on the initial tobacco OV content, i.e., pre-impregnation OV content, and the tobacco temperature after venting of the pressure vessel. Tobacco with a higher initial OV
35 content requires a lower tobacco post-vent temperature

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than tobacco with a lower initial OV content to achieve the same degree of stability.

The effect of OV content on the stability of tobacco impregnated with carbon dioxide gas at 250 psia and -18°C was determined by placing a weighed sample of bright tobacco, typically about 60g to about 70g, in a 300 cc pressure vessel. The vessel was then immersed in a temperature controlled bath set at -18°C. After the vessel reached thermal equilibrium with the bath, the vessel was purged with carbon dioxide gas. The vessel was then pressured to about 250 psia. Gas phase impregnation was assured by maintaining the carbon dioxide pressure at least 20 psi to 30 psi below the carbon dioxide saturation pressure at -18°C. After allowing the tobacco to soak at pressure for about 15 minutes to about 60 minutes the vessel pressure was rapidly decreased to atmospheric pressure in about 3 seconds to about 4 seconds by venting to atmosphere. The vent valve was immediately closed and the tobacco remained in the pressure vessel immersed in the temperature controlled bath at -18°C for about 1 hour. After about 1 hour, the vessel temperature was increased to about 25°C over about two hours in order to liberate the carbon dioxide remaining in the tobacco. The vessel pressure and temperature were continually monitored using an IBM compatible computer with LABTECH version 4 data acquisition software from Laboratories Technologies Corp. The amount of carbon dioxide evolved by the tobacco over time at a constant temperature, can be calculated based on the vessel pressure over time.

FIG. 3 compares the stability of about 12%, 14%, 16.2% and 20% OV bright tobacco impregnated with carbon dioxide gas at 250 psia at -18°C as described above. Tobacco with an OV content of about 20% lost

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about 71% of its carbon dioxide pickup after 15 minutes at -18°C, while tobacco with an OV content of about 12% lost only about 25% of its carbon dioxide pickup after 60 minutes. The total amount of carbon dioxide evolved
5 after increasing the vessel temperature to 25°C is an indication of the total carbon dioxide pickup. This data indicates that, for impregnations at comparable pressures and temperatures, as tobacco OV content increases, tobacco stability decreases.

10 In order to achieve sufficient tobacco stability, it is preferred that the tobacco temperature be approximately about 0°F to about 10°F after venting of the pressure vessel when the tobacco to be expanded has an initial OV content of about 15%. Tobacco with
15 an initial OV content greater than about 15% should have a post-vent temperature lower than about 0°F to about 10°F and tobacco with an initial OV content less than 15% may be maintained at a temperature greater than about 0°F to about 10°F in order to achieve a
20 comparable degree of stability. For example, FIG. 4 illustrates the effect of tobacco post-vent temperature on tobacco stability at various OV contents. FIG. 4 shows that tobacco with a higher OV content, about 21%, requires a lower post-vent temperature, about -35°F, in
25 order to achieve a similar level of carbon dioxide retention over time as compared to a tobacco with a lower OV content, about 12%, with a post-vent temperature of about 0°F to about 10°F. FIGS. 5 and 6, respectively, show the effect of tobacco OV content and
30 post-vent temperature on equilibrated CV and specific volume of tobacco expanded after being held at its indicated post-vent temperature for the indicated time.

Figures 4, 5, and 6 are based on data from Runs 49, 54, and 65. In each of these runs, bright
35 tobacco was placed in a pressure vessel with a total

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volume of 3.4 cubic feet, 2.4 cubic feet of which was occupied by the tobacco. In Runs 54 and 65, approximately 22 lbs. of 20% OV tobacco was placed in the pressure vessel. This tobacco was pre-cooled by
5 flowing carbon dioxide gas through the vessel at about 421 psig and at about 153 psig for Runs 54 and 65, respectively, for about 4 to 5 minutes prior to pressurization to about 800 psig with carbon dioxide gas. In Run 49, approximately 13.5 pounds of tobacco
10 at about 12.6% OV was placed in the pressure vessel which was then pressurized to about 800 psig with carbon dioxide gas without an intermediate cooling step. The mass of carbon dioxide in the vessel at 800 psig, the mass of tobacco loaded into the vessel at the
15 lower packing density of 12.6% OV tobacco and the lower heat capacity of the tobacco at 12.6% OV were such that the amount of carbon dioxide condensed on the tobacco required to achieve the final post-vent temperature of about 0°F to 10°F was negligible for Run 49.

20 Impregnation pressure, mass ratio of carbon dioxide to tobacco, and heat capacity of tobacco can be manipulated in such a manner that under specific circumstances, the amount of cooling required from the evaporation of condensed carbon dioxide is minimal
25 relative to the cooling provided by the expansion of carbon dioxide gas upon depressurization. However, as the mass ratio of carbon dioxide gas to tobacco decreases, i.e., as the tobacco bulk density increases, the cooling required from the evaporation of condensed
30 carbon dioxide increases. In order to achieve increased process throughput and more uniform tobacco expansion by pre-compacting the tobacco, it is necessary to achieve the controlled formation and evaporation of condensed carbon dioxide according to
35 the invention.

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In each of Runs 49, 54, and 65, after reaching the impregnation pressure of about 800 psig, the system pressure was held at about 800 psig for about 5 minutes before the vessel was rapidly depressurized to atmospheric pressure in approximately 90 seconds. The mass of carbon dioxide condensed per lb. of tobacco during pressurization after cooling was calculated for Runs 54 and 65 and is reported below. The impregnated tobacco was held at its post-vent temperature under a dry atmosphere until it was expanded in a 3-inch diameter expansion tower by contact with steam set at the indicated temperature and at a velocity of about 135 ft/sec for less than about 5 seconds.

		TABLE 1		
Run		49	54	65
Feed OV%		12.6	20.5	20.4
Tobacco Wt. (lbs.)		13.5	22.5	21.25
CO ₂ flow-thru cooling press. (psig)		none	421	153
Impreg. press (psig)		800	800	772
Pre-cool temp (°F)		N/A	10	-20
Post-vent temp. (°F)		0-10	10-20	-35
Expansion Tower gas temp (°F)		475	575	575
Eq CV (cc/g)		10.4	8.5	10.0
SV (cc/g)		3.1	1.8	2.5
Calculated CO ₂ condensed (lb./lb. tob.)		negligible	0.19	0.58

The degree of tobacco stability required, and hence, the desired tobacco post-vent temperature, is dependent on many factors including the length of time after depressurization and before expansion of the

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- tobacco. Therefore, the selection of a desired post-vent temperature should be made in light of the degree of stability required. As a further improvement of the process according to the invention, it is suggested
- 5 that the impregnated tobacco be handled between the impregnation and expansion steps so as to maintain the tobacco's retention of carbon dioxide. For example, the tobacco should be conveyed by an insulated and
cooled conveyor, and should be isolated from any
- 10 moisture laden air.

- The desired tobacco post-vent temperature may be obtained by any suitable means including pre-cooling of the tobacco before introducing it to the pressure vessel, in-situ cooling of the tobacco in the pressure
- 15 vessel by purging with cold carbon dioxide or other suitable means, or vacuum cooling in situ augmented by flow through of carbon dioxide gas. Vacuum cooling has the advantage of reducing the tobacco OV content without thermal degradation of the tobacco. Vacuum
- 20 cooling also removes non-condensable gases from the vessel, thereby allowing the purging step to be eliminated. Vacuum cooling can be effectively and practically used to reduce the tobacco temperature to as low as about 30°F. It is preferred that the tobacco
- 25 is cooled in situ in the pressure vessel.

- The amount of pre-cooling or in-situ cooling required to achieve the desired tobacco post-vent temperature is dependent on the amount of cooling provided by the expansion of the carbon dioxide gas
- 30 during depressurization. The amount of tobacco cooling due to the expansion of the carbon dioxide gas is a function of the ratio of the mass of the carbon dioxide gas to the mass of tobacco, the heat capacity of the tobacco, the final impregnation pressure, and the
- 35 system temperature. Therefore, for a given

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which contains
is compared to a vessel of identical non-compacted
tobacco,

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This results in less
carbon dioxide being
available for

impregnation, when the tobacco feed and the system pressure, temperature and volume are fixed, control of the final post-vent temperature of the tobacco may be achieved by controlling the amount of carbon dioxide permitted to condense on the tobacco. The amount of tobacco cooling due to evaporation of the condensed carbon dioxide from the tobacco is a function of the ratio of the mass of condensed carbon dioxide to the mass of tobacco, the heat capacity of the tobacco, and the temperature or pressure of the system.

When the tobacco is compacted prior to impregnation with carbon dioxide, a greater packing density results and allows a greater tobacco mass to be filled into the impregnation vessel. The carbon dioxide mass within the vessel is decreased, resulting in a decreased expansion cooling. Therefore in order to achieve a desired post-vent temperature, additional cooling must be provided by vaporization of an increased amount of condensed carbon dioxide.

The required tobacco stability is determined by the specific design of the impregnation and expansion processes used. FIG. 13 illustrates the tobacco post-vent temperature required to achieve the desired tobacco stability as a function of OV for a particular process design. The lower shaded area 200 illustrates the amount of cooling contributed by carbon dioxide gas expansion and the upper area 250 illustrates the amount of additional cooling required by carbon dioxide liquid evaporation as a function of tobacco OV to provide the required stability. For this example, adequate tobacco stability is achieved when the tobacco temperature is at or below the temperature shown by the "stability" line. The process variables which determine the tobacco post-vent temperature include the variables discussed previously and other

The greater mass of tobacco in the impregnation vessel results in less post-vent cooling due to the condensed carbon dioxide. When the vessel is filled with tobacco, the amount of available cooling is reduced.

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Tobacco

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Curves similar to FIG. 13 may be drawn
for other process conditions.

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variables including, but not limited to, vessel temperature, vessel mass, vessel volume, vessel configuration, flow geometry, equipment orientation, heat transfer rate to the vessel walls, and process
5 designed retention time between impregnation and expansion.

For the 800 psig process illustrated in FIG. 13, with a post-vent hold time of about 1 hour, no pre-cooling is required for 12% OV tobacco to achieve
10 the required stability, whereas 21% OV tobacco requires sufficient pre-cooling to achieve a post-vent temperature of about -35°F. It should be understood that other process conditions will result in other stability lines, i.e., acceptable stability may be
15 achieved in a stability region generally tracking the stability line of FIG. 13.

The desired tobacco post-vent temperature of the present invention, from about -35°F to about 20°F, is significantly higher than the post-vent
20 temperature -- about -110°F -- when liquid carbon dioxide is used as the impregnant. This higher tobacco post-vent temperature and lower tobacco OV allow the expansion step to be conducted at a significantly lower temperature, resulting in an expanded tobacco with less
25 toasting and less loss of flavor. In addition, less energy is required to expand the tobacco. Moreover, because very little, if any, solid carbon dioxide is formed, handling of the impregnated tobacco is simplified. Unlike tobacco impregnated with only
30 liquid carbon dioxide, tobacco impregnated according to the present invention does not tend to form clumps which must be mechanically broken. Thus, a greater usable-tobacco yield is achieved because the clump-breaking step which results in tobacco fines too small
35 for use in cigarettes is eliminated.

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Moreover, about 21% OV tobacco at about -35°F to about 12% OV tobacco at about 20°F, unlike any OV tobacco at about -110°F, is not brittle and, therefore, is handled with minimum degradation. This property results in a greater yield of usable tobacco because less tobacco is mechanically broken during normal handling, e.g., during unloading of the pressure vessel or transfer from the pressure vessel to the expansion zone.

Chemical changes during expansion of the impregnated tobacco, e.g., loss of reducing sugars and alkaloids upon heating, can be reduced by increasing the exit tobacco OV, i.e., the tobacco OV content immediately after expansion, to about 6% OV or higher. This can be accomplished by reducing the temperature of the expansion step. Normally, an increase in tobacco exit OV is coupled with a decrease in the amount of expansion achieved. The decrease in the amount of expansion depends strongly on the starting feed OV content of the tobacco. As the tobacco feed OV is reduced to approximately 13%, minimal reduction in the degree of expansion is observed even at a tobacco moisture content of about 6% or more exiting the expansion device. Therefore, if the feed OV and the expansion temperature are reduced, surprisingly good expansion can be attained while chemical changes are minimized. This is shown in FIGS. 7, 8 and 9.

FIGS. 7, 8, and 9 are based on data from Runs 2241 thru 2242 and 2244 thru 2254. This data is tabulated in Table 2. In each of these runs a measured amount of bright tobacco was placed in a pressure vessel similar to the vessel described in Example 1.

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TABLE 2

Run No.	2241	2242	2244-46 (3rd)	2245 (2nd)
Tobacco wt (lb.)	100	100	325	325
CO ₂ condensed (lb./lb.) (calculated)	Not applicable	Not applicable	0.36	0.36
Tower Temp (°F)	625	675	500	550
Feed: As Is OV	18.8	18.9	17.0	17.2
Eq OV	12.2	12.1	12.2	12.1
Eq CV (cc/g)	4.5	4.6	4.8	4.9
SV (cc/g)	0.8	0.9	0.8	0.8
Tower: As Is OV	2.5	2.2	4.6	3.3
Eq OV	11.5	11.2	11.9	11.8
Eq CV (cc/g)	9.5	10.8	7.1	8.2
SV (cc/g)	3.0	3.1	1.8	2.3
Feed:				
Alkaloids*	2.71	2.71	2.71	2.71
Reducing Sugars*	13.6	13.6	13.6	13.6
Tower Exit:				
Alkaloids*	2.12	1.94	2.47	2.42
% Reduction	21.8	28.4	8.9	10.7
Reducing Sugars*	11.9	10.6	13.3	13.3
% Reduction	12.5	22.0	2.2	2.2

* weight %, dry weight basis

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TABLE 2 (cont'd)

Run No.	2246(1st)	2247-48(1st)	2248(2nd)	2249-50(1st)
Tobacco wt (lb.)	325	240	240	240
CO ₂ Condensed (lb./lb.) (calculated)	0.36	0.29	0.29	0.29
Tower Temp (°F)	600	400	450	500
Feed: As Is OV	17.5	14.30	14.2	15.2
Eq OV	12.0	11.6	11.8	11.8
Eq CV (cc/g)	4.9	5.2	5.3	5.3
SV (cc/g)	0.8	0.8	0.8	0.8
Tower: As Is OV	3.1	6.1	4.6	4.4
Eq OV	11.6	12.0	11.6	11.5
Eq CV (cc/g)	9.5	7.4	8.7	9.4
SV (cc/g)	2.8	2.2	2.6	2.9
Feed:				
Alkaloids*	2.71	2.71	2.71	2.71
Reducing Sugars*	13.6	13.6	13.6	13.6
Tower Exit:				
Alkaloids*	2.12	2.61	2.49	2.36
% Reduction	21.8	3.7	8.1	12.9
Reducing Sugars*	11.2	13.6	13.6	13.2
% Reduction	17.6	0	0	2.9

* weight %, dry weight basis

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TABLE 2 (cont'd)

	<u>2250 (20-1)</u>	<u>2251-52 (1st)</u>	<u>2252 (2nd)</u>	<u>2253-54 (1st)</u>	<u>2254 (2nd)</u>
	210	210	210	210	210
	0.22	0.25	0.25	0.25	0.25
	350	375	425	475	525
	15.0	12.9	13.0	12.8	12.9
	11.9	12.0	11.6	11.8	12.0
	5.3	5.4	5.4	5.3	5.4
	0.8	0.8	0.8	0.8	0.8
	2.8	6.5	5.0	3.60	2.9
	11.4	12.2	12.1	11.8	11.7
	9.4	8.6	8.9	8.9	9.1
	7.0	2.6	2.8	3.1	3.2
	2.71	2.71	2.71	2.71	2.71
	13.6	13.6	13.6	13.6	13.6
	2.26	2.54	2.45	2.39	2.28
	18.6	6.3	9.6	11.8	15.9
	13.2	13.6	13.5	13.1	12.9
	2.9	0	0.7	3.7	5.1

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Liquid carbon dioxide at 430 psig was used to impregnate the tobacco in Runs 2241 and 2242. The tobacco was allowed to soak in the liquid carbon dioxide for about 60 seconds before the excess liquid was drained. The vessel was then rapidly depressurized to atmospheric pressure, forming solid carbon dioxide in situ. The impregnated tobacco was then removed from the vessel and any clumps which may have formed were broken. The tobacco was then expanded in an 8-inch expansion tower by contact with a 75% steam/air mixture set at the indicated temperature and a velocity of about 85 ft/sec for less than about 4 seconds.

The nicotine alkaloids and reducing sugars content of the tobacco prior to and after expansion were measured using a Bran Luebbe (formerly Technicon) continuous flow analysis system. An aqueous acetic acid solution is used to extract the nicotine alkaloids and reducing sugars from the tobacco. The extract is first subjected to dialysis which removes major interferences of both determinations. Reducing sugars are determined by their reaction with p-hydroxybenzoic acid hydrazide in a basic medium at 85°C to form a color. Nicotine alkaloids are determined by their reaction with cyanogen chloride, in the presence of aromatic amine. A decrease in the alkaloids or the reducing sugars content of the tobacco is indicative of a loss of or change in chemical and flavor components of the tobacco.

Runs 2244 thru 2254 were impregnated with gaseous carbon dioxide at 800 psig according to the method described in Example 1. In order to study the effect of expansion temperature, tobacco from a single impregnation was expanded at different temperatures. For example, 325 lbs. of tobacco were impregnated and then three samples, taken over the course of about 1

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hour, were tested and expanded at 500°F, 550°F, and 600°F, representing Runs 2244, 2245, and 2246, respectively. In order to study the effect of OV content, batches of tobacco with OV contents of about 13%, 15%, 17%, and 19% were impregnated. The notation 1st, 2nd, or 3rd next to the run number indicates the order in which the tobacco was expanded from a particular impregnation. The impregnated tobacco was expanded in an 8-inch expansion tower by contact with a 75% steam/air mixture set at the indicated temperature and a velocity of about 85 ft/sec for less than about 4 seconds. The alkaloids and reducing sugars content of the tobacco were measured in the same manner as described above.

Referring to FIG. 2, tobacco to be treated is introduced to the dryer 10, where it is dried from about 19% to about 28% moisture (by weight) to from about 12% to about 21% moisture (by weight), preferably about 13% to about 15% moisture (by weight). Drying may be accomplished by any suitable means. This dried tobacco may be stored in bulk in a silo for subsequent impregnation and expansion or it may be fed directly to the pressure vessel 30 after suitable temperature adjustment and compaction, if necessary.

Optionally, a measured amount of dried tobacco is metered by a weighbelt and fed onto a conveyor belt within the tobacco cooling unit 20 for treatment prior to impregnation. The tobacco is cooled within the tobacco cooling unit 20 by any conventional means including refrigeration, to less than about 20°F, preferably to less than about 0°F, before being fed to the pressure vessel 30.

The block diagram of FIG. 2A is similar to that of FIG. 2 but additionally shows a compacting device 80 for compacting the tobacco prior to its

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5 impregnation with carbon dioxide according to the
improved embodiment of the present invention. The
compacting device 80 compresses or compacts the tobacco
from an initial loose bulk density of from about [4] to
about [7 lbs./cu.ft.] up to a compacted bulk density of
from about [10] to about [15 lbs./cu.ft.], and
preferably about [11] to about [14^{1/2} cu.ft.]. The
compacted bulk density of the tobacco is substantially
uniform throughout the entire tobacco bed. This is
10 because tobacco in a bed is naturally compacted from
the weight of the overlying tobacco until it reaches a
certain threshold density. For example, when [TYPE]
tobacco of [NUMBER] 4 OV was loosely filled into a
cylinder of [HEIGHT ? 6 ft.?] height and [DIAMETER]
15 diameter, the measured packing density was [DENSITY]
essentially uniform⁷ at measurement points at [e.g.,
1, 2, and 3 feet from the bottom] and diminished
essentially linearly from [e.g., 3 to 6 feet from the
bottom.] If a tobacco bed is compacted to at least the
20 threshold packing density of about [DENSITY], the
gravitational compacting effect is negligible, and the
packing density will be substantially uniform
throughout the bed.

25 The tobacco may be compacted in situ in the
pressure vessel or in a separate compacting vessel, or
~~both, wherein the tobacco must be transported into the~~
~~pressure vessel while maintaining a compacted packing~~
~~density.~~ Thus, the compacting device 80 may be
independent from or integral with the pressure vessel
30 30, and includes the appropriate compacting arrangement
and transport arrangement.

35 The cooled and compacted tobacco is fed to
the pressure vessel 30 through the tobacco inlet 31
where it is deposited. Preferably, the pressure vessel
30 is a cylinder having a vertically extending

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5 longitudinal axis, with a carbon dioxide supply inlet 33 arranged at or near the bottom of the vessel 30 and a carbon dioxide vent outlet 32 arranged at or near the top of the vessel 30. However, venting may be achieved in any convenient direction, ^{e.g., vertical, horizontal, radial, etc.} because the process of the invention achieves ~~very~~ uniform temperatures throughout the tobacco bed, *which is essentially homogenous and uniform and allows a uniform gas flow in any direction.*

10 The pressure vessel 30 is then purged with gaseous carbon dioxide, to remove any air or other non-condensable gases from the vessel 30. It is desired that the purge be conducted in such a manner as not to significantly raise the temperature of the tobacco in the vessel 30. Preferably, the effluent of this purge step is treated in any suitable manner to recover the

15 carbon dioxide for reuse or it may be vented to atmosphere through line 34.

Following the purge step, carbon dioxide gas is introduced to the pressure vessel 30 from the supply tank 50 where it is maintained at about 400 psig to

20 about 1050 psig. When the inside pressure of the vessel 30 reaches from about 300 psig to about 500 psig, the carbon dioxide outlet 32 is opened allowing the carbon dioxide to flow through the tobacco bed cooling the tobacco to a substantially uniform

25 temperature while maintaining the pressure of the vessel 30 at from about 300 psig to about 500 psig. After a substantially uniform tobacco temperature is reached, the carbon dioxide outlet 32 is closed and the pressure of the vessel 30 is increased to from about

30 700 psig to about 1000 psig, preferably about 800 psig, by the addition of carbon dioxide gas. Then the carbon dioxide inlet 33 is closed. At this point, the tobacco bed temperature is approximately at the carbon dioxide saturation temperature. While pressures as high as

35 1050 psig might be economically employed, and a

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pressure equal to the critical pressure of carbon dioxide, 1057 psig, would be acceptable, there is no known upper limit to the useful impregnation pressure range, other than that imposed by the capabilities of the equipment available and the effects of supercritical carbon dioxide on the tobacco.

During pressurization of the pressure vessel, it is preferred that a thermodynamic path is followed that allows a controlled amount of the saturated carbon dioxide gas to condense on the tobacco. FIG. 1 is a standard temperature ($^{\circ}\text{F}$) - entropy ($\text{Btu/lb}^{\circ}\text{F}$) diagram for carbon dioxide with line I-V drawn to illustrate one thermodynamic path in accord with the present invention. For example, tobacco at about 65°F is placed in a pressure vessel (at I) and the vessel pressure is increased to about 300 psig (as shown by line I-II). The vessel is then cooled to about 0°F by flow-thru cooling of carbon dioxide at about 300 psig (as shown by line II-III). Additional carbon dioxide gas is introduced to the vessel, raising the pressure to about 800 psig and the temperature to about 67°F . However, because the temperature of tobacco is below the saturation temperature of the carbon dioxide gas, a controlled amount of carbon dioxide gas will uniformly condense on the tobacco (as shown by line III-IV). After holding the system at about 800 psig for the desired length of time, the vessel is rapidly depressurized to atmospheric pressure resulting in a post-vent temperature of about -5°F to about -10°F (as shown by line IV-V).

In-situ cooling of the tobacco to about 10°F prior to pressurization generally will allow an amount of the saturated carbon dioxide gas to condense. Condensation generally will result in a substantially uniform distribution of liquid carbon dioxide

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throughout the tobacco bed. Evaporation of this liquid carbon dioxide during the vent step will help cool the tobacco in a uniform manner. A uniform post-impregnation tobacco temperature results in a more
5 uniform expanded tobacco. The uniform condensation of carbon dioxide and the resultant uniform cooling is especially achieved according to the improved embodiment wherein the tobacco has been pre-compressed to an increased, substantially uniform packing density.
10 The uniformity of the condensation is shown by the uniformity of the temperature distribution within the tobacco bed.

This uniform tobacco temperature is illustrated in FIG. 10, which is a schematic diagram of
15 the impregnation vessel 100 used in Run 28 showing the temperature, in °F, at various locations throughout the tobacco bed after venting. For example, the tobacco-bed temperature at cross-section 120, 3 feet from the top of vessel 100, was found to have
20 temperatures of about 11°F, 7°F, 7°F, and 3°F. About 1800 lbs. of bright tobacco with an OV content of about 15% was placed in a 5 ft (i.d.) x 8.5 ft (ht) pressure vessel. The vessel was then purged with carbon dioxide gas for about 30 seconds before pressurizing to about
25 350 psig with carbon dioxide gas. The tobacco bed was then cooled to about 10°F by flow-thru cooling at 350 psig for about 12.5 minutes. The vessel pressure was then increased to about 800 psig and held for about 60 seconds before rapidly depressurizing in about 4.5
30 minutes. The temperature of the tobacco bed at various points was measured and found to be substantially uniform as shown in FIG. 10. It was calculated that about 0.26 lbs. of carbon dioxide condensed per lb. of tobacco.

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Returning to FIG. 2, the tobacco in the pressure vessel 30 is maintained under carbon dioxide pressure at about 800 psig for from about 1 second to about 300 seconds, preferably about 60 seconds. It has been discovered that tobacco contact time with carbon dioxide gas, i.e., the length of time that the tobacco must be maintained in contact with the carbon dioxide gas in order to absorb a desired amount of carbon dioxide, is influenced strongly by the tobacco OV content and the impregnation pressure used. Tobacco with a higher initial OV content requires less contact time at a given pressure than tobacco with a lower initial OV content in order to achieve a comparable degree of impregnation particularly at lower pressures. At higher impregnation pressures, the effect of tobacco OV on contact time with the carbon dioxide gas is reduced. This is illustrated in Table 3.

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TABLE 3

Effects Of Impregnation Pressure And Tobacco OV On Contact Time With CO₂

Run	<u>20</u>	<u>14</u>	<u>21</u>	<u>59</u>	<u>49</u>	<u>33</u>	<u>32</u>	<u>35</u>	<u>30</u>	<u>27</u>
Initial Tob OV (%)	12.2	11.7	11.8	12.3	12.6	16.7	16.4	16.9	16.5	16.0
Impregnation Pressure (psig)	471	462	465	802	800	430	430	430	460	450
Contact Time at Impregnation Press. (minutes)	5	15	60	1	5	0.25	5	10	15	20
Tower Exit:										
Eq CV (cc/g)	7.5	8.7	10.1	9.8	10.4	8.5	9.3	10.5	11.1	10.5
SV (cc/g)	1.8	2.1	2.8	3.1	3.1	2.1	2.6	3.4	3.1	2.9
Control*										
Eq CV (cc/g)	5.3	5.4	5.2	5.6	5.7	5.5	5.5	5.7	5.5	5.5
SV (cc/g)	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8

* CV and SV of feed tobacco

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After the tobacco has soaked sufficiently, the pressure vessel 30 is depressurized rapidly to atmospheric pressure in from about 1 second to about 300 seconds, depending on vessel size, by venting the carbon dioxide first to the carbon dioxide recovery unit 40 and then through line 34 to atmosphere. Carbon dioxide which has condensed on the tobacco is vaporized during this vent step, helping to cool the tobacco, resulting in a tobacco post-vent temperature of from about -35°F to about 20°F.

Impregnated tobacco from the pressure vessel 30 may be expanded immediately by any suitable means, e.g., by feeding to the expansion tower 70. Alternatively, impregnated tobacco may be maintained for about 1 hour at its post-vent temperature in the tobacco transfer device 60 under a dry atmosphere, i.e., an atmosphere with a dewpoint below the post-vent temperature, for subsequent expansion. After expansion and, if desired, reordering, the tobacco may be used in the manufacture of tobacco products, including cigarettes.

As already described, the process according to the invention may be advantageously adapted to a short-cycle impregnation of tobacco in relatively small batches, so that the process becomes essentially continuous. A preferred embodiment of such a process will now be described with reference to FIGS. 14 to 19.

FIG. 14 is a schematic top view of an apparatus for carrying out the preferred process according to the invention. A turntable 2 is mounted on a frame 1, to rotate counterclockwise (arrow R) about a substantially vertical axis A. An upper frame 1' carries a pressure vessel 30 and associated apparatus described below, as well as tobacco feed

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devices 9, 11 and 12, a compression assembly 13 and a discharge assembly 3 shown in Fig. 15. The turntable 2 is driven to rotate (arrow R) in steps of substantially 90° by a drive arrangement. For example a motor and blockable gear train or a stepper motor, which is not shown. Mounted in the turntable 2 as described below are four similar cylindrical tubes, namely tube 4 shown in feed or filling position, tube 5 shown in a pressing position, tube 6 shown below an impregnation station position, and tube 7 shown in a discharge position. As the drive arrangement rotates turntable 2 in 90° rotational steps, each tube 4, 5, 6 and 7 is rotated to and momentarily held at each successive process station as described below.

FIG. 15 is a cylindrical sectional projection of the apparatus of Fig. 14. The rotating turntable 2 is arranged directly above a stationary table 2'. Conventional bearings can support turntable 2 and table 2' to allow their relative rotational motion. The tubes 4, 5, 6 and 7 are each arranged in a corresponding hole in the turntable 2, so that each tube remains open from the top and from the bottom through the turntable 2. A wiper seal 8 may be arranged around the lower inner circumferential edge of each tube to seal against table 2' to prevent tobacco from entering the space between turntable 2 and table 2'. A feed conveyor 9 delivers bulk tobacco (e.g., 15% or content tobacco) in an essentially continuous stream into a surge chute or surge tube 11. The tobacco may, for example, have been pretreated by a dryer 10 and cooler 20 referenced in FIG. 2, before being delivered by feed conveyor 9. The tobacco falls through the surge tube 11 and through

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an open slide gate 12 into the tube 4 in the feed position. The tobacco feed rate is controlled so that tube 4 is filled substantially to the top during a one-station cycle time of 96 seconds. Turntable 2 then rotates within 4 seconds to move tube 4 into the compacting or pressing station occupied by tube 5 in the view of FIG. 15.

While the turntable 2 rotates between successive stopped positions as described, the slide gate 12 closes and stops the flow of loose tobacco which then backs-up or stockpiles in surge tube 11 until the next tube (e.g., tube 7) is positioned below slide gate 12, whereupon slide gate 12 opens. Each tube is about 24" in length, with an inner diameter of 14" and a wall thickness adequate to withstand compaction forces on the tobacco. When a filled tube is in the pressing position of tube 5, a compaction piston assembly 13, which may, for example, be a hydraulically driven piston and cylinder, compresses or compacts the tobacco to about half of its initial loose fill volume and almost twice its initial loose fill packing density, i.e., raising the packing density from about 7 lbs./cu.ft. to about 13 lbs./cu.ft.

After compressing the tobacco, the compaction piston assembly 13 retracts before a one-station cycle time of 96 seconds has expired. Then the tube containing compacted tobacco is rotated in 4 seconds to the impregnation position of tube 6 and positioned in alignment with a hole 6' in table 2'. A pressure vessel piston assembly 14 moves from a position shown by broken lines below table 2', through hole 6' and through tube 6, piston assembly 14 carries the pre-compacted tobacco out of tube 6 and into pressure vessel 30. Piston assembly 14 then compresses the

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tobacco further, to a packing density of about 14 lbs./cu.ft. Then locking pin 15 locks piston assembly 14 into place, and the compressed tobacco is impregnated with carbon dioxide within pressure vessel 30 according to the process of the invention as more particularly described below.

Locking pin 15 is moved to an unlocked position, piston assembly 14 is withdrawn from pressure vessel 30, and simultaneously ejection piston 16 is driven downward to assure that the impregnated bed of tobacco is completely cleared from the pressure vessel. Once piston assembly 14 is clear of the bottom of tube 6 and piston 16 is retracting back toward its starting position, tube 6 may be rotated to carry the impregnated tobacco to the discharge station of tube 7 in FIG. 15.

A discharge assembly 3, such as a piston, moves down through tube 7 to assure that the impregnated tobacco is completely cleared from tube 7 and then retracts. The tobacco falls through a hole 71 in table 21 and into a discharge hopper assembly 17. Hopper assembly 17 is insulated and cooled with dry 0° F air to preserve the carbon dioxide impregnation of the tobacco. Hopper assembly 17 includes a surge hopper 18 and a conveyor 19. The hopper assembly evenly out the individual batches of impregnated tobacco (about 14 lbs. each) into a continuous bulk flow of tobacco to prevent "choke-feeding" the expansion apparatus. Also, the hopper assembly and conveyor helps reconfigure the tobacco bulk flow from an essentially cylindrical shape to the rectangular shape of the inlet of the expansion apparatus.

FIG. 16 is an enlarged sectional view of the pressure vessel arrangement 30 of FIG. 15, after the pressure vessel piston 14 has pushed a pre-compacted

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tobacco bed (not shown for better clarity) into the pressure vessel, further compacted the tobacco (A) and been locked in place by locking pin 15. Pressure vessel 30 includes a cylinder 34 such as an autoclave cylinder 34 such as an autoclave cylinder having a 14" internal diameter. Cylinder 34 is preferably lined with a thermally insulating liner 35 having a wall thickness of about 0.25". A cover 36 forms the top of pressure vessel 30. The ejection piston assembly 16 is arranged to move in the directions of arrow 16' through a hole fitted with a pressure seal 37 in cover 36. A shaft 38 of piston assembly 16 carries an upper gas distributor plate 39a, an upper gas chamber plate 41a and an upper screen 42a. The screen 42a, plate 41a and plate 39a are dimensioned to fit closely but movably within the insulating liner 35, with a wiper 43a arranged around the circumference of screen 42a. At the opposite end of pressure vessel 30, the piston assembly 14 carries a similar arrangement of a lower screen 42b with a wiper 43b, a lower gas chamber plate 41b and a lower gas distributor plate 39b. The components 42b, 41b and 39b have a diameter to fit slidably within the inner diameter of cylinder 34, e.g., 14". Thus, a tobacco impregnation cavity is formed, bounded radially by the inner walls of liner 35, on top by screen 42a, and on the bottom by screen 42b. A pressurizable cavity is larger than the tobacco cavity. Pressure seal 37 around the shaft of ejection piston 16 and a pressure seal 44 around the upper portion of pressure vessel piston 14 are high pressure seals to confine the carbon dioxide gas at impregnation pressures. A lower pressure seal 45a is arranged between gas distributor plate 39a and cover 36, and a lower pressure seal 45b is arranged between

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the circumference of gas chamber plate 41b and the inner wall of cylinder 34. Low pressure seals 45a and 45b may be O-ring seals, which only need to withstand the low pressure differential across the respective gas distributor plate, gas chamber plate and screen. These seals 45a and 45b ensure that gas is properly distributed through the screens rather than passing along the walls of the pressure vessel. In order to impregnate the compacted tobacco with carbon dioxide according to the process of the invention, a control valve (not shown) is opened so that carbon dioxide gas is introduced (arrows 33') through gas inlet 33, then through gas plenum 46b, plates 49b and 41b and screen 42b to permeate the tobacco bed.

As carbon dioxide gas flows in, air is purged from the tobacco bed and escapes through screen 42a. plates 41a and 39a, and then via gas plenum 46a through gas outlets 32 to a control valve (not shown) by which gas may be vented to atmosphere or recovered in a recovery arrangement 40 (FIG. 2). Preferably, inlets 33 are arranged at the bottom of plenum 46b to allow any condensation to drain, and outlets 32 are arranged at the top of plenum 46a to allow any heat of compression to vent rather than forming trapped "hot spots."

Initially, the upper control valve is fully open to allow an air purge for 5 seconds. Then the upper control valve is throttled to a pressure of 250 psig, whereupon the pressure vessel pressure-up to 250 psig in about 1.2 seconds. During the first pressure-up to 250 psig, heat of compression will develop uniformly throughout the minimal voids of the tobacco bed. In order to cool the tobacco and form uniformly condensed carbon dioxide according to the invention, carbon dioxide gas is allowed to flow through the bed at 250 psig for

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about 56 seconds. The bed of tobacco is cooled uniformly to under 0° F at saturation conditions for the carbon dioxide at 250 psig (see e.g., FIG. 1). Then, the upper control valve is throttled to 800 psig, whereupon carbon dioxide flows into the bed and pressures up to 800 psig in about 6.4 seconds. As the pressure increases, the saturation temperature of the gas increases, so carbon dioxide condenses onto the tobacco uniformly through the bed. As the condensation warms the tobacco, the tobacco temperature lags behind the increasing saturation temperature of the carbon dioxide gas. Thus, condensation continues to form until the pressure reaches 800 psig. Any heat of compression that develops is eliminated in that it will simply cause evaporation of the condensed carbon dioxide. It has been found that no additional "soak time" is required to achieve sufficient impregnation. Therefore, when 800 psig pressure is attained, the upper and lower control valves are both opened to allow a second venting of carbon dioxide back down to atmospheric pressure. This process is summarized below in Table 5.

short cycle

During venting, some cooling is provided by expansion of the gas, but the majority of cooling is provided by evaporation of condensed carbon dioxide. The cooling effect brings the tobacco bed temperature uniformly to about [FILL IN TEMPERATURE] in this example. The post vent temperature can be controlled by controlling mechanical pre-cooling of the tobacco and the pressure-up cycle parameters, such as the flow through pressure and the maximum pressure, in order to control the amount of condensation achieved. Therefore, uniform cooling, impregnation and post-vent stability can be achieved regardless of bed density.

[FILL IN MORE SPECIFIC EXAMPLES AND DESCRIPTION OF
ADDITIONAL DATA FIGURES]

The following examples are illustrative:

Example 1

- 5 A 240 pound sample of bright tobacco filler
with a 15% OV content was cooled to about 20°F and then
placed in a pressure vessel approximately 2 feet in
diameter and approximately 8 feet in height. The
vessel was then pressured to about 300 psig with carbon
10 dioxide gas. The tobacco was then cooled, while
maintaining the vessel pressure at about 300 psig, to
about 0°F by flushing with carbon dioxide gas near
saturated conditions for about 5 minutes prior to
pressurizing to about 800 psig with carbon dioxide gas.
15 The vessel pressure was maintained at about 800 psig
for about 60 seconds. The vessel pressure was
decreased to atmospheric pressure by venting in about
300 seconds, after which the tobacco temperature was
found to be about 0°F. Based on the tobacco
20 temperature, the system pressure, temperature, and
volume, and the tobacco post-vent temperature, it was
calculated that approximately 0.29 lbs. of carbon
dioxide condensed per lb. of tobacco.

- The impregnated sample had a weight gain of
25 about 2% which is attributable to the carbon dioxide
impregnation. The impregnated tobacco was then, over a
one hour period, exposed to heating in an 8-inch
diameter expansion tower by contact with a 75%
steam/air mixture at about 550°F and a velocity of
30 about 85 ft/sec for less than about 2 seconds. The
product exiting the expansion tower had an OV content
of about 2.8%. The product was equilibrated at
standard conditions of 75°F and 60%RH for about 24
hours. The filling power of the equilibrated product

was measured by the standardized cylinder volume (CV) test. This gave a CV value of 9.4 cc/g at an equilibrium moisture content of 11.4%. An unexpanded control was found to have a cylinder volume of 5.3 cc/g at an equilibrium moisture content of 12.2%. The sample after processing, therefore, had a 77% increase in filling power as measured by the CV method.

The effect of hold time after impregnation prior to expansion on expanded tobacco SV and equilibrated CV was studied in Runs 2132-1 thru 2135-2. In each of these runs, 2132-1, 2132-2, 2134-1, 2134-2, 2135-1, and 2135-2, 225 lbs. of bright tobacco with a 15% OV content was placed in the same pressure vessel as described in Example 1. The vessel was pressured to from about 250 psig to about 300 psig with carbon dioxide gas. The tobacco was then cooled, while maintaining the vessel pressure at about 250 psig to about 300 psig, in the same manner as described in Example 1. The vessel was then pressurized to about 800 psig with carbon dioxide gas. This pressure was maintained for about 60 seconds before the vessel was vented to atmospheric pressure in about 300 seconds. The impregnated tobacco was maintained in an environment with a dewpoint below the tobacco post-vent temperature prior to expansion. FIG. 11 illustrates the effect of hold time after impregnation on the specific volume of expanded tobacco. FIG. 12 illustrates the effect of hold time after impregnation on the equilibrated CV of expanded tobacco.

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Example 2

A 19 pound sample of bright tobacco filler with a 15% OV content was placed in a 3.4 cubic foot pressure vessel. The vessel was then pressured to about 185 psig with carbon dioxide gas. The tobacco was then

cooled, while maintaining the vessel pressure at about 185 psig, to about -25°F by flushing with carbon dioxide gas near saturated conditions for about 5 minutes prior to pressurizing to about 430 psig with carbon dioxide gas. The vessel pressure was maintained at about 430 psig for about 5 minutes. The vessel pressure was decreased to atmospheric pressure by venting in about 60 seconds, after which the tobacco temperature was found to be about -29°F. Based on the tobacco temperature, the system pressure, temperature, and volume, it was calculated that approximately 0.23 lbs. of carbon dioxide condensed per lb. of tobacco.

The impregnated sample had a weight gain of about 2% which is attributable to the carbon dioxide impregnation. The impregnated tobacco was then, over a one hour period, exposed to heating in an 3-inch diameter expansion tower by contact with a 100% steam at about 525°F and a velocity of about 135 ft/sec for less than about 2 seconds. The product exiting the expansion tower had an OV content of about 3.8%. The product was equilibrated at standard conditions of 75°F and 60%RH for about 24 hours. The filling power of the equilibrated product was measured by the standardized cylinder volume (CV) test. This gave an equilibrated CV value of 10.1 cc/g at an equilibrium moisture of 11.0%. An unexpanded control was found to have a cylinder volume of 5.8 cc/g at an equilibrium moisture of 11.6%. The sample after processing, therefore, had a 74% increase in filling power as measured by the CV method.

[EXAMPLES TO BE ADDED]

The term "cylinder volume" is a unit for measuring the degree of expansion of tobacco. As used

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throughout this application, the values employed, in connection with these terms are determined as follows:

Cylinder Volume (CV)

Tobacco filler weighing 20 grams, if
5 unexpanded, or 10 grams, if expanded, is placed in a
6-cm diameter Densimeter cylinder, Model No. DD-60,
designed by the Heinr. Borgwaldt Company, Heinr.
Borgwaldt GmbH, Schnackenburgallee No. 15, Postfach 54
07 02, 2000 Hamburg 54 West Germany. A 2 kg piston,
10 5.6 cm in diameter, is placed on the tobacco in the
cylinder for 30 seconds. The resulting volume of the
compressed tobacco is read and divided by the tobacco
sample weight to yield the cylinder volume as cc/gram.
The test determines the apparent volume of a given
15 weight of tobacco filler. The resulting volume of
filler is reported as cylinder volume. This test is
carried out at standard environmental conditions of
75°F and 60% RH; conventionally, unless otherwise
stated, the sample is preconditioned in this
20 environment for 24-48 hours.

Specific Volume (SV)

The term "specific volume" is a unit for
measuring the volume and true density of solid objects,
e.g., tobacco, using the fundamental principles of the
25 ideal gas law. The specific volume is determined by
taking the inverse of the density and is expressed as
"cc/g". A weighed sample of tobacco, either "as is",
dried at 100°C for 3 hours, or equilibrated, is placed
in a cell in a Quantachrome Penta-Pycnometer. The cell
30 is then purged and pressured with helium. The volume
of helium displaced by the tobacco is compared with
volume of helium required to fill an empty sample cell
and the tobacco volume is determined based on
Archimedes' principle. As used throughout this
35 application, unless stated to the contrary, specific

volume was determined using the same tobacco sample used to determine OV, i.e., tobacco dried after exposure for 3 hours in a circulating air oven controlled at 100°C.

- 5 While the invention has been particularly shown and described with reference to preferred embodiments, it will be understood by those skilled in the art that various changes in form and details may be made without departing from the spirit and scope of the
- 10 invention. For example, as size of the equipment used to impregnate the tobacco varies the time required to reach the desired pressure, or to vent, or to adequately cool the tobacco bed will vary.

CLAIMS TO BE
ADDED

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PROCESS AND APPARATUS FOR IMPREGNATION
AND EXPANSION OF TOBACCO

Abstract Of The Disclosure

A process for expanding tobacco is provided which employs carbon dioxide gas. Tobacco temperature and OV content are adjusted prior to contacting the tobacco with carbon dioxide gas. The disclosed process is suitable for impregnating and expanding tobacco having a high bulk density. In order to achieve a high bulk density, the tobacco may be compacted or compressed to achieve an increased and more uniform bulk density prior to its impregnation with carbon dioxide. The process may be carried out with a short cycle impregnation in an apparatus according to the invention. A thermodynamic path is followed during impregnation which allows a controlled amount of the carbon dioxide gas to condense on the tobacco. This liquid carbon dioxide evaporates during depressurization helping to cool the tobacco bed uniformly. After impregnation, the tobacco may be expanded immediately or kept at or below its post-vent temperature in a dry atmosphere for subsequent expansion.

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Diane Hunter

DRAFT 12/11/92

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DECLARATION AND POWER OF ATTORNEY

5 We: Kwang H. Cho, Thomas J. Clarke,
Joseph M. Dobbs, Eugene B. Fischer, Jose M.G.
Nepomuceno, and Ravi Prasad declare that we are
citizens of the United States of America, residing,
respectively, at: 2141 Carbon Hill Drive, Midlothian,
VA 23113; 1726 Westhill Road, Richmond, VA 23226; 4538
E. Seminary Avenue, Richmond, VA 23227; 12800
10 Nightingale Drive, Chester, VA 23831; Rt. 2 Box 163D4,
Beaverdam, VA 23015; and 10821 Hinshaw Drive,
Midlothian, VA 23113; that we verily believe ourselves
to be the original, first and joint inventors of the
invention or discovery in:

CHECK

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PROCESS AND APPARATUS FOR IMPREGNATION
AND EXPANSION OF TOBACCO

which is described and claimed in the attached
specification and for which a patent is sought;

20 that we have reviewed and do understand the
content of said specification, including the claims,
and acknowledge our duty to disclose information, of
which we are aware, which is material to the
examination of this application in accordance with
Title 37, Code of Federal Regulations, § 1.56(a);

25 that we do not know and do not believe that
this invention or discovery was ever known or used in
the United States of America before our invention or
discovery thereof, or patented or described in any
printed publication in any country before our invention
30 or discovery thereof, or more than one year prior to
this application; or in public use or on sale in the
United States of America more than one year prior to
this application; that this invention or discovery has
not been patented or made the subject of an inventor's
35 certificate issued before the date of this application
in any country foreign to the United States of America

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on an application filed by us or our legal
representatives or assigns more than twelve months
prior to this application; and that no application for
patent or inventor's certificate on this invention or
5 discovery has been filed in any country foreign to the
United States of America by us or our legal
representatives or assigns;

and we hereby appoint James E. Schardt, Esq.,
Reg. No. 23,033, Charles E. B. Glenn, Esq., Reg.
10 No. 29,796, Charles B. Smith, Esq., Reg. No. 16,763,
Albert E. Fey, Esq., Reg. No. 19,018, W. Edward Bailey,
Esq., Reg. No. 30,994, William J. McCabe, Esq., Reg.
No. 33,536, and Walter F. Fasse, Esq., Reg. No. 36,132,
our attorneys, with power of substitution, and with
15 power of appointment of associate attorneys, and of
revocation of their powers, to prosecute this
application and any divisions, continuations in whole
or in part, renewals and reissues of the same, and to
transact all business in the Patent and Trademark
20 Office connected therewith;

and we request that communications be sent
to:

Walter F. Fasse, Esq.
c/o Fish & Neave
25 1251 Avenue of the Americas
New York, New York 10020

and that telephone calls be directed to:

Walter F. Fasse, Esq.
(212) 596-9000.

30 Wherefore, we pray that Letters Patent be
granted to us for the invention or discovery described
and claimed in the attached specification and claims,
and we hereby subscribe our names to the foregoing
specification and claims, declaration, and power of
35 attorney.

We declare, further, that we understand the English language and that all statements made herein of our own knowledge are true, and that all statements made on information and belief are believed to be true; and, further, that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

	Date	Kwang H. Cho
15	Date	Thomas J. Clarke
	Date	Joseph M. Dobbs
	Date	Eugene B. Fischer
20	Date	Jose M.G. Nepomuceno
	Date	Ravi Prasad

Diane Christen?

ARE THESE CORRECT?

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