

PATENT SPECIFICATION

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(54) EXPANDING TOBACCO

(71) We, PHILIP MORRIS INCORPORATED, a Corporation organised and existing under the laws of the State of Virginia, United States of America, of 100 Park Avenue, New York, New York 10017, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to a process and apparatus for expanding tobacco.

The tobacco art has long recognized the 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 was to improve the smoking characteristics of particular tobacco components, namely 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 cigarette, which would have the same firmness and yet would produce lower tar and nicotine than the comparable smoking product made of non-expanded tobacco having a more dense tobacco filler.

Various methods have been proposed for expanding tobacco, including the impregnation of tobacco with a gas under pressure and the subsequent release of the 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 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 tobacco impregnated therewith is heated or the pressure thereon is reduced to thereby expand the tobacco. 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 the tobacco upon their release. More specifically:

A patent to Wilford J. Hawkins, United States Patent 1,789,435, granted in 1931, describes a method and apparatus for expanding the volume of tobacco in order to make up the loss of weight caused in curing tobacco leaf. To accomplish this object, the cured and conditional tobacco is contacted with a gas, which may be air, carbon dioxide or steam under the pressure and the pressure is then relieved, whereby the tobacco tends to expand. The patent states that the volume of the tobacco may, by that process, be increased to the extent of about 5—15%.

A United States property custodian document No. 304,214 to Joachim Bohme, dated 1943, indicates that tobacco can be expanded using a high frequency generator but that there are limitations to the degree of expansion which can be achieved without affecting the quality of the tobacco.

A patent to Frank J. Sowa, United States Patent 2,596,183, granted in 1952, sets forth a method for increasing the volume of shredded tobacco by adding additional water to the tobacco to cause the tobacco to swell and thereafter heating the moisture containing tobacco, whereby the moisture evaporates and the resulting moisture vapor causes expansion of the tobacco.

A series of patents to Roger Z. de la Burde, one of the present coinventors, granted in 1968, specifically United States Patents 3,409,022, 3,409,023, 3,409,027 and 3,409,028, relate to various processes for

(10) enhancing the utility of tobacco stems for use in smoking products by subjecting the stems to expansion operations utilizing various types of heat treatment or microwave energy.

5 A patent to John D. Hind, granted in 1969, United States Patent 3,425,425, which is assigned to the same assignee as the assignee of the present invention, relates to the use of carbohydrates to improve the puffing of
10 tobacco stems. In that process, tobacco stems are soaked in an aqueous solution of carbohydrates and then heated to puff the stems. The carbohydrate solution may also contain organic acids and/or certain salts which are
15 used to improve the flavor and smoking qualities of the stems.

A publication in the "Tobacco Reporter" of November 1969 by P. S. Meyer describes and summarizes tobacco puffing or expansion
20 procedures or investigations for expanding and manipulating tobacco for purposes of reducing costs and also as the means for reducing the "tar" content by reduction in the delivery of smoke. Mention is made in
25 this publication of puffing tobacco by different procedures including the use of halogenated hydrocarbons, low pressure or vacuum operation, or high pressure steam treatment that causes leaf expansion from inside the cell when
30 outside pressure is suddenly released. Mention is also made in this publication of freeze-drying tobacco which can also be employed to obtain an increase in volume.

Since the above-mentioned "Tobacco Reporter" article was published, a number of tobacco expansion techniques, including some of the techniques described in the article, have been described in patents and/or published patent applications. For example:

40 United States Patent 3,524,452 to Glenn P. Moser et al and United States Patent 3,524,451 to James D. Frederickson, both issued in 1970, relate to the expansion of tobacco using a volatile organic liquid, such as a halogenated hydrocarbon.

45 United States Patent No. 3,734,104 to William M. Buchanan et al, which is assigned to the same assignee as the assignee of the present invention, issued in 1973, relates to a particular process for the expansion of tobacco stems.

50 United States Patent 3,710,802 to William H. Johnson, issued in 1973 and British Specification 1,293,735 to American Brands, Inc., published in 1972, both relate to freeze drying methods for expanding tobacco.

55 South African applications 70/8291 and 70/8282 to R. J. Reynolds Tobacco Company, both issued in 1970, relate to tobacco expansion employing chemical compounds which decompose to form a gas or with inert solutions of a gas under pressure to maintain the gas in solution until it impregnates the tobacco.

60 A patent to Robert G. Armstrong et al,

United States Patent 3,771,533, issued in 1973, which is assigned to the same assignee as the assignee of the present invention, involves a treatment of tobacco with carbon dioxide and ammonia gases, whereby the tobacco is saturated with these gases and ammonium carbonate is formed in situ. The ammonium carbonate is thereafter decomposed by heat to release the gas within the tobacco cells and to cause expansion of the tobacco.

70 Despite all of the above-described advances in the art, no completely satisfactory process has been found. The difficulty with the various earlier suggestions for expanding tobacco is that, in many cases, the volume is only slightly or at best only moderately increased, noting for example, the 15% expansion as the maximum achieved by freeze drying, this type of operation has the disadvantages of requiring elaborate and expensive equipment and very substantial operating costs. With respect to the teaching of using heat energy, infrared or radiant microwave energy to expand tobacco stems, the difficulty is that while stems respond to these heating procedures, tobacco leaf has not generally been found to respond effectively to this type of process.

80 The use of special expanding agents, for example, halogenated hydrocarbons, such as are mentioned in the Meyer publication for expanding tobacco, is also not completely satisfactory because these substances are generally required to volatilize or remove the substances after the tobacco has been expanded. The introduction, in considerable concentration, of materials which are foreign to tobacco presents the problem of removing the expansion agent after the treatment has been completed in order to avoid affecting aroma and other properties of the smoke due to extraneous substances used or developed from the combustion of the treated tobacco.

85 The use of solid chemicals to produce a gas upon decomposition has not been found satisfactory, perhaps due to the fact that the chemicals cannot be incorporated in the cells of the tobacco.

90 The use of carbonated water has also not been found to be effective.

95 While the method employing ammonia and carbon dioxide gases is an improvement over the earlier described methods, it is not completely satisfactory and can result, under same circumstances, in undesired deposition of ammonium carbonate during the process.

100 The present process employing liquid carbon dioxide, as described hereinafter, has been found to overcome the disadvantages of the prior art processes and provides an improved method for expanding tobacco.

105 Carbon dioxide has been used in the food industry as a coolant and, more recently, has been suggested as an extractant for food

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flavors. It has also been described in German Offenlegungsschrift 2,142,205 (Anmeldetag; 23 August 1971) for use, in either gaseous or liquid form, to extract aromatic materials from tobacco. However, there has been no suggestion, in connection with these uses, of the use of liquid carbon dioxide for the expansion of these materials.

The present invention provides a process for expanding tobacco which comprises (1) contacting tobacco with liquid carbon dioxide under conditions such that the temperature of the tobacco is maintained at a level no lower than -2°C . and such that substantially all of the liquid carbon dioxide is maintained in liquid form to impregnate the tobacco with the liquid carbon dioxide, (2) subjecting the impregnated tobacco to conditions such that the liquid carbon dioxide is converted to solid carbon dioxide and (3) thereafter subjecting the solid carbon dioxide-containing tobacco to conditions whereby the solid carbon dioxide is vaporised to expand the tobacco.

Preferably the tobacco is contacted in step (1) with liquid carbon dioxide at a pressure of from 490 to 906 psig and a temperature of from -2 to 31°C . for from 0.5 to 60 minutes.

The invention also provides apparatus for expanding tobacco which comprises a pressure vessel, means for introducing and withdrawing tobacco from the pressure vessel, means for introducing liquid carbon dioxide into the pressure vessel to impregnate tobacco contained therein, means for withdrawing liquid carbon dioxide from the pressure vessel after impregnation of said tobacco, means for maintaining carbon dioxide at predetermined temperature and pressure in the pressure vessel whereby it remains liquid during the impregnation, means for converting liquid carbon dioxide impregnating said tobacco into solid carbon dioxide, and means for vaporising solid carbon dioxide impregnating said tobacco to effect expansion of said tobacco.

To carry out the process of the invention, one may treat either whole cured tobacco leaf, tobacco in cut or chopped form, or selected parts of tobacco such as tobacco stems or maybe reconstituted tobacco. In comminuted form, the tobacco to be impregnated may have a particle size of from 20 to 100 mesh, but is preferably not less than 30 mesh. The material treated may be in relatively dry form, or may contain the natural moisture content of tobacco or even more. Generally, the tobacco to be treated will have at least 8% moisture (by weight) and less than about 50% moisture.

The tobacco will generally be placed in a pressure vessel in a manner whereby it can be suitably immersed or contacted by liquid carbon dioxide. For example, a wire cage or platform may be used.

The tobacco-containing pressure vessel is preferably then purged with carbon dioxide gas, the purging operation generally taking from 1 to 4 minutes. The purging step may be eliminated without detriment to the final product. The benefits of purging are the removal of gases that interfere with a carbon dioxide recovery process and to flush foreign gases that may interfere with full penetration of the liquid carbon dioxide.

The liquid carbon dioxide which is employed in the process of this invention will generally be obtained from a storage vessel where it is maintained at a pressure of from 215 to 305 psig and temperatures of from -20 to 0°C . The liquid carbon dioxide may be introduced into the pressure vessel at 215 to 320 psig and -20 to 0°C ., but is preferably preheated, for example, by suitable heating coils around the feed line, to a temperature of from 0 to 25°C ., and a pressure of 490 to 906 psig before being introduced into the pressure vessel. Preheating is preferred because the best operational pressure occurs in the range of 600 to 900 psig and preheating will minimize the heating cycle in the pressure vessel. At the time the liquid 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 0 to 25°C . and pressure at least sufficient to maintain the carbon dioxide in a liquid state.

The liquid carbon dioxide is introduced into the vessel in a manner which permits it to completely contact the tobacco and sufficient liquid carbon dioxide is employed to completely saturate the tobacco. Generally this will comprise using 1 to 10 parts by weight of liquid carbon dioxide per part of tobacco. Excess liquid carbon dioxide will be wasteful but will work. The temperature of the tobacco, during the contact with liquid carbon dioxide should be no higher than 31°C . While the inventors do not wish to be bound by any particular theory, they believe that the conditions of contact between the tobacco and liquid carbon dioxide, under the present invention, are such that the unbound moisture in the tobacco is not frozen after contact, as this would prevent proper saturation of the tobacco by the carbon dioxide, and that the temperature of the tobacco in the vessel be maintained at a level of -2°C . or above or be brought to that level within 2 to 4 minutes after the introduction of the carbon dioxide to the tobacco. Thus, it is believed that it is desirable to have conditions whereby there is simultaneously retention of all or substantially all of the unbound moisture which is present in the tobacco in liquid form. It is believed that by maintaining conditions such that ice is not formed while the liquid carbon dioxide is in contact with the tobacco, adequate penetration

12 of the tobacco by the liquid carbon dioxide is ensured.

5 The temperature of the liquid carbon dioxide should not, during this impregnation step, be permitted to exceed 31°C. since it must be in its liquid condition to be effective.

10 The pressure during the contacting step is preferably maintained at 600 to 900 psig by heating the vessel, for example using heating coils.

The tobacco and carbon dioxide may be maintained in contact under these conditions for a period of 0.5 to 60 minutes.

15 After the liquid carbon dioxide has been permitted to saturate the tobacco, generally for a total period of from 0.5 to 60 minutes and preferably from 2 to 4 minutes, any excess liquid carbon dioxide which may be present is drained out of the vessel, preferably while maintaining the conditions of temperature and pressure at the same levels as during the contacting step.

20 The pressure in the vessel is then released, by venting the gases in order to bring the contents of the vessel to atmospheric pressure. This venting should require from 0.75 to 50 minutes, depending on the size of the vessel but should preferably take no longer than 3 minutes, after which the temperature in the vessel will be from -85 to -95°C and the liquid carbon dioxide in the tobacco will be converted to solid carbon dioxide. The pressure need not be reduced to atmospheric, but need only be reduced below 60 psig. Obviously, this is not as preferable from a commercial viewpoint.

25 After the carbon dioxide in the tobacco is converted to its solid form, the solid carbon dioxide-containing tobacco is then exposed to expansion conditions by subjecting the treated product to heat or the equivalent in order to vaporize and remove the solid carbon dioxide from the tobacco. This may comprise the use of hot surfaces, or a stream of hot air, a mixture of gas and steam, or exposure to other energy sources such as radiant microwave energy or infrared radiation. A convenient means of expanding the solid carbon dioxide-containing tobacco is to place it or to entrain it in a stream of heated gas, such as superheated steam or to place it in a turbulent air stream maintained for example, at a pressure of from 0 to 10 psig and at a temperature of from 100° to 370°C, preferably 100° to 300°C, respectively 150° to 260°C, for 0.2 to 10 seconds. The impregnated tobacco may also be heated by being placed on a moving belt and exposed to infrared heating, by exposure in a cyclone dryer, by contact in a tower with superheated steam or a mixture of steam and air or the like. Any such contacting steps should not raise the temperature of the atmosphere with which the tobacco is in contact to above 370°C. and should preferably be from at 100°C. to

300°C., most preferably 150°C. to 260°C., when conducted at atmospheric pressure.

As is well known in processing of any organic matter, overheating can cause damage, first to color, such as undue darkening, and finally, to the extent of charring. The necessary and sufficient temperature and exposure time for expansion without such damage is a function of these two variables as well as the state of subdivision of the tobacco. Thus, to avoid undesirable damage in the heating step, the impregnated tobacco should not be exposed to the higher temperature levels, e.g. 370°C., for more than several tenths of a second.

One method for causing the expansion of the tobacco cells is to use the radiation methods described in either United States Patent 3,409,022 or 3,409,027. Another method involves the use of heat gun such as the Dayton heat gun or the equivalent, operating at an exit air temperature of 190-344°C. for a period of 0.2 second to 4 minutes, the shorter times, of course, being given for the higher temperatures. In this operation, the tobacco never attains a temperature above 140°C., being cooled by the rapid evolution of gases. The presence of steam during heating assists in obtaining optimum results.

Another system, usually preferred, is to use a dispersion dryer, for example, one that is supplied either with steam alone or in combination with air. An example of such a dryer is a Proctor & Schwartz PB dispersion dryer. The temperature in the dryer may range from 121 to 371°C. with contact time in the dryer of about 4 minutes at the lowest temperature to 0.1 to 0.2 second at the highest temperature. In general, a 0.1 to 0.2 second contact time is utilized when the hot gas temperature is 260-315°C. or somewhat higher. As stated before, other known types of heating means may be used as long as they are capable of causing the impregnated tobacco to expand without excessive darkening. It should be noted that where a high percentage of oxygen is present in the hot gases, it will contribute to darkening, so that if a hot-steam mixture is employed, a high proportion (e.g. over 80% volume) of steam is preferred. The presence of a steam atmosphere of 20% or more of the total hot gas composition aids in obtaining the best expansion.

The present process may be conducted in various forms of apparatus specific embodiments of which will be described in detail later in this specification.

It is important that the apparatus in which the liquid carbon dioxide-containing tobacco is converted to solid carbon dioxide-containing tobacco is able to contain gases at elevated pressures, as high as 1000 psig or more. This vessel is preferably employed for the initial contact of the liquid carbon dioxide with the



tobacco; however, such contact could be made in a separate vessel, if desired, and the liquid carbon dioxide-containing tobacco could then be placed in the pressure vessel. There may be numerous arrangements of the pressure vessel. However, there should preferably be a valved inlet from a source of liquid carbon dioxide and a valved outlet at the bottom of the vessel whereby liquid may be drained off; a second valved outlet near the top, for venting, may be added, and could be inserted as part of the inlet line, if desired, placed between the vessel, such as external heating coils, is necessary. Supporting the vessel on a load cell greatly simplifies measuring the carbon dioxide charge. A supplementary vessel similarly equipped with weighing means and heating coils is advantageous, though not essential, because it permits preheating a charge of liquid carbon dioxide from its usual low storage temperature of -20°C . (which may be about 215 psig). This arrangement helps to prevent the formation of solid carbon dioxide on charging the main vessel, and shortens the time the tobacco might be kept below the freezing point of its moisture. In operation, the filler may be placed in the main pressure vessel in a suitable holder such as a wire basket suspended above the bottom of the vessel. The closed vessel may then be purged with carbon dioxide gas and the outlets closed, then liquid carbon dioxide is introduced from storage, for example, at 250 psig, in an amount sufficient to cover all of the tobacco present in the vessel. The temperature is raised within 3 to 20 minutes by the heating means, e.g. heating coils, to bring the tobacco to at least -2°C . but less than 31°C . (the critical temperature of carbon dioxide) and this condition is preferably maintained for 1 to 20 minutes while impregnation takes place. Excess liquid carbon dioxide is then drained off by opening the lower outlet of the vessel to a suitable reservoir or the like disposal system, and when all excess liquid has been removed from the vessel, the vessel is vented to atmospheric pressure. The tobacco, which will now be generally contain from 5 to 25% of solid carbon dioxide by weight is then subjected to an operation to volatilize the solid carbon dioxide, preferably by removing the solid carbon dioxide-containing tobacco from the vessel and passing it through any of several rapid heating systems to achieve expansion. As indicated earlier in this specification, systems for this expansion process are most satisfactory which provide rapid, turbulent contact with the hot gas or vapor. With proper control of temperature and exposure time the product may be recovered in the expanded state at a desired moisture content such as 8 to 12% so that little or no reordering is required as it is with products from many expansion processes. Representing embodiments of apparatus

which can be used in the practice of the present invention are depicted in the drawings in which:—

Figure 1 is an elevational view with portions broken away of one form of apparatus in which tobacco can be expanded in accordance with the principles of the present invention.

Figure 2 is a sectional view as taken along the line II—II in Figure 1.

Figure 3 is an elevational view of a wire basket in which tobacco can be received, the basket in turn being received within the apparatus shown in Figure 1.

Figures 4 and 4a are respectively front and end elevational views and depict a somewhat different form of apparatus in which the pressure vessel is mounted for rotation about a fixed axis to facilitate intimate mixing of the liquid carbon dioxide and tobacco during impregnation as well as vaporization of the solid carbon dioxide during expansion of the tobacco.

Figure 5 is a further alternative form of apparatus in which a mixer unit is embodied in the pressure vessel to facilitate uniform distribution of the liquid carbon dioxide and intimate mixing thereof with the tobacco, the mixer shaft further being provided with a central passage and distribution ports for delivery of steam and/or hot air therethrough during the expansion step.

Throughout the following description like reference numerals are used to denote like parts in the drawing.

Referring now to Figures 1—3, there is depicted one form of apparatus 10 in which tobacco can be expanded in accordance with the principles of the present invention. The apparatus 10 includes a pressure vessel 12 fitted with a hinged cover 14 at the top, and provided further with ported connections 16 and 18 near the top as well as inlet and outlet lines 20 and 22 at the bottom which ported connections and lines allow for purging the interior of vessel 12 with a gaseous medium as well as for introducing the removing liquid carbon dioxide therefrom. The tobacco to be expanded can be loaded in the cages 24 (Figure 3) and the cages 24 in turn are removably received in a suitable rack structure 26 within the vessel 12 in any convenient arrangement, as for example in the circular array depicted in Figure 2. The cover 14 is then tightly closed on the vessel 12. The interior of the vessel then can be purged of any gaseous residue therein through the agency of gaseous carbon dioxide flow through inlet at line 20 and outlet through port 16. Line 20 is then closed and the vessel filled with liquid carbon dioxide through port 16, the unit being filled until the tobacco contained in the cage 24 is completely immersed in the liquid carbon dioxide, port 16 then being closed. The pressure inside the vessel

is then brought to a desired pressure range for example in the range of 450 to 850 psig. After a suitable soaking period, the liquid carbon dioxide is drained from the vessel (through line 22) and the interior of the vessel vented to atmosphere by opening port 18 to thus convert the liquid carbon dioxide remaining in the tobacco to a solid state. A steam and/or hot air mixture is then circulated through line 22 with continuous venting through the port 18 to effect expansion of the tobacco. On completion of the expansion operation, cover 14 is opened and the tobacco removed for such further processing as may be desired.

Another form of apparatus which can be used is shown in Figures 4 and 4a. The apparatus 40 includes a shell 42 showing an outer wall 52 and an inner wall 54 which define therebetween a jacketed chamber 56 in the pressure vessel. Further the pressure vessel 42 is journaled as at 48 to 50 on supports 44, 46 respectively for rotation about a fixed axis. Apparatus 40 also includes ports 60 and 62 provided for purging and liquid carbon dioxide filling purposes and a hinged cover 64. The jacketed chamber 56 is provided so that a heating medium can be circulated therethrough for maintaining the carbon dioxide in liquid form, e.g. at a temperature of -2°C . to 31°C . Further the jacketed chamber is provided to facilitate the evaporation step and the rotatable mounting of the vessel allows for intimate mixing of the tobacco and liquid carbon dioxide as well as enhancing vaporization by reason of the rotative movement causing the tobacco to contact the heated inner wall surface 54.

Figure 5 depicts a further form of apparatus 70 which includes an elongated preferably horizontal disposed pressure vessel 72 fitted with a number of ports 74, 76, 78, 80 used in connection with purging and liquid carbon dioxide supply and withdrawal, a filling hatch 82 and a withdrawal hatch 84 through which the tobacco can be moved from the vessel following expansion.

Externally disposed around the periphery of the pressure vessel 72 are a series of heating coils 86 which can be used in conjunction with raising the pressure of the liquid carbon dioxide but more particularly to maintain it at a temperature of between -2°C . and 31°C . during impregnation. The vessel 72 is also provided with mixing means 90 in the form of an elongated axially disposed shaft 92 carrying an arrangement of mixing vanes 94, with the shaft being provided further with an axial directed passage 96 and radial branches 98 communicating with the exterior surface of the shaft. Tobacco is introduced through filling hatch 82, and upon filling of the

vessel with tobacco, the hatch is closed. The interior of the vessel 72 is then purged with gaseous carbon dioxide through ports 76 and 80. After purging liquid carbon dioxide is introduced through port 80 and until suitable pressure (for example 600 psig) is reached. The shaft 92 is then rotated to facilitate achieving intimate mixing of the tobacco and liquid carbon dioxide. After a suitable contact time the mixing is stopped and the liquid carbon dioxide is drained from the vessel through port 78 and following, which the interior of the vessel is vented to atmosphere at through port 74 to convert the liquid carbon dioxide remaining in the tobacco to solid form. Steam and/or hot air is then introduced through the central passage 92 and the radial branches 98 of the shaft to effect expansion of the tobacco. Further the shaft is rotated to facilitate contact of the impregnated tobacco with the steam and/or hot air. Following the completion of the expansion operation, the tobacco is removed through hatch 84.

The following examples are illustrative:—

EXAMPLE 1

Ten pounds of bright tobacco particles of normal filler size having a moisture content of about 12% by weight was placed in a wire cage and placed in a pressure vessel. The vessel was then purged by running carbon dioxide gas through it at room temperature for a period of three minutes, after which carbon dioxide liquid was introduced into the vessel from a pressure container, where it had been maintained at a pressure of about 200 psig and a temperature of about -18°C . Sufficient liquid carbon dioxide was used to cover the tobacco sample. This comprised about 30 pounds of liquid carbon dioxide per pound of tobacco. The pressure in the vessel was then raised from 300 psig to about 700 psig by heating the vessel up to a temperature of 14°C . and this pressure was then maintained for about three minutes. Excess carbon dioxide liquid was removed from the tobacco by draining it off the tobacco, and the gas in the vessel was then vented rapidly from the vessel (vent time 45 seconds) whereby solid carbon dioxide was formed in the tobacco structure. Two samples of treated tobacco were taken. The first sample (about five pounds) was heated in a tower with hot air at 220°C . The second (about five pounds) was heated in a tower with superheated steam at a temperature of 220°C .

In each the percentage of oven volatiles (% O.V.) was measured. This is a measure of the moisture content plus a minor fraction of other components and is determined as follows:

(15)

$$\text{O.V. \%} = \frac{\text{weight loss of sample after 3 hrs. at } 100^{\circ}\text{C.}}{\text{sample weight}}$$

The filling power of each sample was also determined, as cc/10 g by the following method: 10 g of filler is placed in a graduated cylinder and subjected to a known load of 1860 g. The volume after depression is measured as a reflection of the filling power

of filler as used in cigarettes. A control consisting of 10 g of untreated tobacco particles taken from the same batch of bright tobacco particles as the pound of tobacco particles treated as described above.

The following results were obtained:

	Filling Power cc/100 grams	% O.V.	Corrected Filling Power at 12% O.V.
15 Sample Control	33	13.2	38
220°C.	78	8.3	51
Superheat Steam	97	8.5	71

20 It will be seen from the above data that the expansion due to the vaporization of carbon dioxide was considerable and that the low loss in O.V. decreases the need for a reordering tobacco step prior to using it in a smoking product and that superheated seems necessary for best expansion.

impregnated with carbon dioxide liquid as described in Example 1. A two pound sample of the treated tobacco was expanded by a saturated steam flow (at 166°C.) as described in Example 1. One pound samples of the unexpanded filler (control) and carbon dioxide-expanded filler were evaluated for moisture (O.V.) as in Example 1. The results of the analysis are set forth on the following page:

EXAMPLE 2

Five pounds of bright tobacco filler having a moisture content of 12% by weight was

	Control Sample	Test Sample Carbon Dioxide- Expanded
40 Analysis Moisture (O.V.)	12.5%	12.5%
Total Alkaloids*	2.1%	1.9%
45 Reducing Sugars*	12.1%	11.8%

*Analysis by method described in "Tobacco Science", Vol. 13, pp. 13—15 (1969).

EXAMPLE 3

50 It will be seen that the carbon dioxide-expanded filler shows little change from the control, and that the original composition of the tobacco is essentially preserved during the expansion process.

A series of 10 pound samples of bright tobacco filler having a moisture content of 12% by weight was impregnated with carbon dioxide liquid as described in Example 1 at pressures that varied from 400 to 900 psig. The impregnated filler was heated in a tower with superheated steam at 270°C.

The following results were obtained:—

	Pressure	% O.V.	Filling Power cc/10 grams	Corrected Filling Power at 12% O.V.
65	400	8.9	61.1	43.3
	500	8.6	69.3	45.9
	600	6.4	104.5	65.4
	700	6.0	114.8	68.0
	800	6.5	130.8	72.5
70	900	6.4	130.9	71.2

It can be seen that in order to obtain optimum product the pressure during impregnation should be above 600 psig and preferentially above 700 psig.

EXAMPLE 4

Another series of bright tobacco samples were impregnated as in Example 1. The vent time (time of gaseous pressure release) was

gradually increased from 49 seconds to 420 seconds. The impregnated filler was heated in a tower by superheated steam at 525°F. (270°C.). The results are listed below:

	Vent Time Seconds	% O.V.	Filling Power at Tower Exit O.V. cc/10 grams	Corrected Filling Power at 12% O.V.
10	49	3.8	121.2	65.5 (± 3.6)
	65	4.0	116.5	69.2
	148	4.1	95.9	60.0
15	195	4.4	112.7	72.1
	247	4.2	101.4	61.6
	420	4.6	116.7	67.7

The results indicate that the rate of pressure release does not affect product quality.

The soak times (time of contact of the filler with the carbon dioxide liquid) were varied from 60 seconds to 869 seconds. The impregnated filler was heated in a tower with superheated steam at 525°F. (270°C.) at a gas velocity of 100 feet/second.

The following results were obtained:

EXAMPLE 5

A third series of bright tobacco filler samples was impregnated as in Example 1.

	Soak Time seconds	% O.V.	Filling Power at Tower Exit O.V. cc/10 grams	Corrected Filling Power at 12% O.V.
30	60	3.5	109.3	59.7
	180	4.2	110.0	62.1
35	300	3.0	112.1	60.0
	600	3.2	117.3	62.2
	869	3.6	111.4	60.9

It can be seen from the above data that the expansion of the carbon dioxide impregnated filler is not changed by the length of contact with carbon dioxide liquid during the impregnation step.

In another series, input moistures were

varied from 8% to 19% O.V.—and the filler was impregnated as in Example 1. The impregnated filler was heated in a tower at 240°C. gas velocity of 100 feet/second in a saturated steam atmosphere.

The following results were obtained:

	Input % O.V.	Filling Power cc/10 grams	% O.V.	CV Correct to 12% O.V. cc/10 grams
50	8.0	57.1	5.8	37.2
	11.5	97.7	7.4	63.6
	13.4	127.6	7.8	67.3
55	16.5	124.9	7.6	71.2
	17.2	115.4	8.0	73.2
	19.0	83.3	9.6	67.3

The results indicate that the input moisture of filler should be above 8% and that the moistures of 13—19% are beneficial for good expansion with good tower exit moisture so that less "reordering" (rehumidification) is required.

The following experiment was also conducted.

EXPERIMENT 1

One pound of 12% O.V. bright cut filler was impregnated with carbon dioxide gas at a pressure of 900 psig. After an equilibration time of 10 minutes the gas was vented to the atmosphere and the same was removed. A portion of the filler was heated at 66°C. in a

low velocity air stream. A second portion was allowed to come to ambient temperature conditions (21°C.). The results are listed below:

	Filling Power Temperature	cc/10 grams
Sample 1	66°C.	37
Sample 2	21°C.	34
Control	—	32

The results indicate that the immersion of the filler in the carbon dioxide liquid seems necessary and that the entrapment of carbon dioxide under pressure followed by the pressure release does not exert sufficient force to effect expansion.

WHAT WE CLAIM IS:—

1. A process for expanding tobacco which comprises (1) contacting tobacco with liquid carbon dioxide under conditions such that the temperature of the tobacco is maintained at a level no lower than -2°C . and such that substantially all of the liquid carbon dioxide is maintained in liquid form to impregnate the tobacco with the liquid carbon dioxide, (2) subjecting the impregnated tobacco to conditions such that the liquid carbon dioxide is converted to solid carbon dioxide and (3) thereafter subjecting the solid carbon dioxide-containing tobacco to conditions whereby the solid carbon dioxide is vaporised to expand the tobacco.
2. A process according to claim 1 wherein tobacco containing from 8 to 50% by weight of water is contacted in step (1) with at least 1 part, by weight per part of tobacco, of liquid carbon dioxide.
3. A process according to claim 2 wherein 5 to 10 parts by weight of liquid carbon dioxide are used per part of tobacco.
4. A process according to claim 1, 2 or 3 wherein the tobacco is contacted in step (1) with liquid carbon dioxide at a pressure of from 490 to 906 psig and a temperature of from -2 to 31°C . for from 0.5 to 60 minutes.
5. A process according to any one of claims 1 to 4 wherein the solid carbon dioxide-containing tobacco is subjected in step (3) to a pressure of from 0 to 10 psig and a temperature of from 100 to 370°C . for from 0.2 to 10 seconds to vaporise the solid carbon dioxide.
6. A process for expanding tobacco according to claim 1 substantially as described in any one of the Examples.
7. Tobacco when expanded by a process as claimed in any one of the preceding claims.
8. Apparatus for expanding tobacco which comprises a pressure vessel, means for introducing and withdrawing tobacco from the pressure vessel, means for introducing liquid carbon dioxide into the pressure vessel to impregnate tobacco contained therein, means for withdrawing liquid carbon dioxide from the pressure vessel after impregnation of said tobacco, means for maintaining carbon dioxide at predetermined temperature and pressure in the pressure vessel whereby it remains liquid during the impregnation, means for converting liquid carbon dioxide impregnating said tobacco into solid carbon dioxide, and means for vapourising solid carbon dioxide impregnating said tobacco to effect expansion of said tobacco.
9. Apparatus according to claim 8 in which the means for maintaining carbon dioxide in the liquid state includes temperature control means effective to maintain liquid carbon dioxide at a temperature of between -2°C . and 31°C .
10. Apparatus according to claim 9 in which said temperature control means comprises coils disposed externally on said pressure vessel.
11. Apparatus according to claim 9 or 10 in which the means for converting liquid carbon dioxide impregnating said tobacco to solid carbon dioxide comprises means for venting the pressure vessel to the atmosphere.
12. Apparatus according to any one of claims 8 to 11 further comprising means for purging the interior of said pressure vessel with a gaseous purging agent to remove undesirable gaseous residue therefrom.
13. Apparatus according to any one of claims 8 to 12 further comprising a plurality of tobacco receivable receptacles, and means in said vessel for removably receiving said receptacles in said vessel.
14. Apparatus according to any one of claims 8 to 13 which said pressure vessel has inner and outer walls defining a jacketed structure therebetween, means for vaporising said solid carbon dioxide comprising a heated medium circulated in said jacket.
15. Apparatus according to claim 14 in which said pressure vessel is rotatably supported for rotation about a fixed axis whereby said vessel can be rotated to intimately mix said liquid carbon dioxide with said tobacco and during the vaporisation of said solid carbon dioxide to facilitate contact of said tobacco with said inner wall.
16. Apparatus according to any one of claims 8 to 15 in which said pressure vessel is an elongated structure, there further being provided mixing means within said pressure vessel operable to effect intimate mixing of said liquid carbon dioxide with said tobacco and in cooperation with said vaporising means to facilitate expansion of said tobacco.
17. Apparatus according to claim 16 in which said mixing means comprises an elongated shaft extending interiorly of and rotatably within said pressure vessel, said shaft being fitted with mixing vanes fixed thereto.
18. Apparatus according to claim 17 in which said shaft is provided with an axial directed passage and radial branches opening at the surface of said shaft through which a heating medium can be flowed into contact



(18)

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with said tobacco to effect vapourisation of said solid carbon dioxide.

19. Apparatus for expanding tobacco according to claim 8 substantially as described herein with reference to and as illustrated in the accompanying drawings.
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