United States Patent [19]

Egri

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[54] **TOBACCO DENICOTINIZATION PROCESS**

[75] Inventor: **Laszlo Egri,** Basel, Switzerland

[73] Assignee: **Tamag Basel AG,** Birsfelden, Switzerland

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[21] Appl. No.: **195,960**

Related U.S. Application Data

[63] Continuation-in-part of Ser. No. 823,508, May 9, 1969, abandoned.

[30] **Foreign Application Priority Data**

May 13, 1968 Switzerland 7072/68

[52] **U.S. Cl.** **131/143**

[51] **Int. Cl.** **A24b 15/02**

[58] **Field of Search** 131/143, 140

[56] **References Cited**

UNITED STATES PATENTS

1,671,259	5/1928	Schloesing	131/146
3,139,435	6/1964	Staley et al.	131/143 X
3,399,680	9/1968	Egri	131/134

FOREIGN PATENTS OR APPLICATIONS

714,867	8/1965	Canada	131/143

Primary Examiner—Melvin D. Rein

Attorney, Agent, or Firm—Werner W. Kleeman

[57] **ABSTRACT**

A method for denicotinizing and/or mellowing tobacco by treating the tobacco with a gaseous treatment medium containing at least water vapor and/or at least ammonia wherein there is contemplated controlling the process conditions, especially when working with an ammonia-containing treatment medium controlling the quantity of ammonia added thereto and, during an exclusive or subsequent treatment with an ammonia-free water vapor-containing treatment medium controlling the termination of the tobacco treatment on the basis of the pH-value and/or the titration value of the condensate of the treatment medium leaving the treated tobacco.

28 Claims, 6 Drawing Figures

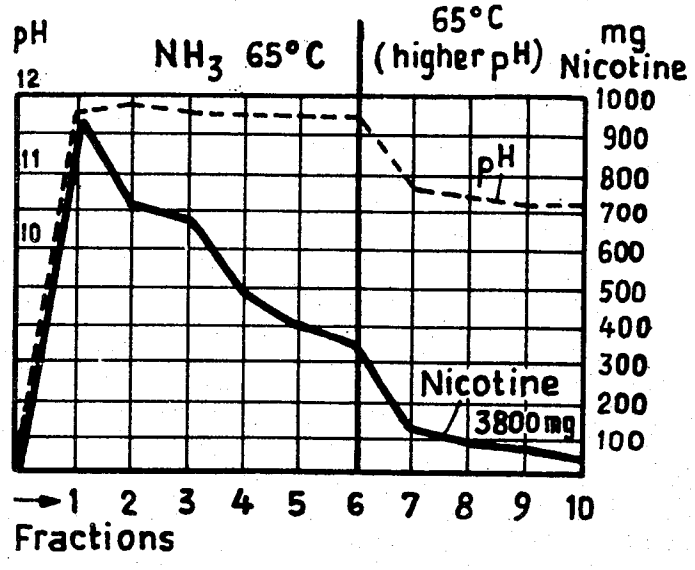


Fig. 1a

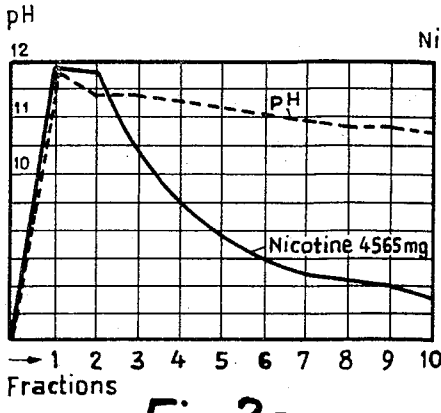


Fig. 1b

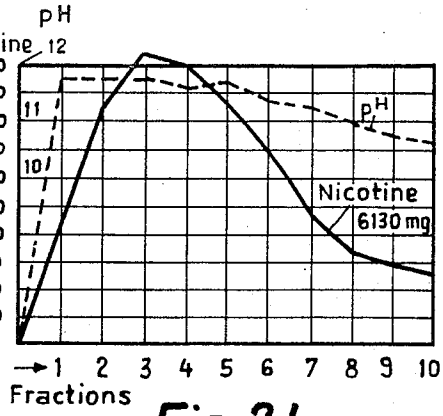


Fig. 2a

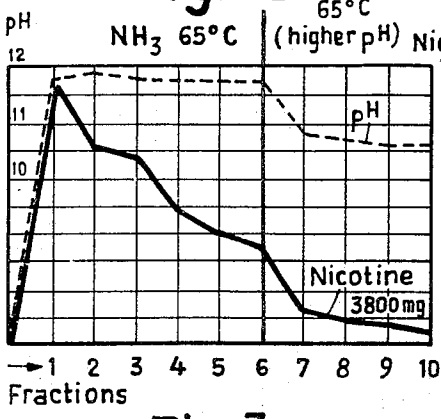


Fig. 2b

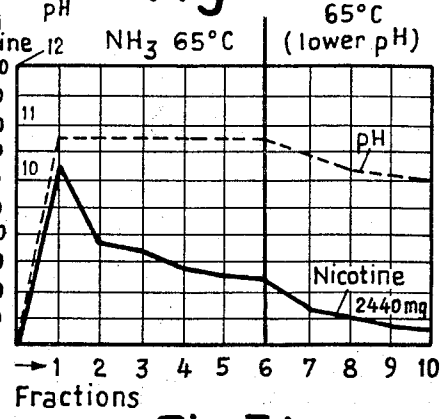


Fig. 3a

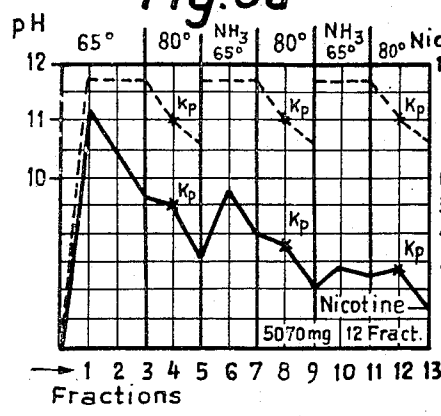
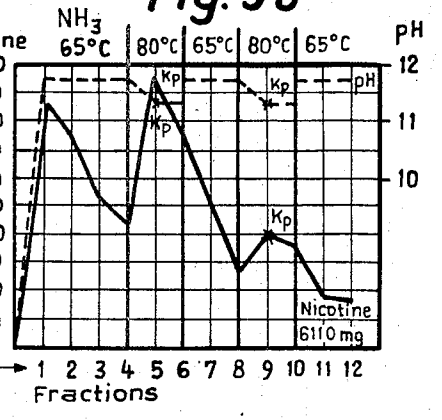


Fig. 3b



TOBACCO DENICOTINIZATION PROCESS

CROSS-REFERENCE TO RELATED APPLICATION

This is a continuation-in-part application of my commonly assigned, copending United States application, Ser. No. 823,508, filed May 9, 1969, now abandoned and entitled METHOD FOR TREATING TOBACCO.

BACKGROUND OF THE INVENTION

The present invention relates to a new and improved method for treating tobacco, and especially concerns itself with the denicotinizing and/or mellowing of tobacco.

By virtue of the teaching set forth in U.S. Pat. No. 896,124, granted Aug. 18, 1908, and listing as the inventors G. B. Lindenberger and R. B. Arnold, it has been known for quite some time that it is possible to reduce the nicotine content of tobacco by treating such with a gaseous medium containing, if desired, steam, treatment preferably taking place at elevated temperatures.

However, it was not long until it was determined that when utilizing this technique, in principle constituting a steam distillation technique, it was possible to only remove a very slight proportion of the nicotine present in the tobacco, namely the nicotine existing in the form of a free base.

Therefore, as for instance disclosed in the U.S. Pat. No. 999,674 granted on Aug. 1, 1911 to J. Sartig and U.S. Pat. No. 1,671,259, of Schloesing, attempts were made with a certain degree of success to free the nicotine from the nicotine salts by moistening the tobacco with an ammonia solution prior to the steam treatment, thereby obtaining a more extensive denicotinization. However, this technique is neither economically completely satisfactory nor does it render possible sufficient denicotinization of the tobacco.

Even if a practically complete separation of the nicotine, as a general rule, is only then of decisive importance if there is to be obtained as the primary product nicotine and not a tobacco suitable for smoking having a reduced nicotine content, still, with these techniques what is disturbing is the limits which can be drawn with regard to the thereby obtainable degree of denicotinization, especially the frequently occurring loss in aroma of the treated tobacco.

A more efficient method in this connection is, for instance, taught in an Austrian Pat. No. 147,672 and the previously mentioned U.S. Pat. No. 1,671,259. When working with these known procedures ammonia in gaseous form is added to the steam- or water vapor-containing treatment medium. However, apart from the difficulties in working at optimum conditions throughout all phases of the treatment, this technique is associated with the decisive disadvantage that the obtained tobacco product is, in certain instances, so "sharp", that is, produces such a sharp and bitter or acrid tasting smoke, that as a practical matter it can not be used as smoking tobacco.

As has become known to the art, for instance, from U.S. Pat. No. 2,136,485, granted Nov. 15, 1938 to F. Berka and A. Giller, attempts have been made, while using a treatment medium containing steam and ammonia gas, to free the denicotinized tobacco from excess

ammonia by subjecting such to an aftertreatment with an ammonia-free treatment medium, as a general rule containing steam.

Due to such type aftertreatment or post-treatment there is obtained a mellowing of the tobacco, which in any event is absolutely necessary for tobacco which has been denicotinized by a treatment medium containing ammonia, and sometimes however also for untreated tobacco which is particularly sharp, in order to obtain a marketable product, that is, a tobacco which will not be rejected by the smoker as too sharp.

Unfortunately, when resorting to such mellowing treatments, this likewise being the case for all of the previously mentioned denicotinizing techniques, there results a more or less pronounced impairment of the taste and/or aroma of the treated tobacco, the degree of which, similar to the denicotinizing- and/or mellowing effect strived for by such treatment, is dependent upon numerous process parameters which cannot be exactly predetermined, especially upon temperature, pressure, composition of the treatment medium, the treatment duration, the type of tobacco treated, and the apparatus employed in the treatment.

The resultant danger of too intensive treatment, in other words overtreatment, oftentimes producing a "strawy" or "flat" tobacco, has been attempted to be mitigated through the use of treatment conditions which are as protective as possible, for instance by working at reduced pressure and at temperatures which are as low as possible. While this produced certain improvements such did not however provide a solution of the problem which could really be considered satisfactory in practice. One of the primary difficulties in the selection of suitable parameters and conditions for the denicotinizing and/or in particular tobacco mellowing previously resided in the fact that during treatment, while it was possible to determine relatively quickly the degree of denicotinization reached at any given period of time by knowing the starting nicotine content and the quantity of tobacco processed as well as by determining rather rapidly up to a certain point during the course of treatment the quantity of extracted nicotine, still it was not possible to determine the stage of treatment which had been reached for the treated tobacco as concerns its sharpness and other taste-and aroma properties. The latter situation was particularly extremely disturbing, since it was not possible to reliably select on the basis of experience suitable values for the treatment conditions, especially an appropriate treatment duration, because identical treatment conditions produced non-predictable differing results and because tobacco as a product of nature possesses different properties from one supply to the next, even in fact at times from one bale to the next.

The taste characteristics of tobacco at a certain period of time during treatment for a long time previously could only be determined by organoleptical checking of a test sample removed at that given period of time and checked by very time-consuming test procedures, as well as in certain cases by carrying out a pH-value measurement of the smoke condensate obtained by smoking special test cigarettes or cigars in complicated smoking machines and which test cigarettes or cigars were produced from samples of the tobacco. In other words, methods had to be resorted to which only gave results after a long time delay, so that as a practical matter these techniques could not be seriously consid-

ered for controlling the treatment of tobacco for denicotinization and/or mellowing purposes.

OBJECTS OF THE INVENTION

Hence, it is a primary object of the present invention to provide a new and improved method for the denicotinization and/or mellowing of tobacco suitable for smoking purposes, by treating the tobacco with a gaseous treatment medium containing steam and/or ammonia, wherein on the basis of certain characteristic values which can be easily and rapidly determined the treatment conditions can be controlled in such a manner that:

a. denicotinization can be optimumly carried out, that is, as rapidly as possible until reaching a desired degree of denicotinization and under the best possible utilization of the active component(s) of the treatment medium and/or

b. mellowing is carried out protectively, effectively and while preventing overtreatment and providing reproducible results.

A further significant object of the present invention is to provide a new and improved method of treating tobacco and, in particular, controlling the mellowing thereof, such that the tobacco products obtained through the practice of the inventive method posesses a much better quality than tobacco obtained by treatment techniques wherein mellowing is not controlled.

A further object of this invention is to provide a method of treating tobacco wherein the addition of ammonia is controlled during denicotinization in a manner resulting in a reduction in the ammonia consumption and increased economies in the treatment process.

Still a further significant object of the present invention resides in treating tobacco in a manner providing a decrease in the consumption of steam during mellowing of the tobacco and also affording reproducible results, so that the treated tobacco products even from one charge to the next, are relatively uniform in quality as desirably required for marketing purposes.

SUMMARY OF THE INVENTION

Now it has been surprisingly found that a certain correlation exists between the pH-value and/or the titratable base content of the condensate of the treatment medium leaving the treated tobacco and the course of the treatment, as well as also with regard to the efficiency of denicotinization and the characteristics of the treated tobacco. This correlation renders it possible to determine or follow the course of the treatment on the basis of relatively simple and quickly ascertainable analysis data, namely the pH-value and/or the titration value of the condensate of the treatment medium leaving the treated tobacco and thereby appropriately controlling the actual treatment.

Accordingly, the invention proposes an improved method for the denicotinization and/or mellowing of tobacco by treating the tobacco with a treatment medium containing at least steam and/or at least ammonia and steam in that, during treatment there is determined either continuously or periodically the pH-value and/or the titration value of the condensate of the treatment medium leaving the treated tobacco, and treatment of the tobacco on the basis of the determined data is controlled in such a manner that:

a. during an eventual treatment with an ammonia containing treatment medium, the treatment conditions, especially the introduction of the ammonia, is regulated in such a fashion that the pH-value of the condensate is between approximately 10.5 and 12 and/or the titration value of a 10 ml sample of the condensate is between approximately 45 ml and approximately 80 ml 0.1 n H_2SO_4, and/or

b. In the event mellowing is undertaken by treatment with an ammonia-free treatment medium, the initially the tobacco is treated under defined conditions, especially with regard to temperature, flow velocity, and pressure of the treatment medium, with an ammonia-free steam or water vapor-containing treatment medium, and the pH-value and/or the titration value of the condensate, obtained by condensing the treatment medium, is determined as soon as possible following the establishment of stable conditions, (these values constituting the reference-pH and reference titration value). The sole or last treatment with an ammonia-free, steam-containing treatment medium is then terminated when, under the treatment conditions employed for the determination of the reference pH value and/or reference titration value, the pH-value of the condensate is in a range of approximately 1.0 unit of pH value beneath the reference pH value as determined above up to approximately 0.5 unit of pH value above such reference pH and/or the titration value amounts to between 70 and 110 percent of the reference titration value, and further, under the condition that, if the tobacco after the determination of the reference pH value and/or the reference titration value as explained above, has not been treated at least one time with an ammonia containing treatment medium, then treatment is interrupted at the earliest if, under the same treatment conditions as prevailed for the determination of the reference values, the pH-value of the condensate is at least approximately 0.1 unit of pH less than the reference pH value and/or the titration value of the condensate is at least approximately 5 percent smaller than the reference titration value.

The titration values refer to the condensate emanating from approximately 100 kg tobacco after throughpassage of the treatment medium at approximately 60°C – 80°C and with a velocity of 1–2 kg/min.

BRIEF DESCRIPTION OF THE DRAWINGS

The invention will be better understood and objects other than those set forth above will become apparent when consideration is given to the following detailed description thereof.

FIGS. *1a*, *1b*, *2a*, *2b*, *3a* and *3b* are respective graphs serving to elucidate the method aspects of the invention in the practice of treating tobacco as taught herein.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS OF THE PRESENT INVENTION

As will be recognized from the preceding discussion, the invention relates to an improvement in the method of treating tobacco for consumption purposes, wherein the tobacco is subjected to a treatment producing either only denicotinization, only mellowing, or, however, both denicotinization as well as mellowing, at least to a degree beneath the increased degree of tobacco sharpness which is unavoidable when carrying

out denicotinization with an ammonia-containing treatment medium.

Tobacco Denicotinization

As will be more fully appreciated from the various illustrative examples given hereinafter and the comparative tests, it has been found that a better, that is to say, a more rapid denicotinization can be attained if, while using the otherwise similar treatment conditions, initially a treatment medium containing ammonia and steam or water vapor, and then following a certain amount of time a steam-containing, ammonia-free treatment medium is permitted to flow through the tobacco, instead of allowing for a certain period of time initially a stationary steam-containing and ammonia-saturated atmosphere to act upon the tobacco to be treated and then to treat such with a flowing steam-containing, ammonia-free treatment medium.

Furthermore, it has been found that the speed of denicotinization, which of course more or less rapidly drops during the course of the treatment as a function of the decreasing nicotine content of the treated tobacco, among other things is considerably dependent upon the concentration of the ammonia in the treatment medium. Whereas, however, the prior publications in this particular art, as concerns the dosage of ammonia, previously merely proposed roughly using approximately a 2 to 3-fold excess of ammonia, based upon the quantity of ammonia required for freeing the bound nicotine in the tobacco present in the form of salts, it has been determined in accordance with the teachings of this invention, as likewise will be more fully demonstrated by the hereinafter given examples and comparative tests, that such a rough control of the denicotinization with an ammonia-containing treatment medium does not reliably lead to a treatment which is as economical as possible, wherein the employed ammonia is optimumly utilized and for the prevailing remaining operating conditions results in the best possible denicotinization speed.

Instead, it has been found that in order to attain an optimum control of the process during denicotinization, it is much more the case that it is important to continuously or from time to time determine the pH-value of the condensate of the treatment medium leaving the tobacco undergoing treatment and/or the titratable base content of the aforementioned condensate and with other constant process conditions, such as temperature, pressure, flow velocity of the treatment medium and so forth, to regulate dosing of the ammonia in such a way that the pH-value of the condensate is between approximately 11.0 and 12.0, preferably between approximately 11.2 and 11.8. Denicotinization occurs especially favorably then if the pH-value of the condensate is in a range of about 11.5 to 11.8, and especially then if such pH-value is preferably at approximately 11.7. As has been determined by experiments, the denicotinization speed for condensate pH-values of less than approximately 11.6 to 11.7 always increasingly drops, whereas pH-values greater than approximately 11.7 can only be achieved through an extraordinarily high increase in the ammonia concentration of the treatment medium, without thereby, however, achieving a further increase or at least any appreciable further increase in the denicotinization speed. Instead of, or in addition to, the pH-value of the condensate it is also possible to determine its titration value and to use such for controlling the treatment conditions during denicotinization, especially to make use of such titration value for controlling the dosage of ammonia. However, the determination of the titration value is much more difficult to arrive at and much more time-consuming than the determination of the pH-value which can be continuously carried out with conventional pH-measuring devices. Furthermore, the increased accuracy of the titration value as well as the thereby more exact control possibility of the treatment conditions during denicotinization is not absolutely necessary in order to obtain a sufficiently economical process, so that the control of the treatment during tobacco denicotinization is preferably undertaken merely as a function of the pH-value of the condensate. However, by way of completeness in the disclosure of the method aspects of this development, the titration value ranges, within which it is possible to practice the teachings of the present invention process, will be given. As already explained, during denicotinization the titration value of a 10 ml condensate sample should be in the range of 45 to 80 ml 0.1 normal sulfuric acid (H_2SO_4). It is preferable to operate under conditions at which this titration value is in a range of about 50 to 60 ml 0.1 normal sulfuric acid.

Finally, with respect to the denicotinization operation it has been found that the average denicotinization speed and therefore the degree of total denicotinization which can be obtained with a denicotinization treatment extending over a certain time period can be considerably increased if, instead of carrying out a "continuous" treatment, that is a treatment under essentially constant conditions, wherein if desired there can be carried out a subsequent treatment with an ammonia-free, steam-containing treatment medium, there is carried out a so-called "pulsating" treatment, wherein from time to time the temperature of the treatment medium is briefly increased for short intervals. With the inventive preferred employed pulsating treatment it is especially advantageous, during the treatment phases at relatively low temperatures, to add ammonia to the treatment medium, the ammonia being absorbed much better by the tobacco undergoing treatment at the relatively low temperatures than at the higher temperatures, and therefore to provide for a more complete and effective utilization thereof. Further, during the treatment phases undertaken at the higher temperatures there is advantageously introduced into the treatment zone an essentially ammonia-free, steam-containing treatment medium. The so-called pulsating treatment is especially then advantageous if it is desired to obtain as the essential or singular product not nicotine, rather a tobacco suitable for smoking purposes, since in this case it is especially advantageous to maintain the total treatment time as short as possible and on the average to work with conditions which are as mild or protective as possible, that is to say, to especially work at as low an average temperature as possible in order to maintain losses in aroma of the tobacco as low as possible which cannot be avoided in any event when carrying out such treatment.

As concerns the duration of the individual treatment steps or phases during such pulsating treatment as well as the most favorable increase of the thus to be employed temperature increments or jumps there cannot be really formulated any generally valid rules, since the course of the treatment in this case is dependent upon too many conditions. However, the course of the pH-

value curves, especially in combination with the denicotinization curves which can be determined by employing known techniques, and as will be more fully explained in detail hereinafter in conjunction with the examples, nonetheless provides a certain guideline or starting point, whereby especially the transition from a high temperature treatment phase to the next lower temperature treatment phase can be determined on the basis of the pH-value curve of the condensate, since, as will be appreciated from what will be explained hereinafter, from the pH-value curves portrayed in FIGS. 4*a* and 4*b*, during a pulsating denicotinization treatment during the high temperature phases there can more or less always be ascertained a rather pronounced curved knick or bend, from which point on the initially relatively rapid dropping of the pH-value slows down, whereby approximately at the same time there is a reduction in the denicotinization speed. Experience has shown that it is particularly advantageous to interrupt the high temperature phase and to switch over to a new lower temperature phase when, following the occurrence of the previously mentioned bend in the curve of the pH-value, approximately the same time has expired as existed between the beginning of the relevant high temperature phase and the occurrence of such bend in the pH-value curve.

With the method of the invention in order to obtain as intensive as possible treatment effect one should work at as high temperatures as possible, whereas in consideration of providing as protective as possible treatment of the tobacco material and in order to obtain optimum utilization of the employed ammonia the treatment should be carried out at the lowest possible temperatures.

In practice one should not fall below a treatment temperature which is in the range of 40°C to 50°C so as to prevent excessively long treatment times. On the other hand, at least then when treating especially sensitive and aromatic tobaccos a maximum temperature of approximately 100°C should not be reached, or at best only briefly, in order to prevent damage to the treated tobacco. With the inventive preferred pulsating treatment one operates preferably at a lower temperature in the range of about 55°C to 65°C and an upper temperature in the range of about 75°C to 95°C, whereby, however, especially then if the treated tobacco is to be after-fermented, an upper temperature range of approximately 75°C to 85°C should not be exceeded in order not to destroy the enzyme activity required for after-fermentation.

Mellowing of the Tobacco

With so-called mellowing of tobacco, that is to say, during the treatment of a tobacco which by its very nature is too sharp or acrid or which has become too sharp by virtue of having been subjected to a denicotinization treatment with a treatment medium containing ammonia, and wherein the previously mentioned tobacco mellowing is carried out at increased temperature with an ammonia-free treatment medium containing at least water vapor, the absence of any quickly determinable objective measurement value enabling monitoring and controlling the course of the treatment, and in particular the determination of the optimum point of time for the interruption of the treatment is particularly grave. This is so because the marketability of smoking tobacco is considerably dependent upon the taste- and aroma characteristics of the tobacco product. Since, as already described and discussed above, previously there was not known any analysis value which could possibly provide such control of the treatment process and suitable treatment conditions also could not be determined on the basis of values gained from experience, since both the treatment of one and the same tobacco batches or portions in different installations as well as also the treatment of different tobacco batches or portions in one and the same installation proceeds differently as a general rule, the mellowing of tobacco through treatment with an ammonia-free at least steam or water vapor-containing treatment medium previously time and again always resulted in unpredictable and oftentimes unusable results. The prior art theoretically taught the possibility for each tobacco batch or portion to be mellowed to carry out in an industrial installation utilized for this purpose a full-scale run for such length of time until the treated tobacco was surely overtreated and therefore strawy or flat, and then at brief time intervals to remove samples of the tobacco material which then could be tested with regard to their taste characteristics.

Surprisingly, it has now been found on the basis of the teachings of this invention that a direct correlation exists between the taste characteristics of the treated tobacco and the base content of the treatment medium leaving the tobacco which allows controlling the treatment, independently of the installation in which the tobacco is treated, on the basis of easily determinable analytic data. This extremely advantageous result can be realized in that the pH-value and/or the titration value, that is, a comparison is made between the required ml 0.1 n H_2SO_4 needed to neutralize 10 ml condensate of treatment medium departing from the tobacco at the end of treatment with that value of treatment medium condensate which under the same treatment conditions, especially with respect to temperature, pressure, flow velocity of the treatment medium and its content of water vapor, departs from the tobacco at the beginning of the treatment, and specifically directly after constant conditions have been established or regulated.

The pH-value which is a measure for the type and quantity of the bases which are contained in the treatment medium and which can be easily continuously measured is thus chosen as the primary criteria for termination of the treatment. If the critical pH-value has been reached, then, — as the second criteria for the final determination of the terminal point of treatment — there may be determined at short time intervals the titration value of the condensate which is a measure for the total content of the condensate of titratable basis, and similar to the pH-value, can be compared with the corresponding starting values, as above explained.

If there is desired to be obtained as the product produced in accordance with the inventive method a tobacco which is milder than the untreated tobacco, in other words if for example a tobacco which by its very nature is too sharp is to be mellowed by treatment with a water vapor-containing treatment medium, then, the pH-value of the condensate of the treatment medium leaving the tobacco directly prior to interruption of treatment, as already previously mentioned, must be smaller by at least 0.1 than the so-called "reference-pH-value", which it will be recalled corresponds to the pH-value of the treatment medium condensate leaving the treated tobacco immediately following the start of

treatment. In order to prevent overtreatment of the tobacco, on the other hand treatment should be interrupted before the pH-value of the condensate is more than 1.0 below the reference-pH-value. It is preferable to terminate the treatment when the condensate pH-value is below the reference pH-value by approximately 0.2 to approximately 0.5. Extremely favorable results can be obtained as a general rule if the condensate pH-value at the point of time of termination of the treatment is approximately 0.3 to 0.4 below the reference pH-value.

On the other hand, if a tobacco which by its very nature is sufficiently mild or possibly even too mild is initially denicotinized through the use of an ammonia-containing treatment medium and owing to such treatment the tobacco becomes sharper than the untreated tobacco, so that following denicotinization the tobacco must again be mellowed by treating same with an ammonia-free steam-containing treatment medium, then with the method of the invention the condensate pH-value at the point of time of interruption of the mellowing treatment can be above the reference pH-value up to an amount approximating 0.5 units of pH. When carrying out this embodiment of the invention the treatment is preferably only then interrupted when the condensate pH-value is at least at a value which at most is greater by 0.2 units than the reference pH-value, and especially has dropped to a value which at most is the same as the reference pH-value and preferably up to a value of 0.3 units below the reference pH-value. For the termination of the treatment at a pH-value of the condensate which is below the reference pH-value for this embodiment of the invention the same rules and the same pH-value ranges are applicable as was the case for the previously discussed embodiment of the invention where the mellowing is not proceeded by any denicotinization while using an ammonia-containing treatment medium.

A measure for the base concentration of the treatment medium leaving the treated tobacco is the titration value of the condensate, that is the amount of 0.1 normal sulfuric acid which is necessary for neutralizing 10 ml condensate. (Since for the purposes of the invention the titration value is always compared with the so-called "reference titration value", that is to say, the titration value of a condensate sample which has been obtained during discharge from the tobacco immediately after establishment or adjustment of stable conditions in the treatment system during treatment of the raw tobacco material with an ammonia-free, steam-containing treatment medium, it is of course possible to employ for the determination of the titration value also greater or smaller condensation samples, strong acids other than sulfuric acid and titration solutions with other acid concentrations.) Since the determination of the titration value is somewhat more cumbersome than the determination of the pH-value, it is preferable when practicing the method aspects of the invention to continuously measure the pH-value, while the titration value is then only measured during critical phases of treatment, that is, particularly then when from the pH-value there can be recognized that the desired treatment condition of the tobacco undergoing treatment soon will be reached, the titration value then being determined at short-time intervals.

As previously explained, with the inventive embodiment of the method of this development, wherein a tobacco which by its very nature is too sharp is essentially only mellowed by treatment with an ammonia-free, steam or water vapor-containing treatment medium, treatment is terminated when the titration value is beneath the reference titration value by approximately 10 to approximately 30 percent preferably by approximately 15 to 25 percent and in particular about 20 percent below the reference titration value.

With the previously likewise discussed preferred embodiment of the invention in which a tobacco which is to be treated is initially denicotinized by treatment with an ammonia-containing treatment medium and therefore automatically rendered sharper, as already explained and depending upon the type of tobacco, treatment is terminated when the titration value during the closing mellowing treatment with an ammonia-free, steam-containing treatment medium at least has dropped to a value which is at most approximately 70 percent of, and preferably at most approximately 110 percent of the reference titration value. For the interruption of the treatment with titration values which are the same as the reference titration value or below the reference titration value there are applicable the values previously mentioned for the embodiments of the inventive method serving essentially only for mellowing of the tobacco.

Measures for Attaining as Uniform and Protective Treatment of the Tobacco as Possible

In order to obtain as protective as possible treatment of tobacco which is sensitive to high temperatures during the practice of the method aspects of the invention the tobacco to be treated, preferably while subjected to reduced pressure, especially at a pressure in the range of approximately 120 to 700 mm. mercury pressure (Torr), it has been found to be advantageous to vary from time to time the pressure prevailing in the treatment space of compartment during treatment, for instance in that a valve leading to the suction line is periodically closed in each instance for 5 to 10 minutes and then again opened for approximately 10 to 20 minutes.

EXAMPLES

The examples and comparative tests given hereinafter further serve to explain this invention, yet it should be understood that the invention itself is not to be considered limited to the details and numerical values given in the examples which are here presented purely by way of illustration and not limitation.

Example 1

Since publications have reported about the recirculation of the "excess" ammonia through the tobacco, which is comparable to a predetermined, required reaction time for freeing the nicotine of its salts, comparative tests were initially carried out in order to determine whether a longer exposure time of the gases is necessary or whether there can be obtained the same quantity of nicotine directly after passing the ammonia through the tobacco.

2,000 grams loose tobacco leaves were subdivided into two equal parts, and

a. 1,000 grams introduced into a vacuum apparatus. After evacuation to 150 mm Hg pressure (Torr) there was introduced water vapor and 30 grams of ammonia and such was permitted to stand for 15 minutes. Thereafter, the tobacco was steamed-out for 30 minutes under vacuum conditions at 60° C. Every 3 minutes

fractions were removed and analyzed. The pH-value and the nicotine content of the condensate was determined.

b. In case *b* the other half of the tobacco was immediately steamed-out under vacuum conditions at 60° C, and during the first 15 minutes there was simultaneously continuously introduced 30 grams of NH_3. In both cases the moisture content of the tobacco amounted to 15 percent. Here also during 30 minutes (plus the 15 minutes required during procedure *a* for the reaction time) fractions were removed every 3 minutes and analyzed.

The results of these tests have been charted in the following Tables I(a) and I(b) and have been graphically illustrated in FIGS. **1***a* and **1***b*, respectively.

TABLE I(a)

Treatment: Denicotinization during different effective times of the gas, 15 minutes permitted to stand in a saturated NH_3 atmosphere 30 minutes steaming-out, treatment temperature 65° C.
Tobacco Type Treated: Geudertheimer HGT 65 leaf tips
Quantity: 1 kg.

Fraction	Time Min.	Quantity ml	Temp. °C	Condensate pH	Nicotine (mg/500 ml)
1	3	500	65	11.7	975
2	6	500	65	11.2	960
3	9	500	65	11.2	670
4	12	500	65	11.1	490
5	15	500	65	11.0	365
6	18	500	65	10.9	295
7	21	500	65	10.8	245
8	24	500	65	10.7	210
9	27	500	65	10.7	195
10	30	500	65	10.6	160
					4565

TABLE I(b)

Treatment: Denicotinization during different effective times of the gas, 15 minutes treatment with water vapor and with NH_3 30 minutes steaming-out, treatment temperature 65°C.
Tobacco Type Treated: Geudertheimer HGT 65 leaf tips.
Quantity: 1 kg.

Fraction	Time Min.	Quantity ml	Temp. °C	Condensate pH	Nicotine (mg/500 ml)	Comments
1	3	500	65	11.7	388	
2	6	500	65	11.7	835	
3	9	500	65	11.7	1025	
4	12	500	65	11.6	974	1–5
5	15	500	65	11.7	859	4080
6	18	500	65	11.3	694	
7	21	500	65	11.2	460	
8	24	500	65	11.0	337	
9	27	500	65	10.8	277	1–10
10	30	500	65	10.7	234	6080
11	33	500	65	10.7	215	
12	36	500	65	10.6	222	
13	39	500	65	10.5	193	
14	42	500	65	10.5	159	1–15
15	45	500	65	10.5	137	7010
					7020	

From Tables I(a) and I(b) it is apparent that during the trial *a* where the tobacco previously was permitted to stand for 15 minutes in an annomia atmosphere, for the purpose of checking the effect of the reaction time, after 30 minutes steaming out, with the ten fractions only 4,565 mg nicotine was found, whereas for the same quantity of ammonia during trial *b* which was directly fed through the tobacco for 15 minutes and for a further 15 minutes steamed-out, there was obtained a yield of 6,080 mg. nicotine.

For the trail *b* there was selected as the starting basis the same steaming time, that is to say the same consumption of steam as for the trial *a*.

If the nicotine yield is compared for the same treatment times, that is, during the trial *a* 15 minutes effective time plus 30 minutes steaming, and for the trial *b* 15 minutes gas throughflow plus 30 minutes steaming, then there will be observed a nicotine content of 7,020 mg. in the condensate of trial *b* in contrast to 4,565 mg. nicotine in the condensate of trial *a*, that is to say 53.78 percent more yield.

Example 2

After it was apparent from Tables I(a) and I(b) that an additional reaction time did not bring with it a correspondingly increased yield of nicotine and a larger portion of the ammonia remained unused, the question arose as to what actually is the required or economical dosage of NH_3.

There was, for instance, already proposed using the alkalinization agent (NH_3) in an approximately 2 to 3-fold excess of the quantity theoretically required for converting or transforming the nicotine salts.

Independent of the fact that for different tobaccos the nicotine is present in still not yet explained different pronounced bonds with different acids, the gas quantity to be used and especially the dosage — something which is important for a gas which can be removed during treatment — cannot be determined on the basis of theoretical calculations. An evaluation of the test results carried out in Example 1 (cf., in particular, Table I(b) starting from fractions 5–10) showed that the pH-value of the condensate and the corresponding nicotine quantity are in a narrow correlation to one another. In other words, the optimum nicotine yield is present at a pH-value of 11.6 to 11.7, at lower pH-values on the other hand this yield is considerably less.

A homogeneous tobacco sample which had been subdivided into two equal portions, each of 200 grams, had passed therethrough under vacuum conditions vapor mixtures at 60°C for different pH-values. The condensate was analyzed at individual fractions. The results of these tests have been graphically plotted in FIGS. **2***a* and **2***b* and clearly indicate that the pH of the condensate plays a decisive role for the nicotine yield and that at 60°C a pH of 11.7 affords the optimum yield. A 2 to 3-fold, or even greater fold increased ammonia dosage cannot increase the pH and accordingly the yield, rather only leads to unnecessary gas usage.

From the previously mentioned publications it is clearly apparent that the nicotine yield for constant distillation conditions reduces with progressive time. Reduction of the yield is particularly significant at pH-values exceeding 10. The maximum nicotine concentration of the condensate at a pH of 11.7 and approximately 200 mm Hg mercury (Torr) pressure, and at a temperature of 63°C amounted to 0.1 percent.

Example 3

Continuous and "Impulse"-Treatment

Since for ammonia-absorption, something which is important for the nicotine yield, lower temperatures, which it is to be mentioned simultaneously render possible a relatively protective tobacco treatment, are more desirable than higher temperatures, it appears important to examine whether through temperature-and pressure and concentration-changes the yield can be increased during the treatment time. The comparative tests set forth in Tables II(a) and II(b) and corresponding to the results graphically illustrated in FIGS. *3a* and *3b* show that if the gas dosage is undertaken at 65°C while simultaneously passing through the steam, the nicotine yield reduces with time. By increasing the temperature to 80°C during the next phase, without infeed of gas or NH_3 the reduction of the yield is briefly eliminated, thereafter the reduction is much more rapid. This points to an intensive driving-out of the nicotine and ammonia at increased temperature. Treatment with this pulsating temperature and gas-dosing conditions provides a considerably better yield than during constant temperatures and gas dosage conditions. Furthermore, attention is invited to the bend or knee points indicated at * and Kp in the pH-curves and the denicotinization and nicotine yield curves of FIGS. *4a* and *4b*, the significance of which for the control of the high temperature phases has already been explained above.

TABLE II(a)

Treatment: Denicotinization at periodic temperature increases and discontinuous gas dosages. Treatment temperature 65°–80°C.
Quantity: 200 grams.
Size or Format: 2+2 cm

Fraction	Time Min.	Quantity ml	Temp. °C	Condensate pH	Nicotine (mg/250 ml)		Comments
1	2.45	250	65	11.7	816		Total Nicotine
2	2.45	250	65	11.7	678)	65°C NH_3	5170 mg.
3	2.45	250	65	11.7	521)		Fraction 1–12
4	2.45	250	80	11.0	495)		5070 mg.
5	2.45	250	80	10.5	312)	80°C	
6	2.45	250	65	11.7	524)		
7	2.45	250	65	11.7	391)	65°C NH_3	
8	2.45	250	80	10.9	378)		
9	2.45	250	80	10.5	178)	80°C	
10	2.45	250	65	11.7	253)		
11	2.45	250	65	11.7	237)	65°C NH_3	
12	2.45	250	80	10.9	261)	80°C	
13	2.45	250	80	10.5	125		
					5170 mg		

TABLE II(b)

Treatment: Denicotinization during periodic temperature increases and continuous gas dosage. Treatment temperature 65°–80°C.
Quantity: 200 grams.
Size or Format: 2+2 cm.

Fraction	Time Min.	Quantity ml.	Temp. °C	Condensate pH	Nicotine (mg/250 ml)		Comments
1	2.45	250	65	11.7	875	)	Fraction 12
2	2.45	250	65	11.7	731	) 65° NH_3	6110 mg.
3	2.45	250	65	11.7	537	)	
4	2.45	250	65	11.7	447	)	
5	2.45	250	80	11.4	941	) 80° NH_3	
6	2.45	250	80	11.4	732	)	
7	2.45	250	65	11.7	467	) 65° NH_3	
8	2.45	250	65	11.7	265	)	
9	2.45	250	80	11.4	398	) 80° NH_3	
10	2.45	250	80	11.4	373	)	
11	2.45	250	65	11.7	176	) 65° NH_3	
12	2.45	250	65	11.7	168	)	

Upon repeating these comparative tests with a respective 2 kilograms likewise homogeneous tobacco material, wherein the tobacco on the one hand was

treated at 60°C and at 80°C under constant temperature and gas dosage conditions and, on the other hand, the mentioned pulsating treatment was undertaken between 60°C and 80°C, there were obtained the results set forth in Table III.

TABLE III

Treatment: Denicotinization — Treatment temperature: 60°C constant
Tobacco Type: Beneventano (1125 I)
Quantity: 2 kg

Fraction	Time Min.	Quantity l	Temp. °C	Condensate	Nicotine 25 ml
1	20	3.4	60	11.7	26.8 mg
2	20	3.3	60	11.7	19.6
3		3.4	60	11.7	19.5
	60	10.1			8,200 mg

Treatment: Denicotinization — Pulsating treatment
Type: Beneventano (1128 I)
Quantity: 2 kg

Fraction	Time Min.	Quantity	Temp. °C	Condensate	Nicotine 25 ml
1	10	1.5	60	11.7	33.5 mg
2	3	—	80	—	—
3	10	2.2	60	11.7	31.7
4	4	—	80	—	—
5	10	2.3	60	11.7	25.2
6	3	—	80	—	—
7	10	2.1	60	11.7	21.4
8	3	—	80	—	—
9		2.2	60	11.7	17.7
	63	10.3			10,300 mg

The greatest yield was obtained at 80°C. As already previously mentioned, this relatively high treatment temperature however, when employed during longer time periods has a negative effect upon the structure and quality of the tobacco.

During the so-called pulsating treatment there is obtained 10,300 milligrams nicotine in contrast to 8,100 milligrams during the treatment variant employing a constant temperature of 60°C. The increased yield therefore amounted to 27.2 percent.

It is here to be mentioned that the temperature increases were only always briefly carried out (a total of 13 minutes for a treatment time of 60 minutes). The quality of the tobacco was judged to be the best with this pulsating treatment.

Example 4

In order to further examine the dependency of the denicotinization effect of the ammonia dosage to the treatment medium three further tests were carried out under the following conditions:

In each instance 300 grams homogeneous cigar inserts (Kentucky) were treated for 30 minutes with a quantity of water vapor producing a total of 1,000 ml condensate, treatment was carried out at a temperature of 60°C under reduced pressure conditions, and the quantity of ammonia being varied from test to test. The condensate of the treatment medium emanating from the tobacco was continuously examined with regard to its pH-value and titration value. Additionally, the employed quantity of ammonia was determined and the nicotine yield measured at the vapor condensate.

The nicotine content of the untreated tobacco amounted to 1.33 percent, corresponding to 3.99 grams (21.9 mM) nicotine in 300 grams tobacco.

The results obtained during these tests have been collected in the following Table IV.

Example 5

(Directed and controlled denicotinization of raw tobacco)

The nicotine content of 100 kg tobacco (Rio Grande) having a starting value of 1.54 percent nicotine should be reduced to 0.8 percent. This means that 740 grams nicotine must be removed from the tobacco.

For this purpose 100 kg tobacco was introduced into an apparatus and treated for approximately 5 minutes with saturated water vapor at a temperature of 65°C. A part of the water vapor emanating from the tobacco was condensated and the pH-value of this condensate measured. Additionally, 10 ml of this condensate was titrated with 0.1 n sulfuric acid. The values amounted to 9.8 pH and 1.85 ml H_2SO_4 0.1n. Now ammonia gas was added to the water vapor until the condensate of the treatment medium escaping from the tobacco had a pH-value of 11.3 and (for 10 ml condensate) a titration value of 45 ml 0.1 n H_2SO_4 had been reached. The steam or water vapor emanating from the tobacco and charged with nicotine is freed from the ammonia and nicotine in a heated sulfuric acid-absorber. After a treatment time of 1 hour of the above mentioned condition the volume of sulfuric acid in the absorber was determined and an aliquot sample removed. At this sample the nicotine content was determined by means of a Technicon-Autoanalyzer. With a quantity of absorption agent amounting to 80 liters and a nicotine content of 0.74 percent of nicotine removed at this point of time from the tobacco amounted to 592 grams. These measurements now were repeated approximately every 10 minutes. After a further 40 minutes treatment time there was determined at the absorption agent a total nicotine content of 720 grams. Thereafter, the dosage of ammonia was stopped, the temperature increased to 75°C and the tobacco further treated with pure steam or water vapor for the purpose of driving out the ammonia remaining in the tobacco. The condensated portion of the treatment medium escaping from the tobacco was delivered past a pH-electrode and thus the pH-value continuously measured and recorded by a suitable recording instrument. After 50 minutes treatment with pure steam, the vapor condensate possessed a pH-value of 9.65. For obtaining more precise results the titration value of the condensate was

TABLE IV

Test No.	Condensate pH-value	Ammonia addition in mg and g (mM)	Nicotine yield in mg and g (mM)	Ratio of Consumed NH_3: Nicotine present in tobacco	Denicotinization in %
1	11.1	820 mg (48.3)	975 mg (6.0)	1.94	24.1
2	11.7	14.25 g (839.8)	2.18 g (13.5)	33.73	54.2
3	12.0	36.75 g (2160)	2.54 g (15.7)	86.75	64.0

also determined in the above described manner, resulting in a value of 3.6 ml H_2SO_4 0.1 n. The determination of the titration value was carried out from this point of time every 5 minutes. After a further 20 minutes treatment time the vapor condensate possessed a pH-value of 9.6 and a titration value of 1.75 ml 0.1 n H_2SO_4. The total nicotine content at the absorption agent amounted to 752 grams. The nicotine content of the treated tobacco was thus reduced to 0.78 percent.

Example 6

In order to be able to demonstrate the differences between the inventive controlled mode of operation and the previously employed method of denicotinization and/or mellowing of tobacco, where the treatment conditions, especially the duration of the mellowing treatment was only carried out on the basis of "empirical values", a further series of comparative tests was carried out where six different tobacco types were each denicotinized and mellowed or only mellowed, both by carrying out the method of the invention (determining the duration of the treatment with steam according to the condensate pH-value and the titration value) as well as also by the conventional techniques (duration of the treatment with steam on the basis of empirical values, amounting in each case to 30 minutes, with the exception of test *5b* where, because of the very poor smoke result test during the control, mellowing was carried out for 150 minutes).

All of the tobacco types were subjected both before and after treatment to an organoleptic qualitative check, where, by means of smoke evaluation grades, the best grade or note being the mark 1 and the poorest grade being the grade or mark 5, the smoke performance was evaluated, these grades being based upon the following individual judgments or evaluations:

Inhalation:	very pronounced	= 5
	pronounced	= 4
	average	= 3
	slight	= 2
	very slight	= 1
Sharpness:	very sharp	= 5
	sharp	= 4
	average	= 3
	trace of sharpness	= 2
	no sharpness	= 1
Character plus Aroma:	flat	= 5
	little aroma	= 4
	average aroma	= 3
	good aroma	= 2
	very good aroma	= 1
Taste plus general impression:	poor	= 5
	not open	= 4
	coated	= 3
	not quite clean	= 2
	open and clean	= 1

The results of these trials have been collected together in Table VI, from which can be seen that the smoke judgment or grading for all of the tobaccos

TABLE VI

Starting Phase				Mellowing Phase			Tobacco Analysis				Smoke Grading	
Vapor Treatment (Min)	pH of Vapor Condensate (Reference pH-Value)	Titration of 10 ml Condensate with H_2SO_4 0.1 N in ml (Reference Titration Value)	Duration of Treatment with Vapor-Ammonia Mixture (Min)	Duration of Treatment with Pure Water Vapor (Min)	pH of Vapor Condensate	Titration of 10 ml Condensate with H_2SO_4 0.1 N (ml)	NH_3		Nicotine			
							a	*b*	*a*	*b*	*a*	*b*
					Rio Grande 6788		Denicotinization Treatment					
[c]5	9.8	4.5	120	75	9.6	3.0	1.27	0.93	2.33	1.18	4.5	2.0
[d]	—	—	120	30	—	—	1.27	1.23	2.33	1.28	4.5	3.5
					Geudertheimer OGT 64		Denicotinization Treatment					
[c]5	9.9	4.8	120	90	9.6	2.8	1.30	0.89	2.45	1.22	4.5	1.5
[d]	—	—	120	30	—	—	1.30	1.61	2.43	1.42	4.5	5.0 K
					Rio Grande 8092		Denicotinization Treatment					
[c]5	9.6	3.8	140	60	9.4	3.1	1.12	0.90	3.24	2.08	5.0	2.0
[d]	—	—	140	30	—	—	1.15	1.82	3.21	2.32	5.0	5.0 K
					Paraguay 18 B		Mellowing Treatment					
[c]5	9.8	4.8	—	60	9.5	2.4	1.42	0.98	1.34	1.11	4.0	1.5
[d]	—	—	—	30	—	—	1.43	1.21	1.30	1.20	4.0	3.0
					Geudertheimer HGT 65		Mellowing Treatment					
[c]5	10.1	5.9	—	80	9.5	3.1	1.87	0.85	0.97	0.83	5.0	1.5
[d]	—	—	—	150	—	—	1.85	0.43	0.97	0.63	5.0	4.5 L
					Burley HGT 65		Mellowing Treatment					
[c]5	10.0	5.4	—	70	9.6	2.9	1.51	0.92	1.13	1.02	4.5	1.5
[d]	—	—	—	30	—	—	1.52	1.34	1.10	1.08	4.5	4.0 K

[a]untreated
[b]treated
[c]Inventive Method
[d]Conventional Method
NOTE: 1. Constant Conditions for all Trials
Vapor: 1.5 kg/min.
Temp.: 60°C
Tobacco: 100 kg
2. best grade=1
poorest grade=5
K=irritating smoke
L=flat or empty aroma

treated according to the invention was considerably better than for the corresponding untreated tobacco and is at grade 2.0 or even better, whereas none of the tobaccos which have treated according to the conventional techniques received a better smoke grading than the grade 3.0 and in the case of a number of the treated tobaccos (tests 2*b* and 3*b*) were even associated with a poorer smoke grade or judgment than the untreated tobacco.

Example 7

For the purpose of denicotinization 100 kg tobacco was introduced into an installation suitable for the flow of the treatment medium through the tobacco and for approximately 5 minutes saturated steam at a temperature of 60°C was permitted to flow through the tobacco. A portion of the steam emanating from the tobacco was condensated and the pH-value thereof was measured. Similarly, 10 ml condensate was titrated with 0.1 n H_2SO_4 in order to obtain characteristic values concerning the volatile bases of the untreated tobacco. Now ammonia gas was introduced into the steam until the vapor condensate escaping out of the tobacco possessed a pH-value of approximately 11 and for 10 ml condensate there was obtained a titration value of approximately 45 ml 0.1 n H_2SO_4. After 45 minutes the temperature of the vapor was raised to 70°C and after a further 45 minutes to 80°C, then held for an additional 45 minutes at this temperature. Throughout the entire previous treatment times the alkalinity of the treatment medium was held at the desired values through appropriate infeed dosage of ammonia.

After these three treatment stages the ammonia dosage was interrupted and the temperature increased to approximately 85°C and, for the purpose of driving out the ammonia which remained in the tobacco, pure steam was permitted to flow through the tobacco. At time intervals of approximately 5 minutes there was determined the pH-value and the alkalinity of the condensate of the treatment medium emanating from the tobacco. After a further 60 minutes treatment time the vapor condensate exhibited a pH-value which was less by 0.2 units of pH and a titration value which was less by 2.5 ml 0.1 n H_2SO_4 per 10 ml condensate, than the starting values prior to the addition of ammonia. The flow direction of the treatment medium was reversed every 20 minutes.

Example 8

For the purpose of mellowing 100 kg tobacco such was placed into an installation suitable for the flow of the treatment medium through the tobacco. For approximately 5 minutes saturated steam at a temperature of 60°C was passed through the tobacco. Now analogous to the procedures of Example 1 the characteristic values of the volatile bases of the untreated tobacco were determined. Then the temperature of the steam was increased within 5 minutes to 100°C, held at this value for approximately 2 minutes, and then within 5 minutes again brought back to 60°C, and held at this temperature for 8 minutes. These temperature changes were repeated 5 times. The temperature-intervals were automatically programed. A portion of the steam or water vapor emanating from the tobacco was condensated and the condensate transferred by means of a suitable device to the electrode of a pH-measuring device of standard design and the pH-value continuously measured. In similar fashion 10 ml of this condensate was titrated with 0.1 n H_2SO_4, something which also can be automated, in order to obtain a continuous measurement. After a treatment time of 1 hour and 45 minutes the vapor condensate emanating from the tobacco exhibited a pH-value which was lower by the amount of 0.3 units of pH and a titration value which was lower by the amount of 3 ml 0.1 n H_2SO_4 per 10 ml condensate, than the starting values of the untreated tobacco. The flow direction of the treatment medium was reversed every 20 minutes.

Example 9

For denicotinization of the tobaccc one also proceeds in the fashion that after measuring the starting values of the untreated tobacco the temperature course of the treatment medium was programed in accordance with the teachings of Example 2. The ammonia was only dosed-in at the lower temperatures and the addition always interruped upon reaching the higher temperature. The determination of the alkalinity value of the vapor condensate emanating from the tobacco was performed analogous to the teachings of Example 2. The pH-value of approximately 11 was maintained for 2 hours, then the infeed of ammonia was shut-off. The ammonia remaining in the tobacco was driven-off with pure steam or water vapor until the vapor condensate emanating from the tobacco had a pH-value which was lower by the amount 0.3 and a titration value lower by the amount 3.5 ml 0.1 n H_2SO_4 per 10 ml condensate than at the beginning of treatment. The drive-off time amounted to 45 minutes. Also, for this treatment the flow direction of the treatment medium was reversed every 20 minutes.

While there is shown and described present preferred embodiments of the invention, it is to be distinctly understood that the invention is not limited thereto but may be otherwise variously embodied and practiced within the scope of the following claims.

Accordingly, what is claimed is:

1. A method for denicotinization and mellowing of tobacco, comprising the steps of passing through the tobacco a gaseous treatment medium containing at least ammonia and steam for denicotinization purposes, then passing through the tobacco an ammonia-free, steam-containing gaseous treatment medium for tobacco mellowing purposes, controlling the ammonia concentration during the denicotinization step such that the pH-value of the condensate of the treatment medium departing from the tobacco is in a range between approximately 11.0 and 12.0, and for tobacco mellowing passing the ammonia-free, steam-containing treatment medium through the tobacco for such length of time until the pH-value of the condensate of the treatment medium departing from the tobacco is in a range of approximately 1.0 units of pH below to approximately 0.5 units of pH above a reference-pH-value of the condensate of the treatment medium obtained under the same conditions prior to tobacco denicotinization.

2. The method as defined in claim **1**, wherein during denicotinization there is controlled the delivery of am-

monia such that the pH-value of the condensate of the treatment medium departing from the tobacco is in a range between approximately 11.0 and 12.0

3. The method as defined in claim 1, wherein the throughflow of the ammonia-free, steam-containing treatment medium through the tobacco during mellowing is carried out for such length of time until the pH-value of the condensate of the treatment medium departing from the tobacco at most is about 0.2 units of pH above the reference pH-value.

4. The method as defined in claim 3, wherein the throughflow of the treatment medium is carried out for such length of time until the pH-value of the condensate of the treatment medium departing from the tobacco is at least approximately 0.3 units of pH below the reference pH-value.

5. The method as defined in claim 1, including the step of at least increasing for certain time periods the temperature of the treatment medium.

6. The method as defined in claim 5, including the step of controlling the temperature of the treatment medium at the beginning of its flow through the tobacco so as to be less than 75°C, and alternately increasing and again decreasing the temperature of the treatment medium during its flow through the tobacco such that each temperature increase is greater than the following temperature decrease.

7. The method as defined in claim 6, including the step of maintaining the temperature of the treatment medium for 5 to 20 minutes at a higher value and for 10 to 20 minutes at a lower value, wherein the amount of each temperature increase is the same as each temperature decrease.

8. The method as defined in claim 6, including the step of introducing the ammonia during periods of lower temperature.

9. A method for denicotinization and mellowing of tobacco comprising the steps of passing through the tobacco a gaseous treatment medium containing at least ammonia and steam for denicotinization purposes, forming a condensate and determining the titration value of said condensate, then controlling the ammonia concentration in the ammonia steam treatment medium during the denicotinization step such that said condensate when titrated as 10 ml sample indicates an acid consumption in a range amounting to approximately 45 to 80 ml 0.1 acid, and then passing through the tobacco, an ammonia-free, steam-containing gaseous treatment medium for tobacco mellowing purposes, said ammonia-free, steam-containing medium being passed through the tobacco for such length of time until a sample of the condensate from this latter treatment medium departing from the tobacco, upon titration thereof, indicates an acid consumption which amounts to 70 to 110 percent of an initial reference titration value which is obtained prior to denicotinization and which is of the same size sample of the initial condensate.

10. The method as defined in claim 9, wherein the infeed of ammonia during denicotinization is controlled such that the acid consumption of a 10 ml sample of the treatment medium departing from the tobacco lies between approximately 45 and 80 ml 0.1 n acid.

11. The method as defined in claim 9, including the step of at least increasing for certain time periods the temperature of the treatment medium.

12. The method as defined in claim 11, including the step of controlling the temperature of the treatment medium at the beginning of its flow through the tobacco so as to be less than 75°C, and alternately increasing and again decreasing the temperature of the treatment medium during its flow through the tobacco such that each temperature increase is greater than the following temperature decrease.

13. The method as defined in claim 11, including the step of maintaining the temperature of the treatment medium for 5 to 20 minutes at a higher value and for 10 to 20 minutes at a lower value, wherein the amount of each temperature increase is the same as each temperature decrease.

14. The method as defined in claim 12, including the step of introducing the ammonia during periods of lower temperature.

15. The method as defined in claim 9, including the step of controlling the temperature of the treatment medium at the beginning of its flow through the tobacco so as to be less than 75°C, and alternately increasing and again decreasing the temperature of the treatment medium during its flow through the tobacco such that each temperature increase is greater than the following temperature decrease.

16. The method as defined in claim 9, including the step of maintaining the temperature of the treatment medium for 5 to 20 minutes at a higher value and for 10 to 20 minutes at a lower value, wherein the amount of each temperature increase is the same as each temperature decrease.

17. The method as defined in claim 15, including the step of introducing the ammonia during periods of lower temperature.

18. A method for mellowing tobacco, comprising the steps of passing an ammonia-free, steam-containing gaseous treatment medium through the tobacco, passing the treatment medium through the tobacco for such length of time until the pH-value of the condensate of the treatment medium departing from the tobacco is in a range of approximately 0.1 units of pH to approximately 1.0 units of pH below a reference-pH-value of condensate of the treatment medium obtained under the same conditions prior to mellowing.

19. The method as defined in claim 18, wherein the treatment medium is passed through the tobacco for such length of time until the pH-value of the condensate of the treatment medium departing from the tobacco is approximately 0.2 units of pH to approximately 0.5 units of pH below the reference pH-value.

20. The method as defined in claim 19, including the step of passing the treatment medium through the tobacco for such length of time until the pH-value of the condensate of the treatment medium departing from the tobacco is approximately 0.3 units of pH to approximately 0.4 units of pH below the reference pH-value.

21. The method as defined in claim 18, including the step of at least increasing for certain time periods the temperature of the treatment medium.

22. The method as defined in claim 21, including the step of controlling the temperature of the treatment medium at the beginning of its flow through the tobacco so as to be less than 75°C, and alternately increasing and again decreasing the temperature of the treatment medium during its flow through the tobacco

in a manner such that each temperature increase is greater than the following temperature decrease.

23. The method as defined in claim **21,** including the step of maintaining the temperature of the treatment medium for 5 to 20 minutes at a higher value and for 10 to 20 minutes at a lower value, wherein the amount of each temperature increase is the same as each temperature decrease.

24. A method for mellowing tobacco, comprising the steps of passing an ammonia-free, steam-containing treatment medium through the tobacco, controlling the flow of the treatment medium through the tobacco for such length of time until a sample of the condensate of the treatment medium departing from the tobacco during titration thereof exhibits an acid consumption which is at least approximately lower by the value of 5 than a reference titration value determined by titrating a condensate sample of the treatment medium departing from the tobacco under the same conditions prior to tobacco mellowing.

25. The method as defined in claim **24,** including the step of at least increasing for certain time periods the temperature of the treatment medium.

26. The method as defined in claim **25,** including the step of controlling the temperature of the treatment medium at the beginning of its flow through the tobacco so as to be less than 75°C, and alternately increasing and again decreasing the temperature of the treatment medium during its flow through the tobacco such that each temperature increase is greater than the following temperature decrease.

27. The method as defined in claim **24,** including the step of maintaining the temperature of the treatment medium for 5 to 20 minutes at a higher value and for 10 to 20 minutes at a lower value, wherein the amount of each temperature increase is the same as each temperature decrease.

28. A method for mellowing tobacco and for reducing the nicotine content thereof comprising the steps:

A. contacting the tobacco with steam,
B. condensing a small portion of the steam,
C. analyzing the condensate for the purpose of determining a reference-pH-value,
D. contacting the tobacco with a gaseous mixture comprising steam and ammonia for reducing the nicotine content of the tobacco, said gaseous mixture being regulated to provide a sufficient concentration of ammonia such that the condensate after contact with the tobacco has a pH-value between 11 and 12,
E. continuing denicotinization for a time sufficient to reduce the nicotine content to a desired level,
F. Contacting the tobacco with ammonia-free steam for a time sufficient to reduce the pH-value of the condensate emanating from the tobacco to from about 1.0 units below and from about 0.5 units above the reference-pH-value established in steps A–C inclusive.

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