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PROCESS FOR REMOVING NICOTINE FROM TOBACCO

Filed July 18, 1961

2 Sheets-Sheet 1

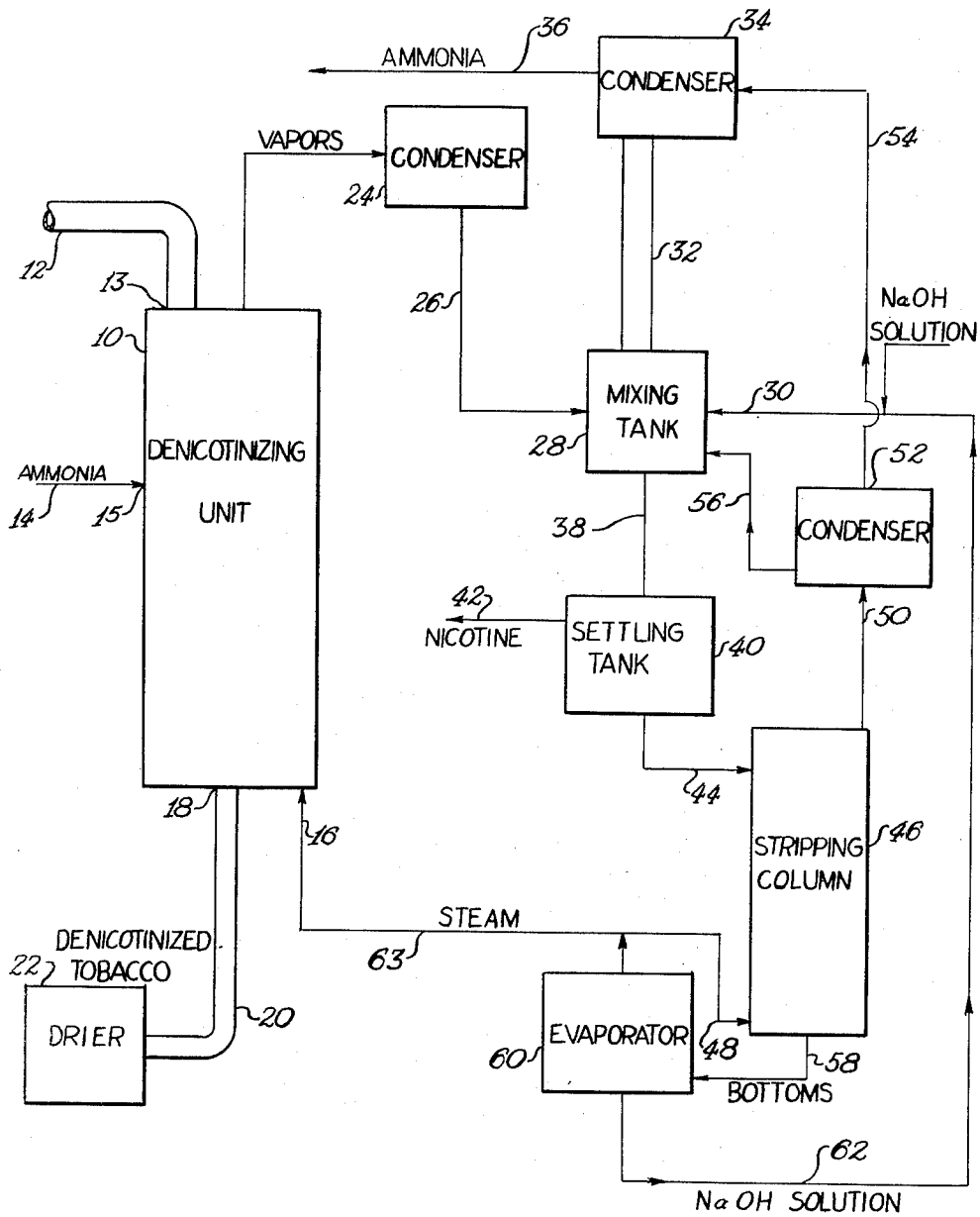


Fig I

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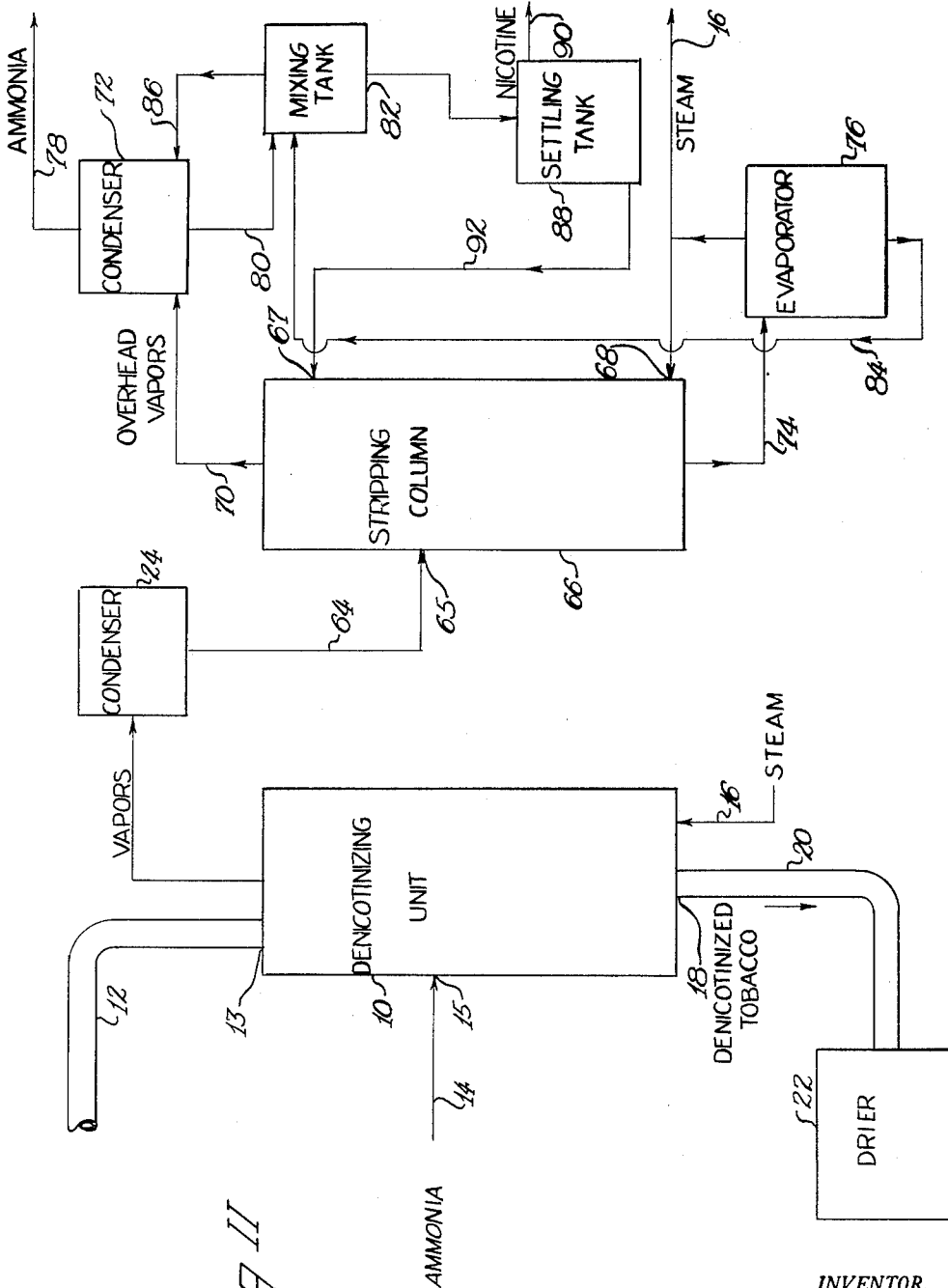


Fig II

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PROCESS FOR REMOVING NICOTINE FROM TOBACCO

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9 Claims. (Cl. 260-291)

This invention relates to a process for removing nicotine from tobacco and more particularly to a process for removing nicotine from tobacco wherein the nicotine and the reagents employed for removing the nicotine are recovered.

It is known in the art to remove nicotine from tobacco by treating the tobacco with ammonia and steam. This process involves treating tobacco with ammonia in order to disengage the nicotine from the salts in which it is present in the tobacco by replacing it with ammonia. The free nicotine and ammonia are then removed from the tobacco with the aid of steam. This method is generally satisfactory with respect to removal of the nicotine from the tobacco but for economical operation on a commercial scale it is necessary to provide for the efficient recovery of the ammonia employed as well as the nicotine removed from the tobacco which in itself has commercial value.

It is, therefore, one of the objects of this invention to develop a more satisfactory method of denicotinizing tobacco.

The principal object of this invention is to provide a new process for removal of nicotine from tobacco wherein increased overall efficiency and economy are realized from improved operating conditions and wherein the ammonia employed to remove the nicotine from the tobacco and the nicotine so removed are recovered in substantially quantitative amounts.

The process of the present invention is described in detail in conjunction with the accompanying drawing in which FIG. I is a diagrammatic flow sheet illustrating a presently preferred embodiment of the process and in which FIG. II is a diagrammatic flow sheet of a second alternative embodiment of the process.

Referring to FIGURE I: Tobacco which is to be denicotinized is conveyed to the denicotinizing unit 10 by means of conveyor 12. The denicotinizing unit 10 which is operated at a temperature of about 100° C. can be any suitable apparatus such as a rotary drum unit capable of providing intimate contact of the tobacco with vapors of ammonia and steam. Gaseous ammonia is introduced through line 14 into the denicotinizing unit 10 at a point 15 spaced some distance from the tobacco feed inlet 13. Steam is introduced into the denicotinizing unit 10 at 16 adjacent the tobacco discharge outlet 18. Thus, the tobacco flowing within the denicotinizing unit 10 in countercurrent relationship to the introduced vapors initially contacts ammonia which reacts with the nicotine found in the tobacco generally in the form of salts and replaces the nicotine therein. The nicotine is released to the vapor. The tobacco continuing its travel through the unit 10 then contacts steam which serves to drive off the ammonia from the tobacco. The moist denicotinized tobacco is then discharged from the denicotinizing unit at 18 and transferred via line 20 to drier 22 wherein it is contacted with conditioned air to cool the tobacco to approximately 100° F. and reduce the moisture content to a desired level of from approximately 11 to 13% by weight.

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The vapors from the denicotinizing unit 10 comprising water, nicotine and ammonia pass overhead to condenser 24 where the vapors are cooled sufficiently to cause them to condense. A temperature of about 30° C. in condenser 24 is satisfactory. The liquid condensate from condenser 24 consisting of relatively dilute aqueous solution of nicotine and ammonia is then introduced via line 26 into mixing tank 28. Concentrated aqueous sodium hydroxide solution is introduced into the mixing tank 28 through line 30 in sufficient amount to form a mixture in mixing tank 28 containing about 10 to 15 percent sodium hydroxide by weight, and thoroughly mixed with the aqueous nicotine and ammonia solution. The mixture in tank 28 is thoroughly agitated by any suitable means and heated to a temperature of at least about 95° C. or higher to expel the ammonia, the vapors of which pass through conduit 32 to nicotine-water condenser 34. The condenser 34 is maintained at a temperature of from about 60 to 80° C. which temperature is below the boiling points of the water and nicotine thereby causing the water and nicotine to condense while permitting the vapors of ammonia to escape through line 36. The ammonia passing through line 36 can be stored or recycled to a denicotinizing unit 10 to be there used for treating additional quantities of tobacco. Any water and nicotine which may have condensed in condenser 34 are returned via conduit 32 to mixing tank 28.

After thorough mixing in mixing tank 28 the liquid mixture is transferred via line 38 to settling tank 40 where the liquid mixture is permitted to stand quiescent for a period of time resulting in the formation of two readily separable phases: a light nicotine phase and a heavier aqueous sodium hydroxide phase. A temperature of from about 70 to 95° C., and preferably from 85 to 95° C. is maintained in settling tank 40 to improve separation of the two phases. Separation and recovery of the nicotine can be accomplished simply by settling, or the settling tank 40 can be replaced by a centrifuge or other mechanical separator with the separated nicotine phase being withdrawn through line 42 and utilized as desired. The heavier aqueous sodium hydroxide phase is conducted through line 44 to steam stripping column 46. Steam is introduced at 48 into the lower section of stripping column 46 to contact the down-flowing aqueous sodium hydroxide solution to strip therefrom any residual nicotine contained therein. The overhead vapors from stripping column 46 are taken via line 50 to condenser 52 which is operated at a sufficiently low temperature, such as from about 60-80° C., to cause water and nicotine to condense while permitting any uncondensed ammonia to escape via line 54 to condenser 34 from which it is recovered as previously described. The condensate from condenser 52 is recycled via line 56 to the mixing tank 28 for further treatment with sodium hydroxide. The bottoms liquid from stripping column 46 passes via line 58 to evaporator 60 which operates at a temperature from about 120 to 150° C. for concentration of the sodium hydroxide. The steam from the evaporator 60 can be utilized in stripping column 46 while the concentrated sodium hydroxide solution is recycled through line 62 for re-use in the process. Excess steam not required for the stripping operation in column 46 can be recycled via line 63 to the tobacco denicotinizing operation.

A good idea of the effectiveness of the subject process may be obtained from the material balance set forth in Table I. The calculated values presented therein are based on the processing of 1000 pounds of tobacco having

a nicotine content of 3.5% with removal of 50% of the nicotine.

Table I

Stream	Water, lb.	NH ₃ , lb.	Nicotine, lb.	NaOH, lb.
1. Condensate from tobacco denicotinizing operation	3,029	30	18.4	0
2. Ammonia recycle (Line 36)	20	30	0.6	0
3. Recovered nicotine (Line 42)	2	0	17.5	0
4. Steam recycle (Line 63)	2,998	0	0.3	0
5. Stripping column feed (Line 44)	5,188	Trace	13.0	778
6. Stripping column overhead vapor (Line 50)	684	Trace	12.7	0
7. Stripping column bottoms liquid (Line 58)	5,189	0	0.5	778
8. Stripping steam (Line 48)	635	0	0.2	0
9. Recycled caustic soda solution (Line 62)	1,556	0	0	778

As can be seen from Table I, substantially quantitative recovery of the ammonia and nicotine present in the condensate from the denicotinizing operation is achieved. Moreover, the sodium hydroxide employed is recovered in substantially quantitative amounts for re-use in the process.

The embodiment of the process illustrated in FIGURE II is generally similar to the process illustrated in FIGURE I except that the liquid condensate from the tobacco denicotinizing operation is steam stripped prior to treatment with the strong sodium hydroxide solution to effect the phase separation of the nicotine present therein. The various units are operated at substantially the same temperatures as disclosed previously.

Thus, referring to FIGURE II, the tobacco is denicotinized in denicotinizing unit 10 as previously described and the overhead vapors comprising water, nicotine and ammonia are condensed in condenser 24. The liquid condensate passes from condenser 24 via line 64 and is introduced at 65 into stripping column 66. An aqueous solution of sodium hydroxide is introduced at 67 into the upper section of stripping column 66 and serves to displace the ammonia and nicotine from solution. This sodium hydroxide solution should be sufficient to form a mixture in stripping column 66 containing about 10 to 15 percent sodium hydroxide by weight, and conveniently comprises the aqueous sodium hydroxide solution resulting from the phase separation of the nicotine. Steam is introduced at 68 into the lower section of stripping column 66 and strips the ammonia and nicotine from the aqueous sodium hydroxide solution with the overhead vapors comprising these components being conducted via line 70 to nicotine condenser 72. The bottoms liquid from stripping column 66 comprising a relatively dilute aqueous solution of sodium hydroxide is fed via line 74 to evaporator 76. In evaporator 76 concentration of the sodium hydroxide is effected with the concentrated sodium hydroxide being conducted via line 84 to mixing tank 82. The steam produced in evaporator 76 can be employed as a stripping medium in stripping column 66, and excess steam may be recycled through line 16 to denicotinizing unit 10.

In nicotine condenser 72 the nicotine and water vapors condense while the ammonia vapors are taken off through line 78 and recovered or recycled to the denicotinizing operation. The liquid condensate from nicotine condenser 72 comprising an aqueous nicotine solution is conducted via line 80 to mixing tank 82. A sodium hydroxide solution having a concentration of about 30 to 50 percent sodium hydroxide is introduced into the mixing tank 82 through line 84 and thoroughly mixed with the nicotine-water solution. Any dissolved ammonia present is released from the solution by the sodium hydroxide and vented through line 86 to nicotine condenser 72. After thorough mixing in tank 82 the liquid mixture is transferred to settling tank 88 where the liquid mixture is permitted to stand quiescent for a period ranging from several minutes up to several hours or more. Upon standing, in the absence of agitation two readily separable

phases form: a light nicotine phase and a heavier aqueous sodium hydroxide phase. The separation and recovery of the nicotine can be accomplished by settling or the settling tank 88 can be replaced by a centrifuge or other mechanical device with the separated nicotine product being withdrawn through line 90 while the heavier aqueous sodium hydroxide phase is recycled via line 92 to the upper section of stripping column 66 to assist in displacing the ammonia and nicotine from the original condensate solution.

From the foregoing description it is readily seen that the desired objectives such as providing a process for removing nicotine from tobacco, minimizing losses of ammonia, recovering the nicotine removed from the tobacco, and accomplishing the foregoing in a simplified, economically efficient manner have all been accomplished.

Those modifications and equivalents which fall within the spirit of the invention and the scope of the appended claims are to be considered part of the invention.

I claim:

1. In a process for removing nicotine from tobacco material which comprises treating the tobacco with ammonia and steam to drive off in vapor form free nicotine and ammonia, the improvement which consists in condensing the nicotine vapors so-formed, treating the condensed nicotine vapors with an aqueous solution of sodium hydroxide whereby separation of a nicotine phase and an aqueous sodium hydroxide phase occurs, and then removing the so-separated nicotine phase.

2. In a process for removing nicotine from tobacco material which comprises treating the tobacco with ammonia and steam to drive off in vapor form free nicotine and ammonia, the improvement which consists in condensing the nicotine vapors so-formed, treating the condensed nicotine vapors with an aqueous solution of sodium hydroxide whereby separation of a nicotine phase and an aqueous sodium hydroxide phase occurs, removing the so-separated nicotine phase, steam stripping the said aqueous sodium hydroxide phase to strip residual nicotine therefrom, condensing the overhead vapors from said steam stripping operation and recycling the last-mentioned condensate for further treatment with sodium hydroxide.

3. In a process for removing nicotine from tobacco material which comprises treating the tobacco with ammonia and steam to drive off in vapor form free nicotine and ammonia, the improvement which consists in condensing the vapors so-formed to obtain an aqueous condensate of ammonia and nicotine, heating said condensate of ammonia and nicotine to a temperature sufficient to expel ammonia as a vapor, recovering said ammonia vapor, treating at an elevated temperature the condensate from which ammonia has been removed with an aqueous solution of sodium hydroxide whereby separation of a nicotine phase and an aqueous sodium hydroxide phase occurs, removing the so-separated nicotine phase, steam stripping the said aqueous sodium hydroxide phase to strip residual nicotine therefrom, condensing the overhead vapors from said steam stripping operation and recycling the last-mentioned condensate for further treatment with sodium hydroxide.

4. The process of claim 3 wherein the concentration of the aqueous sodium hydroxide solution employed is from about 30 to 50 percent by weight.

5. In a process for removing nicotine from tobacco material which comprises treating the tobacco with ammonia and steam to drive off in vapor form free nicotine and ammonia, the improvement which consists in condensing the vapors so-formed to obtain an aqueous condensate of ammonia and nicotine, heating said condensate to a temperature of at least about 95° C. to expel ammonia as a vapor recovering said ammonia vapor, treating said condensate at a temperature from about 70 to 95° C. with an aqueous solution of sodium hydroxide whereby separation of a nicotine phase and an aqueous sodium hydroxide phase occurs, removing the so-sepa-

rated nicotine phase, steam stripping the said aqueous sodium hydroxide phase to strip residual nicotine and ammonia therefrom, condensing the overhead vapors from said steam stripping operation and recycling the last-mentioned condensate for further treatment with sodium hydroxide.

6. In a process for removing nicotine from tobacco material which comprises treating the tobacco with ammonia and steam to drive off in vapor form free nicotine and ammonia, the improvement which consists in condensing the vapors so-formed to obtain an aqueous condensate of ammonia and nicotine, heating said condensate of ammonia and nicotine to a temperature sufficient to expel ammonia as a vapor, recovering said ammonia vapor, treating at an elevated temperature the condensate from which ammonia has been removed with an aqueous solution of sodium hydroxide whereby separation of a nicotine phase and an aqueous sodium hydroxide phase occurs, removing the so-separated nicotine phase, steam stripping the said aqueous sodium hydroxide phase to strip residual nicotine therefrom, condensing the overhead vapors from said steam stripping operation and recycling the last-mentioned condensate for further treatment with sodium hydroxide, evaporating the bottoms liquid resulting from said steam stripping operation to concentrate sodium hydroxide present therein.

7. In a process for removing nicotine from tobacco material which comprises treating the tobacco with ammonia and steam to drive off in vapor form free nicotine and ammonia, the improvement which consists in condensing the vapors so-formed to obtain an aqueous condensate of nicotine and ammonia, stripping the said aqueous condensate with steam in the presence of sodium hydroxide to remove overhead ammonia and nicotine, condensing the nicotine in said overhead, treating the condensed nicotine vapors with an aqueous solution of sodium hydroxide whereby separation of a nicotine phase and an aqueous sodium hydroxide phase occurs, removing the so-separated nicotine phase.

8. In a process for removing nicotine from tobacco material which comprises treating the tobacco with ammonia and steam to drive off in vapor form free nicotine and ammonia, the improvement which consists in condensing the vapors so-formed to obtain an aqueous condensate of nicotine and ammonia, stripping the said aqueous condensate with steam in the presence of sodium hydroxide to remove overhead ammonia and nicotine, condensing the nicotine in said overhead, treating the condensed nicotine vapors with an aqueous solution of sodium hydroxide to effect separation of a nicotine phase and an aqueous sodium hydroxide phase containing from about 10-15 percent sodium hydroxide by weight, removing the so-separated nicotine phase.

9. In a process for removing nicotine from tobacco material which comprises treating the tobacco with ammonia and steam to drive off in vapor form free nicotine and ammonia the improvement which consists in condensing the vapors so-formed to obtain an aqueous condensate of nicotine and ammonia, stripping the said aqueous condensate with steam in the presence of sodium hydroxide to remove overhead ammonia and nicotine, condensing the nicotine in said overhead, treating the condensed nicotine vapors with an aqueous solution of sodium hydroxide to effect separation of a nicotine phase and an aqueous sodium hydroxide phase containing from about 10 to 15 percent sodium hydroxide by weight, removing the so-separated nicotine phase, evaporating the bottoms liquid resulting from said steam stripping operation to concentrate sodium hydroxide present therein.

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