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EXTRACTING NICOTINE FROM TOBACCO

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The invention herein described may be manufactured and used by or for the Government for governmental purposes without the payment to me of any royalty thereon.

5 This invention relates to the art of extracting nicotine from vegetable material, particularly from tobacco.

One of the objects of this invention is to provide a method for the recovery of practically all the nicotine contained in uncured and cured tobacco. Another object of this invention is to provide an economical method for the extraction of nicotine from tobacco. Still another object of this invention is to provide a process for the removal of nicotine from tobacco which results in the production of a residue which may be used without further chemical treatment as a fertilizer or for incorporation in fertilizer mixtures to obtain the maximum advantage of the valuable constituents contained therein. Other objects of this invention include the provision for an economical method for extracting nicotine from vegetable material containing the same.

25 Unmarketable grades of leaf tobacco, low grade leaf tobacco or even a surplus of high grade marketable leaf tobacco may be processed more extensively to recover the nicotine contained therein provided the cost of extraction is reduced and the consumption of the nicotine increased correspondingly thereby. The cost of the production of the nicotine is dependent, not only on the cost of the raw materials and the cost of the unit operations involved, but on the value of the by-products obtained. Numerous proposals have been made for the extraction of nicotine from tobacco involving the use of heat treatment, water or steam treatment, alkali treatment, and treatment with hydrocarbons, either separately or in a variety of combinations of process steps. The net result has been that either the material is rendered difficult to handle during the process, excessive quantities of unrecoverable reagents have been required, the residue is of such nature that it required further processing which was uneconomical or could not be incorporated with other materials in more than small proportions due to its detrimental effect, or the recovery of the nicotine is low.

50 I have discovered a process for extracting nicotine from vegetable material, particularly from tobacco, by adjusting the water content of the tobacco, by treating the tobacco so conditioned with ammonia for a short period of time, by extracting the ammonia treated material with a liquid, nonaqueous solvent in the liquid phase, and by separating the nicotine from the solvent extract. The traces of solvent are separated from the extracted vegetable material and, together with the solvent separated from the nic-

otine solution, is used over again in the process and the residue, containing valuable nitrogen and potassium compounds as well as organic matter, is of such quality that it can be used in large proportions as a fertilizer or a fertilizer mixture.

Several examples of the operation of my process, with the extent of the extraction obtained, are given below:

1. 100 parts by weight of high grade dark fired leaf tobacco, containing 4.03% by weight of nicotine and 12.7% by weight of water, and cut to pass an 8 mesh screen, were conditioned by adding 20.3 parts by weight of saturated steam. After standing for 30 minutes, 5.8 parts by weight of ammonium hydroxide, containing 22% by weight of NH_3 , were added to the conditioned tobacco and the mixture agitated for 15 minutes with a temperature of 30° C. maintained. The ammonia treated material was thereupon immediately extracted with 6 successive portions of naphtha of 109 parts by weight each at a temperature of 85° C. to remove the nicotine. The nicotine was separated from the extract, resulting in 94.7% by weight of the total nicotine being extracted.

2. 100 parts by weight of the same high grade dark fired leaf tobacco, used in 1 above, were conditioned by adding 10 parts by weight of water followed by thorough agitation. After one minute 4.6 parts by weight of ammonium hydroxide, containing 22% by weight of NH_3 , were added to the conditioned tobacco and the mixture agitated for 14 minutes with a temperature of 30° C. maintained. The ammonia treated material was thereupon immediately extracted with 6 successive portions of fighting grade aviation gasoline of 145 parts by weight each at a temperature of 75° C. to remove the nicotine. The nicotine was separated from the extract, resulting in 96.1% by weight of the total nicotine being extracted.

3. 100 parts by weight of another dark fired leaf tobacco, containing 3.45% by weight of nicotine and 18.43% by weight of water, and cut to pass an 8 mesh screen, were treated with 3.9 parts by weight of ammonium hydroxide, containing 22% by weight of NH_3 , and the mixture agitated for 15 minutes with a temperature of 60° C. maintained. The ammonia treated material was thereupon immediately extracted with 6 successive portions of naphtha of 156 parts by weight each at a temperature of 60° C. to remove the nicotine. The nicotine was separated from the extract, resulting in 94.7% by weight of the total nicotine being extracted.

4. 100 parts by weight of still another dark fired leaf tobacco, containing 3.15% by weight of nicotine and 25.8% by weight of water, and cut to pass an 8 mesh screen, were treated with

3 parts by weight of ammonium hydroxide, containing 22% by weight of NH_3 , and the mixture agitated for 15 minutes with a temperature of 30° C. maintained. The ammonia treated material was thereupon immediately extracted with 6 successive portions of naphtha of 156 parts by weight each at a temperature of 85° C. to remove the nicotine. The nicotine was separated from the extract, resulting in 95.6% by weight of the total nicotine being extracted.

It is evident that there are numerous factors which will influence conditions for the most satisfactory operation of my invention, the actual limits of which cannot be established except by a detailed study of each set of raw materials and the intermediate and finished products involved.

The vegetable material from which the nicotine is to be extracted may be either leaf or stem. The material should be preferably shredded or coarsely ground, since the finely ground material presents additional mechanical difficulties in treatment. My process is effective for the extraction of nicotine from any vegetable material containing the same and particularly effective for the extraction of nicotine from *Nicotiana rustica* and *Nicotiana tabacum*.

Vegetable material, such as leaf tobacco, will ordinarily contain 12 to 18% of water. Such relatively low water content material may be generally shredded or disintegrated without mechanical difficulties and it is, therefore, preferable to so disintegrate such material prior to making any adjustment of the water content. The water content of the material may then be adjusted up to approximately 45%, although it is generally preferable to maintain this range between 16 and 35%.

The vegetable material so prepared is then treated with ammonia, with the amount of the ammonia equal to at least 1 mol for each mol of the nicotine present in the vegetable material and, preferably, 2 mols of ammonia for each mol of nicotine present. The treatment with ammonia is most effective at superatmospheric temperatures, ranging from 30 to more than 100° C. with 60° C. preferred. This temperature may be reached by adjusting the water content with steam or by heating the water content adjusted tobacco prior to the ammonia treatment. In any event, the time for treatment for the ammonia is limited within a critical range of substantially 3 to 15 minutes, respectively, for the range of 80 to 30° C., respectively. The ammonia treatment may be carried out at atmospheric or superatmospheric pressure.

The ammonia may be admitted as anhydrous ammonia or as an aqueous solution. The latter is preferable and when it is used the amount of water in the aqueous ammonia solution is taken into account as supplying a portion of the water required for the adjustment of the water content to the desired value. When anhydrous ammonia is used, it may be added either to the moisture conditioned vegetable material alone or the latter in contact with the solvent used for the extraction of the nicotine.

The ammonia treated vegetable material is extracted, preferably immediately, after the period of ammonia treatment corresponding to the temperature of the treatment with a suitable solvent. The solvent most suitable for this purpose is a

hydrocarbon solvent which may be a single hydrocarbon or a mixture of hydrocarbons. The latter is generally used, in which case it is preferable to have a mixture having a relatively short distillation range with a high initial boiling point and a low maximum boiling point. A hydrocarbon mixture, such as fighting grade aviation gasoline or a short distillation range naphtha, is particularly suitable for this purpose, although gasoline and kerosene may be used for this purpose. The latter two present some difficulty in removing higher boiling traces from the residue. The extraction may be carried out using gaseous hydrocarbons, such as propane or butane, in the liquid phase. However, this is not ordinarily required in the preparation of the commercial grade of nicotine products, such as nicotine or nicotine compounds. Other solvent, such as alcohols, ethers, esters, halogenated hydrocarbons or ketones may be used for the extraction but the advantages incident to their use do not ordinarily justify the increased costs incident to their use.

The nicotine may be separated from the extract by any suitable means but it is ordinarily preferred to use an aqueous solution of a mineral acid for this purpose. An aqueous solution of sulfuric acid is suitable, and with the proper adjustment with the amount of the concentrate used, can be used to produce a solution containing approximately 40% by weight of nicotine sulfate. When an extract is contacted directly with a solution of sulfuric acid alone, an emulsion which is difficult to separate may be formed if the agitation of the constituents of the mixture is too severe. This trouble may be generally eliminated by treating the extract with a solution containing not only sulfuric acid but nicotine sulfate.

The nicotine sulfate solution may be either dilute or in more concentrated form, such as the 40% nicotine sulfate which is generally sold for dilution for insecticidal use or the nicotine may be separated or separated and purified as the free alkaloid for use as such in the arts and sciences.

My invention may be summarized as the discovery of those conditions of temperature, time, moisture content, and ammonia content under which ammonia enters the vegetable particle to liberate the nicotine from its salts while still enough ammonia remains in the surface water film to overcome the partition coefficient between the aqueous solution of free nicotine and a solution of nicotine in the non-aqueous hydrocarbon, thereby forcing the nicotine from its aqueous phase into the hydrocarbon for solution, with the result accomplished without an excessive use of ammonia which would otherwise require the expense of recovery of such an excess of ammonia.

It will be seen, therefore, that this invention actually may be carried out by the modification of certain details without departing from its spirit or scope.

I claim:

In the process of extracting nicotine from a material containing the same which comprises treating the material with a nonaqueous solvent for the nicotine and separating the nicotine from the solvent extract, the step which comprises treating the extract with an aqueous solution of sulfuric acid and nicotine sulfate.