CONTINUOUS PROCESS OF REMOVING NICOtINE FROM TOBACCO

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This invention generally relates to extraction procedures and is particularly directed to a continuous process of removing nicotine from tobacco and recovering the nicotine in the form of aqueous nicotine salt solutions of relatively high concentration. Such processes have become known for extracting the removal of nicotine from tobacco. In some of the prior art procedures, the tobacco is pretreated with, for example, alkaline substances, whereas the nicotine is removed from the tobacco by treatment with heat, steam or solvents. One of the customary procedures for extracting nicotine from tobacco is to leach the tobacco with water, to wit, the most inexpensive solvent. This results in an effective extraction of the nicotine, but at the same time seriously damages the tobacco and negatively affects its qualities.

With a view to avoiding the drawbacks of water extraction, it has previously been proposed to effect the extraction with organic solvents. In these prior art processes, the nicotine-containing solvent obtained as the result of the extraction is usually freed from the nicotine by heating in selective manner and, after concentration, is recycled to the tobacco either by spraying the tobacco with the concentrated solvent or by evaporating the solvent directly on the tobacco. This is done in order to restore to the tobacco those tobacco constituents which, in addition to the nicotine, are dissolved by the original extraction procedure. However, the economical evaporation of large volumes of liquid, it has been attempted to conduct the organic solvent in a cycle during which nicotine in the solvent is removed and the nicotine-free solvent is recycled to the tobacco, while other tobacco constituents dissolved in the solvent remain in solution. The concentration of such other tobacco constituents thus builds up until a solubility or saturation equilibrium is obtained in the solvent in respect to such other tobacco constituents.

For the purpose of selectively removing the nicotine from the organic phase, it has been suggested to employ precipitants or neutral or acid aqueous salt solutions. The prior art procedures which make use of these measures have, however, serious drawbacks. It is thus known that the denicotinization degree obtained in this manner is extremely low and, additionally, the denicotinization speed is unsatisfactory. Furthermore, considerable amounts of undesired salts and other extraneous components are in this manner incorporated into the tobacco. Moreover, since in the prior art procedures the tobacco is stationarily positioned in the extractor while the extracting agent flows through the tobacco, that portion of the tobacco most closely adjacent the inlet zone for the solvent suffers from being strongly contaminated with salts and other extraneous substances contained in the solvent. This, in turn, results in an uneven purity degree of the tobacco and also in uneven extraction. The result is that the tobacco does not have uniform characteristics in respect to its smoking properties and, moreover, is imparted with undesirable constituents which oftimes affect the quality of the tobacco.

The extracted nicotine has to be processed and recovered in order to prevent waste water pollution and contamination. In addition, of course, the recovery of the nicotine is desirable from an economical point of view. The further processing of the nicotine-containing extract to recover the nicotine constitutes an additional disadvantage of the prior art processes. For example, according to the process of U.S. Patent No. 3,046,997, the nicotine salt containing salt solution obtained in the process must thus be rendered alkaline and then again has to be extracted by means of an organic solvent, whereupon finally the solvent is evaporated. Due to the fact that in the known processes only relatively low nicotine salt concentrations of, for example, below 0.5% are obtained in the aqueous phase, a considerable expenditure in respect to solvents and processing costs is evidently required in order to recover the nicotine.

For the reasons described hereinafter, the prior art processes of the indicated kind either result in reduced quality of the tobacco and/or the economies are unsatisfactory.

Accordingly, it is a primary object of this invention to overcome the drawbacks and disadvantages of the prior art procedures and to provide a process according to which nicotine is effectively and inexpensively removed from tobacco and recovered in the form of nicotine salt solutions of high concentrations.

It is also an object of this invention to provide a procedure for effecting the nicotine removal from tobacco and the subsequent recovery of the nicotine in a continuous or cyclic manner.

Another object of the invention is to provide a procedure of the indicated kind which does not affect the quality of the tobacco and does not deprive the tobacco of other valuable and desirable tobacco constituents.

Generally, it is an object of this invention to improve on the art of nicotine removal from tobacco and the subsequent recovery of the nicotine.

Briefly, and in accordance with this invention, it has been ascertained that the disadvantages of the prior art processes are overcome by first extracting the tobacco with organic solvent by moving the tobacco and the solvent in counter flow relation along the same flow path, whereby a nicotine-containing solution is obtained, and by extracting this solution with an acidic aqueous solution of a pH value of below 2.5, whereby the nicotine passes from the organic to the aqueous phase and is obtained therein in the form of a nicotine salt. Contrary to the prior art procedures, not only the extracting solvent but also the tobacco are thus moved during the extraction. It has been established that the inventive procedure effectively overcomes the prior art disadvantages and that the quality of the tobacco is not affected by the extracting operation. In addition, the inventive process is exceedingly economical. Further, the inventive procedure lends itself particularly well to continuous operation.

The organic solvent should exhibit poor or no solubility with water. The solvent thus should be essentially insoluble or only slightly soluble in water. The solvent is conducted through the extraction system in a circuit and after the solvent has been in contact with the tobacco, the nicotine is removed from the solvent by counter-current contact with the aqueous phase, with the acidic aqueous solution, whereafter the solvent is recycled for further tobacco extraction.

Extensive experiments have demonstrated that the contact period between the tobacco and the organic solvent has a decisive influence on the effectiveness of the nicotine extraction. It has thus been established that the contact period between the solvent and the tobacco should not exceed 180 minutes, the preferred period being between about 45 to 90 minutes. Further, the amount of...
solvent in relation to the tobacco quantity to be extracted is also important. It has thus been found that at least 25 liters of solvent per kilogram of tobacco an hour should be passed through the extractor. A preferred quantity is 100 to 200 liters of solvent per kilogram of tobacco an hour.

As previously stated, the organic solvent after its extraction contact with the tobacco is then extracted in countercurrent flow with an acidic aqueous solution of a pH value of below 2.5. It has been established, in accordance with this invention, that the most favorable results are obtained if the countercurrent extraction of the nicotine-containing solvent with the water is continued until the nicotine content in the aqueous phase is at least 5% by weight, the preferred range being 15 to 25%. When the nicotine content in the aqueous phase has reached the desired value, to wit, between about 5 to 25%, the aqueous nicotine solution is discharged and replaced by a fresh amount of acidic aqueous solution.

The inventive procedure results in a tobacco from which a desired amount of nicotine has been removed, without negatively affecting the aroma and smoking characteristics of the tobacco. However, this is not the only advantage of the inventive procedure, because the invention constitutes the first procedure of this kind which can be economically and investment in respect to labor and equipment, and significant solvent losses are incurred.

Since the organic solvent is circulated through the system and is continuously brought into extracting and washing contact with the aqueous acidic solution for selective removal of the nicotine, the solvent, upon recycling to the tobacco, is again able to extract a fresh amount of nicotine therefrom. Other tobacco constituents which are also soluble in the organic solvent are dissolved from the tobacco during the initial stage of the cycle only and the solvent rapidly becomes enriched with such constituents. The solvent thus becomes essentially saturated with such constituents or a solubility equilibrium will be established between the solvent and the tobacco in respect to such other constituents. In respect to constituents of low solubility, the organic phase is relatively quickly saturated therewith and thus no further amounts of these constituents are thereafter removed from the solvent by recycling to the tobacco.

The situation is similar, although somewhat more complicated, in respect to tobacco constituents which have a great or unlimited solubility in organic solvents. The concentration of such constituents in the solvent may theoretically reach high values. However, since, in accordance with this invention, the tobacco, upon completion of extraction, is not separated from the solvent by compression or centrifuging but is dried in advantageous manner with solvent adhering thereto, while at the same time an equivalent amount of fresh solvent is added to the cycle, the high concentration of such constituents which theoretically are feasible do not occur in practice.

Also in respect to these constituents, a kind of equilibrium condition is established which is characterized by the fact that the amount of these constituents which is restored to the tobacco as the result of the drying with the adhering solvent is essentially equal to the amount of the constituents extracted from the tobacco by the solvent. In this manner, the tobacco is not impoverished in respect to valuable tobacco constituents other than nicotine. In practice, therefore, the tobacco, after the desired amount of nicotine has been extracted therefrom, is thus dried with an adhering amount of organic solvent containing dissolved therein a quantity of such other constituents corresponding to the quantity of such constituents which originally has been removed during the extraction. If the tobacco is thus dried, the solvent evaporates and the tobacco constituents dissolved in the solvent are thus restored to the tobacco. The evaporating solvent may be recovered and recycled to the system. It has been established that drying of tobacco with about 1.5 to 4 liters of solvent per kilogram of tobacco adhering thereto restores the originally dissolved tobacco constituents to the tobacco as a result of its extracting contact with such other tobacco constituents prevails in the solvent. In this manner, the tobacco, after terminated treatment, thus contains the same amount of such other constituents than it did prior to the extraction. The amount of solvent which is thus evaporated from the treated tobacco is replenished in the cycle by a fresh amount of solvent or by recycling the condensed solvent vapors. As previously set forth, the amount of solvent used for extracting the tobacco should be at least 25 liters per kilogram of tobacco an hour. This is the lower limit value at which an economical and effective extraction at the prevailing flow and concentration conditions can be effected. However, a larger amount of solvent is preferred and, as previously stated, 100 to 200 liters of solvent per hour and kilogram of tobacco yield excellent results at a relatively short contact time of, for example, 45 to 90 minutes.

If the extraction time, to wit, the contact time between the tobacco and the organic phase, exceeds the 180-minute limit, the extraction effect is not significantly increased and longer extraction periods are therefore unnecessary and only render the procedure more expensive.

The organic solvent which thus becomes enriched with nicotine as well as respect to labor and equipment, is therefore subjected to extraction in two successive extraction stages. In such two-stage extraction procedures a fresh amount of solvent is then supplied to the system to replace the discharged portion. The acid concentration of the fresh acidic phase introduced into the system to make up for the discharged portion is chosen so that not only the pH value is maintained below 2.5 but also that the volume of the acidic phase in the system is replenished. Essentially the same amount of acidic phase is thus present within the system throughout the cycle.

The extraction of the organic phase with the acidic aqueous phase is advantageously effected in two successive extraction stages. In such two-stage extraction pro-
It has been found to be particularly advantageous if the pH value of the aqueous phase in the second stage is below 2.0. For this purpose, a sufficient volume of fresh acidic solution of sufficiently low acidity may be fed to the second extraction stage to obtain the desired pH value and to make up for withdrawn nicotine-containing aqueous solution. A corresponding amount of acidic wash solution is fed from the second stage to the first stage. In this manner, a particularly thorough and extensive transfer of nicotine from the organic phase is obtained within a very short period of time.

In the second extraction stage, which, as set forth, is carried out at a pH value of below 2.0, acid is consumed for binding the nicotine. In order to make sure that the pH value in the first extraction stage therefore does not rise to an undesired high value, but is maintained at a value below 2.5, fresh acidic solution has also to be supplied to the first extraction stage if the acidity of the solution flowing from the second stage into the first stage should not be sufficient to maintain the indicated low pH value in the first stage. The concentration of the acid in such a two-stage procedure should also be chosen so that the volume of aqueous phase carried along by the organic phase is made up by supplied fresh solution.

According to a modification of the invention, a third extraction stage may be arranged behind the two previously mentioned ones, wherein further extraction of the organic phase is carried out with substantially pure water or with a very diluted acidic solution. Such third extraction stage prevents the incorporation of even very small amounts of acid into the organic phase, since the water effectively removes acid from the organic phase. Even traces of dissolved acid or acid in drop form are thus completely and effectively removed from the organic solvent. The aqueous extraction agent for the third extraction stage is continuously or intermittently withdrawn and is advantageously used for preparing fresh acid solution for the first and/or second extraction stages.

The aqueous phase discharged from the first extraction stage contains, in accordance with the teachings of this invention, already more than 5% of nicotine in salt form. Preferably, the nicotine concentration of the aqueous phase emanating from the first stage has already reached a value of 15 to 25% so that, in fact, a preconcentrated nicotine salt solution is obtained. In practice, nicotine salt solutions are customarily employed in concentrations of above 30% and preferably 40% by weight and are marketed in this manner, a further concentration of the withdrawn aqueous phase has to be effected. This may be accomplished by evaporation of the water. In this connection, it should be observed, however, that the requirements in respect to apparatus and energy for this purpose are insignificant. For example, let us assume that the aqueous phase discharged from the first extraction stage has a nicotine salt concentration of 20%. In order to concentrate 100 kilograms of such a solution to a nicotine salt concentration of 40%, only 50 kilograms of water have to be evaporated.

It will be appreciated that the particular choice of the organic solvent used for the extraction of the tobacco is a factor for the efficacy and economy of the procedure. Solvents which have high solubility with water, of course, cannot be used. Therefore, acetone and isopropanol, for example, are unsuitable. Further, solvents having a relatively high mutual miscibility with water as, for example, isobutanol, methyl ethyl ketone, methylecylacetate, and ethylecylacetate, are also undesirable for the inventive purposes. Other solvents again have a very low extraction effect so that their employment does not result in an economically sound procedure. Such solvents are, for example, certain lower aliphatic hydrocarbons as, for example, hexane. Benzene, cyclohexane and isopropyl ether are moderately effective in the inventive purposes. The most suitable solvents for the inventive purposes are solvents pertaining to the group of aliphatic halogen hydrocarbons. However, some of these compounds, again, are less efficient than others. The less efficient are, for example, 1,1,2-trichloro-1,2,2-trifluoroethane, while more efficient compounds of this group are 1,1,1-trichloroethane, trichloroethene, 1,2-

The organic solvent solution for extracting the nicotine from the organic phase may comprise or consist of aqueous sulfuric acid, hydrochloric acid or phosphoric acid. These three acids are particularly suitable for the inventive purpose.

The various features of novelty which characterize the invention are pointed out with particularity in the claims annexed to and forming a part of this specification. For a better understanding of the invention, its operating advantages and specific objects attained by its use, reference should be had to the accompanying drawings and descriptive matter in which there is illustrated and described a preferred embodiment of the invention.

In the drawings:

The single figure of the drawings diagrammatically illustrates a plant for carrying out the inventive extraction cycle.

The plant comprises a solid-liquid extractor 1. Tobacco contained in a storage container 2 is continuously fed through conveyor 99 to one end of the solid-liquid extractor 1, to wit, the righthand end a as indicated in the drawing. The tobacco moves within the extractor 1 from end a toward end b and for this purpose suitable conveying means are provided (not shown). At the same time, organic solvent flows through conduit 98 into the extractor 1. It will be noted that the solvent enters the extractor at end a and flows in counter-current to the tobacco.

The organic solvent exits from the extractor at end a through conduit 97 and flows successively through the liquid-liquid extractors 3, 4 and 5. The solvent thus continuously circulates through the system in the manner described.

In the following, the aqueous circuit will be explained:

The aqueous system comprises an acid preparation vessel 6. Acidic solution of a pH value of below 2.0 is fed from the vessel 6 through line 93 into the bottom end of the extractor 4, where the aqueous acidic solution meets the nicotine-containing organic solvent entering the top of the column through line 96. The organic solvent and the aqueous solution thus flow in counter-current to the aqueous solution being discharged adjacent the top of the column through line 92 to enter the bottom end of the first liquid-liquid extractor 3. Where, again, the aqueous solution flows in counter-current to the organic solvent entering column 3 through line 97. The aqueous phase is discharged from extractor 3 near its top through line 91 and flows into the evaporator 7 where water is turned off.

The concentrated nicotine salt solution formed in evaporator 7 flows through line 96 into the receptacle 8 where the concentrate is collected.

Storage vessel 11 contains water. Water from the storage vessel 11 flows through lines 82, 81a and 87 into the bottom end of the third extraction stage 5 in which the
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last remnants of nicotine and acid are removed from the organic phase. If desired, slightly acidic water may be supplied to the extractor 5 from vessel 9, the latter being connected with the bottom end of extractor 5 through line 87. Supply container 10 contains diluted acid which flows through line 86 into the first extraction stage, to wit, extractor 3. In order to maintain the pH value in the first extraction stage below 2.5, the diluted acids in vessels or containers 6 and 10 are prepared by mixing therein concentrated acid which is supplied from the acid container 12 through lines 84 and 83, respectively, and water emanating from the water storage container 11, which discharges into vessels 6 and 10 through lines 82 and 81, 80, respectively. The water or diluted acid solution which exits from the top of extractor 5 is advantageously conveyed to the container 6 and/or 10 through line 88 via an interposed container (not shown). The distillate obtained in the evaporator 7 may be used for the aqueous phase supplied to the extractor 5.

The tobacco, upon extraction with the organic solvent, is discharged from end b of extractor 1 through line 79 and enters the drying unit 13 with organic solvent adhering to the tobacco. The wet tobacco is dried in the unit 13 and is collected as dry tobacco in the receptacle 14. The solvent vapors during the drying are discharged through conduit 78 and enter the recovery unit 15 where the vapors are condensed or absorbed. The thus recovered solvent is then recycled to the solvent circuit through conduit 75 which discharges into line 94. In order to compensate for any losses of organic solvent, continuously or intermittently fresh solvent is supplied from the solvent storage unit 16 which discharges into line 98 previously referred to.

It will be appreciated that the plant has not been shown with all its details and the pumps, valves, conveyors, flow rate measuring devices and the like have not been indicated, since the showing is a diagrammatical one, the provision of these devices being obvious to a person skilled in this art.

Furthermore, the representation as given hereinafter does not indicate whether the organic solvent is heavier or lighter than water.

In order further to process the extracted tobacco, the latter is first liberated from the adhering solvent as described in connection with the drier 13. In this manner, tobacco constituents originally removed from the tobacco are thus restored to it. The tobacco is then further processed in any manner known to the art.

The various units of the plant described hereinafter may be of conventional construction and the plant may be a suitable combination of units as they are known from the chemical industry. The particular choice in respect to the solid-liquid extractors, the driers, the solvent recovery unit as well as the pumps, filters and measuring devices will be dependent on the circumstances and the desired capacity of the plant.

The invention will now be described in respect to several examples, it being understood, however, that these examples are given by way of illustration and not by way of limitation and that many changes may be effected without affecting in any way the scope and spirit of the invention as recited in the appended claims.

Example I

This experiment was carried out with raw Burley tobacco. 100 kilograms per hour of the tobacco were passed in continuous manner from one end of a solid-liquid extractor to the other end, while at the same time 20 cubic meters of methylene chloride per hour were passed through the extractor in the opposite flow direction. The extraction of the tobacco with the methylene chloride was effected at room temperature. The contact period between tobacco with the solvent in the extractor amounted to 60 minutes. The nicotine enriched organic solvent thus obtained was then fed to the first extraction stage for counter-current extraction with the aqueous phase which consisted of diluted sulphuric acid of a pH value of 2.1. The pH value of the aqueous phase in the second extraction stage was 1.8. The third extraction stage was operated with pure water. The tobacco, upon drying, had a nicotine content of 0.52% by weight while the original nicotine content of the tobacco was 3.50% by weight.

The aqueous nicotine salt solution discharge from the first extraction stage in a continuous manner had a nicotine content, calculated as the alkaloid base, of 15.1% by weight. This aqueous nicotine salt solution was continuously concentrated under vacuum conditions in a circulating evaporator to a nicotine content of 40% by weight.

Example II

This experiment was carried out with raw Virginia tobacco having a nicotine content of 3.80% by weight. 50 kilograms per hour of the tobacco were moved through a solid-liquid extractor and extracted with methylene chloride, flowing through the extractor in counter-current relative to the tobacco movement. The extraction was carried out at room temperature. The amount of methylene chloride which was forced through the extractor per hour amounted to 5 cubic meters. The contact period of the tobacco with the organic solvent was 100 minutes. After drying of the tobacco, the nicotine content was reduced to 0.60% by weight.

The aqueous solution of the first extraction stage had a pH value of an average of 2.0 while the pH value of the aqueous phase in the second extraction stage had an average value of 1.2. The aqueous solution discharged from the first extraction stage had a nicotine content of 24.2% by weight calculated on the nicotine base. The pH value of this solution was first reduced to 5.5 and then introduced into the solution gaseous ammonia. The solution thus obtained was subsequently concentrated in a distillation still at normal pressure to a nicotine content of 40% by weight, calculated as alkaloid base.

Example III

This test was carried out with a tobacco having a nicotine content of 2.90% by weight. 7.0 kilograms of tobacco per hour were fed through a small solid-liquid extractor which was simultaneously and continuously fed with 2 cubic meters per hour of benzene. The benzene was continuously recirculated and passed through the extractor in counter-flow relation to the movement of the tobacco. The tobacco was continuously discharged from the extractor and dried in a drying unit. The contact period of the tobacco with the organic solvent in the extractor amounted to 2 hours. The pH value of the aqueous phase in the first extraction stage was 2.4 while the corresponding value in the second extraction stage was 1.5. No third extraction stage was employed in this test. The nicotine content of the dried tobacco amounted to 0.95%.

The aqueous phase was withdrawn intermittently from the first extraction stage at a nicotine content of 10.0% by weight calculated on the alkaloid base. By evaporating water, the nicotine content was increased to 40.0% by weight.

While the invention has been described in connection with the extraction of nicotine from tobacco, which is the major application for the inventive process, it should be appreciated that other basic constituents of the tobacco, for example, the secondary alkaloids contained in tobacco in small quantities, may also be removed by the inventive procedure. Further, the inventive procedure is applicable to the extraction of other substances, as, for example, alkaloid containing plant material used for pharmaceutical purposes, whereby the basic active ingredients of the plant material can be economically and effectively recovered.

While specific embodiments of the invention have been shown and described in detail to illustrate the application of the inventive principles, it will be understood that the invention may be embodied otherwise without departing from such principles.
What is claimed is:

1. A continuous process of removing nicotine from tobacco which comprises:
   (a) moving tobacco and an organic solvent in counter flow relation along the same flow path, said organic solvent being capable of dissolving nicotine and exhibiting poor miscibility with water, whereby a nicotine-containing solution is obtained;
   (b) extracting the nicotine-containing solution in counter flow relation with an acidic aqueous solution having a pH value of below 2.5, whereby nicotine passes from the organic solvent phase to the aqueous solution;
   (c) recycling thereafter the organic solvent for extraction of a fresh amount of nicotine from tobacco;
   (d) withdrawing a portion of the aqueous solution when the nicotine content therein has reached a value of at least 5.0% by weight; and
   (e) replacing the withdrawn portion of aqueous solution with a fresh amount of acidic aqueous solution having an acidity sufficient to maintain the pH in the aqueous solution at a value below 2.5.

2. A process as claimed in claim 1, wherein the contact time between the tobacco and the organic solvent moving in countercurrent to each other is at most 180 minutes.

3. A process as claimed in claim 2, wherein the contact time is between about 45 to 90 minutes.

4. A process as claimed in claim 1, wherein the amount of organic solvent flowing in counter flow relation to the tobacco is at least 25 liters per hour and per kilogram of tobacco.

5. A process as claimed in claim 1, wherein the amount of organic solvent flowing in counter flow relation to said tobacco is between about 100-200 liters per hour and kilogram of tobacco.

6. A process as claimed in claim 1, wherein the aqueous solution is contacted in counter flow relation with the nicotine-containing organic solvent solution until the nicotine concentration in the aqueous solution is between 15 to 25% by weight.

7. A process as claimed in claim 1, wherein the tobacco, after its extracting contact with the organic solvent, is continuously removed with about 1.5-4.0 liters of organic solvent per kilogram of tobacco adhering thereto and is dried with said organic solvent, whereby the solvent evaporates and tobacco constituents, except nicotine, contained in the solvent are restored to the tobacco.

8. A process as claimed in claim 1, wherein the extraction of the nicotine-containing organic solution with the acidic aqueous solution is performed in two successive extraction stages, to wit, a first extraction stage in which the aqueous solution has a pH value of below 2.5, and a second stage in which the aqueous solution has a pH value of below 2.0.

9. A process as claimed in claim 8, wherein the aqueous solution of the first stage at least partially emanates from said second stage.

10. A process as claimed in claim 8, wherein the aqueous solution in said first stage is made up from the aqueous solution emanating from said second stage and from fresh aqueous acidic solution.

11. A process as claimed in claim 8, wherein at least a major portion of the aqueous solution in said second stage consists of fresh aqueous acidic solution essentially devoid of nicotine.

12. A process as claimed in claim 8, wherein the organic solvent emanating from said second stage is further extracted in a third stage, the extraction in the third stage being effected with pure water.

13. A process as claimed in claim 8, wherein the organic solvent emanating from the second stage is further extracted in a third stage, the extraction in the third stage being effected with a slightly acidic aqueous solution.

14. A process as claimed in claim 12, wherein the aqueous solution obtained in said third stage is used for preparing the aqueous solution of at least one of said first and second stages.

15. A process as claimed in claim 1, wherein the withdrawn aqueous nicotine salt-containing solution is further concentrated to a nicotine content of at least 30% by weight.

16. A process as claimed in claim 1, wherein said solvent is methylene chloride.

17. A continuous process for removing nicotine from tobacco and for recovering the nicotine in the form of a concentrated aqueous nicotine salt solution which comprises:
   (a) continuously feeding tobacco through a solid-liquid extractor while at the same time feeding an organic solvent capable of dissolving nicotine and exhibiting poor miscibility with water through the extractor in counter-current, whereby a nicotine-containing solution is obtained, the amount of solvent fed through said extractor being between about 25 to 200 liters per hour and kilogram of tobacco, the contact time between said solvent and said tobacco being between about 45 and 180 minutes;
   (b) extracting the nicotine-containing solution flowing from the extractor in counter-current with an acidic aqueous solution having a pH value of below 2.5, whereby an aqueous nicotine salt solution is obtained;
   (c) recycling the organic solvent after the nicotine has been extracted therefrom by the aqueous solution for extracting a fresh amount of nicotine from tobacco;
   (d) continuously discharging tobacco from the extractor and drying the tobacco with organic solvent adhering thereto;
   (e) continuing the counter flow extraction of the organic solvent with the acidic aqueous solution, until the nicotine content in the aqueous solution is between about 5 to 25% by weight;
   (f) thereafter withdrawing the aqueous nicotine salt solution and concentrating it to a higher nicotine salt concentration; and
   (g) replacing the withdrawn amount of aqueous nicotine salt solution by a fresh amount of acidic aqueous solution.

18. A continuous process as claimed in claim 17, wherein the amount of organic solvent adhering to the tobacco (d) is about between 1.5-4.0 liters per kilogram of tobacco.

References Cited

UNITED STATES PATENTS

3,046,997 7/1962 Hind ----------------- 131—143
3,096,773 7/1962 Neukomm et al. ------ 131—143 X
3,139,435 6/1964 Stailey et al. ------- 131—143 X

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