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PROCESS FOR EXTRACTING SUBSTANCES FROM PLANT PARTICLES

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2 Sheets-Sheet 1

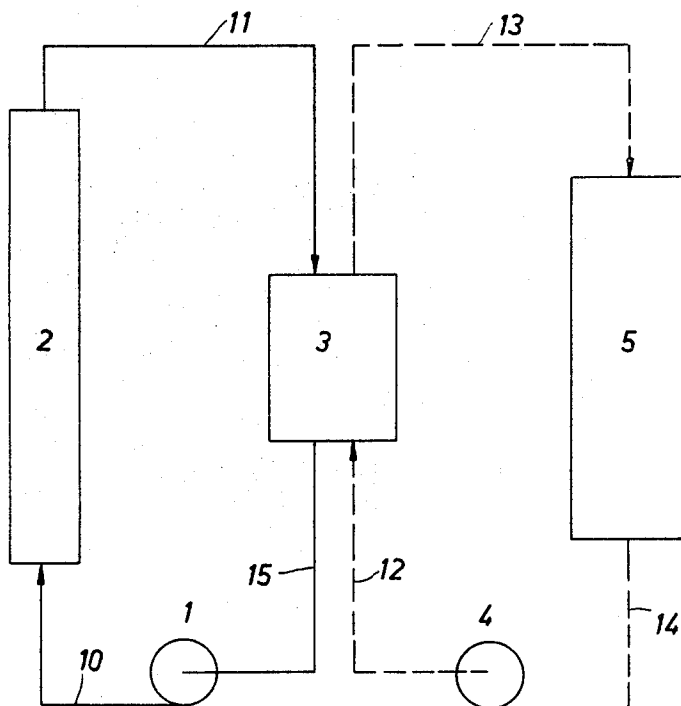


Fig. 1

INVENTORS

MAX FREIHERR VON BETHMANN

GERHARD LIPP

HELMUT BAYER

By *Mendelow and Torrey*
Attorneys

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2 Sheets-Sheet 2

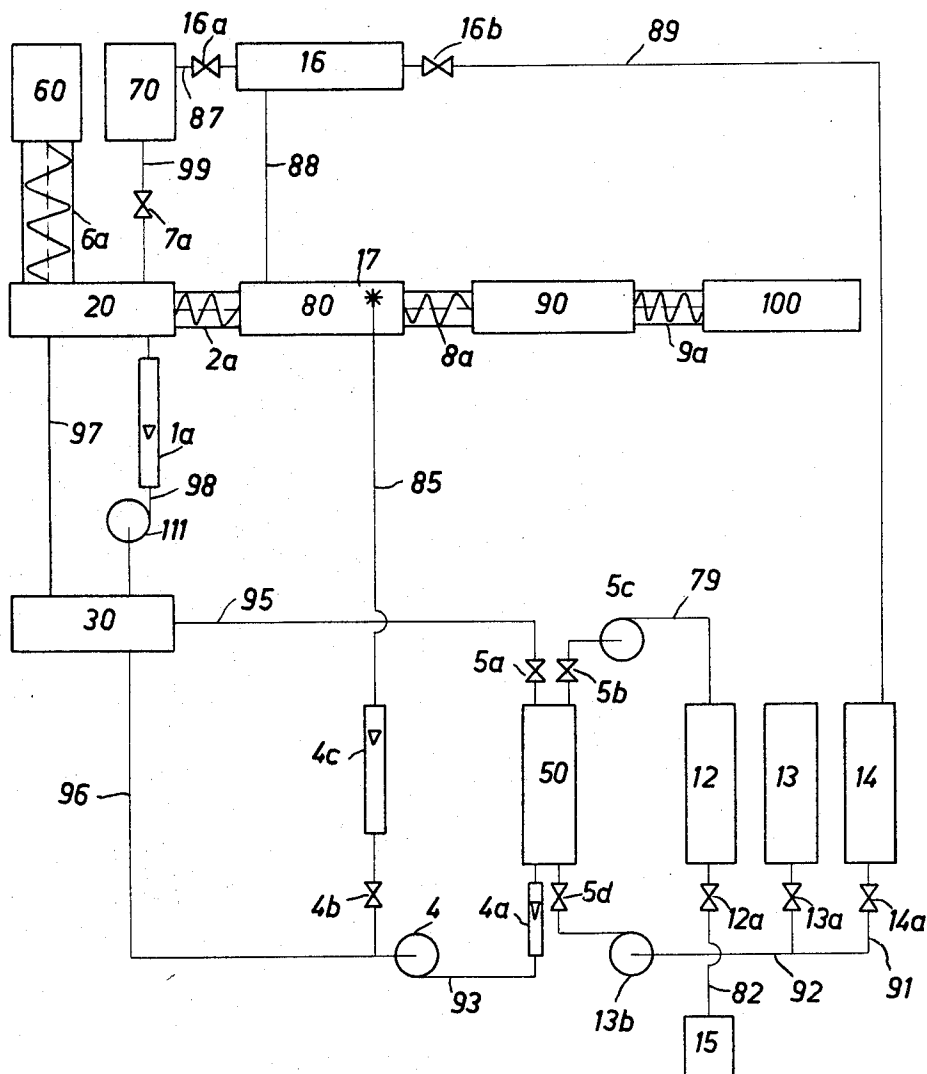


Fig. 2

INVENTORS
MAX FREIHERR VON BETHMANN
GERHARD LIPP
HELMUT BAYER

By Malcolm and Town
Attorneys

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3,390,685

PROCESS FOR EXTRACTING SUBSTANCES FROM PLANT PARTICLES

Max Freiherr Von Bethmann and Gerhard Lipp, Bremen, and Helmut Bayer, Muhlheim (Main), Germany, assignors to Eresta Warenhandels-gesellschaft mit beschrankter Haftung, Muhlheim (Main), Germany

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15 Claims. (Cl. 131-143)

This invention generally relates to extraction procedures and is particularly directed to a process for the selective separation of particular basic and/or acid constituents from solid plant particles by first extracting the plant particles with an organic solvent which is essentially insoluble in water and thereafter subjecting the enriched solvent to extraction with an aqueous solution of acidic or basic reaction.

While the invention may successfully be used for the selective extraction of particular substances from a great variety of plant particles, it is particularly applicable to and will therefore in the following primarily be described in connection with the extraction of nicotine from tobacco.

Various processes have become known for effecting the removal of nicotine from tobacco by extracting the tobacco with a solvent. In developing nicotine extracting processes, it has to be considered that tobacco contains a plurality of substances, other than nicotine, which are soluble in water and organic solvents, the removal of which, however, is undesired. In some of the prior art nicotine extracting processes, the tobacco, sometimes after a suitable pre-treatment, is first extracted with water or an organic solvent, whereafter the nicotine is removed from the solution thus obtained. After the nicotine has been separated from the solution, the remaining solution, which contains other substances dissolved from the tobacco, is then recycled to the tobacco so as to restore said other substances to the tobacco.

If the original extraction of the tobacco is carried out with organic solvents, the nicotine is removed from the solvent-nicotine system, for example, by washing with acids or aqueous salt solutions. Since, as stated, the solvent, after the removal of the nicotine, is recycled to the tobacco, it cannot be avoided in these prior art processes that a portion of the acids or salts in dissolved or suspended form are incorporated into the tobacco and negatively influence the quality of the latter. Further, a portion of the soluble tobacco constituents or components, other than nicotine, which are leached by the solvent remain in the solution which is used for removing the nicotine from the solvent and are thus lost from the tobacco. This again seriously detracts from the tobacco quality.

More recently, it has been proposed to extract tobacco with water and to remove the nicotine from the aqueous solution obtained by ion exchange since in so doing the acids are present in solids, insoluble form and the disadvantages hereinabove referred are thus eliminated. This prior art process, has, however, the drawback that the original condition of the tobacco is altered in an unfavorable manner. For example, the tobacco becomes in this manner impoverished in respect to inorganic cations, a fact which leads to a significant deterioration of the burning characteristics of the tobacco. Due to the fact that many tobacco constituents exhibit a very high solubility in water, the equilibrium concentration which adjusts itself is in this instance at about 30 percent. Moreover, in the last-mentioned prior art process, the tobacco loses some of its desirable physical characteristics. It should

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also be considered that large amounts of heat energy are necessary to dry the considerable amounts of water which are obtained in this process.

U.S. Patent 3,046,997 discloses a procedure for the nicotine removal from tobacco, according to which the tobacco is extracted by means of organic solvents which are miscible with water to a limited extent only. The organic phase which is separated from the tobacco is subsequently subjected to a liquid-liquid extraction with aqueous solutions of inorganic salts and acids and the nicotine is separated from the aqueous solution by renewed solvent extraction. This process has the advantage that the tobacco does not come into direct contact with the water. However, due to the mutual solubility of the aqueous solution and the organic phase, it cannot be prevented that salts, and in particular inorganic mineral acids whose solubility in organic solvents is relatively high, are introduced into the tobacco when the organic phase is recycled to the latter. The mineral acids, which thus are incorporated into the tobacco structure upon recycling of the organic solvent, form salts with the nicotine of the tobacco. Contrary to nicotine salts of organic acids, the nicotine salts of mineral acids have an extremely low solubility in organic solvents. Consequently, even prolonged extraction with organic solvents does not result in a satisfactory denicotinization of the tobacco. The mineral acids thus fix the nicotine on the tobacco in a certain manner, instead of removing it.

Accordingly, it is a primary object of the present invention to overcome the disadvantages of the prior art nicotine removal processes and to provide a process according to which nicotine is effectively removed from the tobacco in a simple and superior manner without affecting the overall quality of the tobacco.

It is also an object of this invention to provide a process of the indicated kind, wherein the tobacco is not impoverished in respect to soluble constituents other than nicotine.

Another object of this invention is to provide a process of the indicated kind which lends itself to continuous operation.

Generally, it is an object of the invention to improve on the art of selective removal of substances from solid plant particles by extraction as presently practiced.

Briefly, and in accordance with this invention, the tobacco is first extracted with an organic solvent to remove nicotine from the tobacco. The solution thus obtained is thereafter extracted with water which is enriched or essentially saturated with those tobacco substances which are soluble in the organic solvent and water, except for nicotine, whereby an aqueous nicotine solution is obtained. The aqueous nicotine solution is then subjected to ion exchange to remove the nicotine therefrom.

From a practical point of view, the aqueous solution used for extracting the organic nicotine-containing solvent should be enriched or essentially saturated with substances emanating from the respective plant particles such as organic acids contained in tobacco.

The organic solvent should be miscible with water to a limited extent only, a characteristic which may be expressed in different language by stating that the solvent should be essentially insoluble in water.

When the tobacco is extracted with the organic solvent, not only nicotine but a number of other soluble tobacco constituents are dissolved from the tobacco. In order to avoid loss of such other constituents from the tobacco, the organic solvent is repeatedly reused for extraction purposes until the concentration of these other constituents in the organic solvent has reached a value so that upon drying of the treated tobacco to remove adhering solvent by evaporation, the amount of such constituents original-

ly dissolved from the tobacco by the solvent is again restored to the tobacco.

In the same manner, the concentration of the tobacco constituents which are soluble and present in the aqueous phase and which are not removed from the aqueous phase by ion exchange should be so high that a solubility equilibrium is created between the organic and the aqueous phase. In accordance with the invention, the aqueous phase does not contain any constituents which are extraneous to tobacco so that after the nicotine has been removed from the aqueous phase by ion exchange, any suitable portion of the aqueous phase may be separated and added to the extracted tobacco, for example, for conditioning purposes. The removed part of water may then be replaced by a corresponding amount of fresh water.

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 are illustrated and described preferred embodiments of the invention.

In the drawings:

FIG. 1 is a diagrammatical representation of the inventive process principle; and

FIG. 2 shows diagrammatically a preferred embodiment for a plant for the continuous performance of the inventive process.

Referring now to the diagrammatic showing of FIG. 1, which explains the inventive process principles, it will be noted that a pump 1 is provided which feeds organic solvent through the conduit 10 into extraction means 2 designed for the extraction of a solid by a liquid. The arrangement furthermore comprises a liquid-liquid extractor 3 which is connected with the solid-liquid extractor 2 by means of the conduit 11. A further pump 4 feeds aqueous solution through the conduit 12 into the liquid-liquid extractor 3. Aqueous liquid emanating from the extractor 3 is withdrawn through conduit 13 and enters the top of an ion exchange column 5 or the like in exchange means. The effluent from the column 5 is conveyed through conduit 14 and back through conduit 12 into the liquid-liquid extractor 3. It will be noted that the aqueous solution which enters the liquid-liquid extractor 3 through conduit 12 flows in countercurrent to the organic solvent solution which enters the extractor 3 through conduit 11 and is withdrawn from the extractor 3 through the conduit 15. Essentially, therefore, two circuits are established, to wit, the organic solvent circuit 10, 2, 11, 3, 15 and the circuit of the aqueous system 14, 12, 3, 13 and 5, the liquid being circulated by means of the pumps 1 and 4, respectively.

The tobacco to be extracted is located in the solid-liquid extractor 2 and the circulating organic solvent thus dissolves from the tobacco both nicotine or its salts and, other soluble tobacco constituents. After some time, however, the organic solvent is enriched with the other soluble tobacco constituents. By contrast, since the nicotine, dissolved in the organic solvent, is constantly removed therefrom by means of the aqueous solution in the liquid-liquid extractor 3, the organic solvent is capable constantly to dissolve fresh amounts of nicotine from the tobacco. It will thus be appreciated that nicotine is continuously removed from the tobacco, and in turn from the organic phase by the aqueous solution, while other soluble tobacco constituents are dissolved in the organic solvent until an equilibrium condition has been established. After a desired extraction degree has been reached, the tobacco is removed from the extractor 2 and is dried in a drier to evaporate adhering organic solvent. It has been ascertained that the equilibrium condition in respect to the other constituents referred to, is established, if the amount of soluble tobacco constituents which is

restored to the dried tobacco by the evaporation of the adhering solvent, is equal to the amount of such constituents dissolved during the extraction with the organic solvent. In this manner, the amount of the other tobacco constituents originally dissolved during the extraction but restored during the subsequent drying will remain constant in the tobacco so that in effect no loss of these desirable constituents takes place.

From a practical point of view it has been found to be particularly advantageous to take out the tobacco from the extraction with an adhering amount of organic solvent which is at least twice the weight of the tobacco proper.

As previously stated, the circulating aqueous solution which flows through conduits 14 and 12 into the liquid-liquid extractor and from there through conduit 13 into the ion exchange column, is essentially saturated with water-soluble tobacco constituents so that essentially no water-soluble tobacco constituents are dissolved by the water during its contact with the organic phase. However, at the beginning of the operation and in the event that ordinary water is used instead of water previously enriched with soluble tobacco constituents, the water, of course, not only dissolves the nicotine contained in the organic phase, but will also be enriched with water-soluble tobacco constituents which have been leached out from the tobacco by the organic solvent. Since, in accordance with the invention, the nicotine is removed from the aqueous phase by means of ion exchange which takes place in the column 5, the aqueous system thus never reaches a state of saturation in respect to nicotine, so that constantly fresh amounts of nicotine can be removed by the water from the organic phase.

The aqueous phase dissolves other water-soluble tobacco constituents from the organic phase until a solubility equilibrium condition has been established. Once this equilibrium condition prevails, the amount of water-soluble tobacco constituents in the aqueous phase remains essentially constant since these constituents are not removed by the ion exchange. If desired, the concentration of these other water-soluble tobacco constituents may be limited in the aqueous phase by continuously or intermittently recycling a portion of the aqueous phase to the treated tobacco and replacing the recycled portion by fresh water. A separate recycling of the water to the tobacco is rendered unnecessary if the aqueous solution is used for the production of the customary tobacco sauces. The aqueous phase is particularly suitable for this purpose since it is essentially free from nicotine and any extraneous components.

Once the equilibrium conditions of the organic and aqueous phases have been reached, only nicotine and maybe some insignificant amounts of other basic compounds are extracted from the tobacco, while all the other tobacco constituents remain in the tobacco. In contrast to prior art processes, this fact also applies to the organic acids of the tobacco, for example citric acid, malic acid and tartaric acid which, as known positively influence the quality of the tobacco.

It will be appreciated from the above that the inventive process is characterized by a three-fold selectivity which is not obtainable by prior art processes. This three-fold selectivity is attained by the advantageous transfer of the nicotine through the three-phase boundaries: tobacco-organic solvent, organic solvent-water, and water-ion exchanger.

Other advantages of the inventive process are due to the fact that no direct contact of the tobacco with the aqueous phase takes place during extraction, and extraneous acids and salts, to wit, substances which do not emanate from tobacco, are advantageously kept away from the system. Since after attainment of the solubility equilibria referred to, no further tobacco constituents, except for nicotine, are extracted from the tobacco, there is no need to enrich the tobacco with such constituents at

a later stage, so that a special recycling of such constituents is not necessary in the present inventive procedure. In this connection it will be appreciated that in the prior art denicotinization processes, due to the required concentration of the extracts which are obtained, quality losses are incurred as well as losses in time and expenditure.

The aqueous solution which exits from the liquid-liquid extractor 3 enriched with nicotine or nicotine salts, enters the top of the ion exchange column 5. The aqueous effluent discharging from the bottom of the column 5 contains only insignificant traces of nicotine and exhibits an acidic reaction. This is due to the tobacco acids which are contained in the aqueous phase and which are liberated from their salt form in the ion exchanger. The pH value of the aqueous phase varies dependent on the particular kind of tobacco and the process conditions, but is always higher before the aqueous phase enters the ion exchanger than it is in the effluent exiting from the column. To the extent that these tobacco acids are bound into salts by the basic components of the tobacco, they are again liberated when the aqueous phase passes through the ion exchanger. In this manner, and in spite of a relatively small quantity of acids in the aqueous phase, a predetermined low pH value is constantly maintained. This low pH value advantageously facilitates the removal of the nicotine from the organic phase into the aqueous phase during the extraction contact.

A further lowering of the pH value in the aqueous phase may be accomplished by adding to the aqueous phase small amounts of extraneous acids as, for example, sulphuric acid, hydrochloric acid, phosphoric acid or citric acid. Contrary to the known processes, however, it is not necessary to use stoichiometric amounts but very small quantities are sufficient, since, as stated, the ion exchanger constantly regenerates the free acids from the salts which are formed by neutralization with the nicotine.

In respect to the ion exchangers, the customary natural or artificial exchange materials as they are available on the market may be employed. Cation exchangers are advantageously used in the acid form because, as mentioned above, the natural acids of the tobacco will thus be retained in the aqueous system and thus may ultimately be restored to the tobacco.

In respect to the organic solvents, many organic liquids which are miscible with water to a limited extent only, are suitable for the inventive process. Such solvents are, for example, ketones, such as butanone and cyclohexanone; various esters such as methylacetate, ethylacetate or butylacetate; ethers, such as diethylether, diisopropylether and diisobutylether; hydrocarbons such as pentane, hexane, cyclohexane, benzene and toluene; further, halogenated hydrocarbons such as tetracarbochloride, chloroform, methylenechloride, trichloroethylene, monofluorodichloroethylene and difluorodichloroethylene; moreover, alcohols which dissolve in water with difficulty only, such as isobutanol, amylalcohol and cyclohexanol are embraced within the inventive concept. This list, however, is not exhaustive, because other solvents may be considered to the extent that they are miscible with water to a limited extent only and do not have a very high boiling point. Generically it may be stated that any solvent is suitable which is substantially insoluble in water.

Halogenated aliphatic hydrocarbons, for example, methylenechloride are particularly preferred as organic solvents in the inventive process, due to their excellent extraction effect and their incombustibility.

Mixtures of two or several different solvents may also advantageously be used, since this may increase the extraction effect and may simplify the process. If solvent mixtures are used, solvents may be employed as additives which are miscible with water. Alcohols such as methanol, or ketones such as acetone, may thus be used as components in solvent mixtures.

As previously set forth, the inventive process is particularly suitable for the extraction of nicotine from

tobacco. The extraction may be carried out at any processing stage of the tobacco, primarily, however, with raw tobacco, cut tobacco and tobacco from which the ribs of the leaves have been removed.

The moisture content of the tobacco to be extracted should be adapted to the respective process conditions and should generally have an average value. If the tobacco is very dry, undesired breakage of the tobacco structure may occur, while very moist tobacco requires additional energy for drying purposes. Therefore, both very dry and very moist tobacco may negatively affect the economics of the process. Experience has demonstrated that in most instances a tobacco moisture content of between 12 to 20% gives excellent results. It should be appreciated, however, that this range is not limiting and that tobacco of a different moisture content may also be processed in accordance with the invention.

Customary apparatus generally used in the tobacco industry and in chemical processing plants in general may be used for carrying out the inventive process, even if the process is performed continuously.

However, for the continuous performance of the inventive procedure, an arrangement as diagrammatically depicted in FIG. 2 is particularly suitable. The apparatus of FIG. 2 comprises a solid-liquid extractor 20 which is supplied with solid material to be extracted, for example, tobacco from a storage container 60 which feeds the solid material into the extractor 20 through a conveying means 6a. Organic solvent is stored in a storage vessel 70 which discharges into the extractor 20 through conduit 99 which is valve controlled by the valve 7a. The solid material, after extraction, is discharged from the extractor 20 into a drier 80, the material being transported from the extractor 20 into the drier 80 by means of the conveying means 2a. The drier 80, in turn, may optionally be connected through conveying means 8a with a conditioning unit 90 and the latter, in turn, may discharge into a storage container 100 through a transporting means 9a.

The organic solvent, after having passed through the extractor 20, passes through conduit 98 into the liquid-liquid extractor 30 and from there back into the extractor 20 through conduit 97. The organic liquid is forced through its circulatory path by means of pump 111, a flow meter 1a being connected in the conduit 98.

Aqueous medium is pumped by pump 4 through conduit 96 into the liquid-liquid extractor 30 where the aqueous phase flows in counter-current to the organic phase. The aqueous phase exits from the liquid-liquid extractor 30 through conduit 95 and enters the top of ion exchange column 50. A valve 5a is connected in the conduit 95. The aqueous effluent exits from the column 50 through conduit 93 and is again introduced into the liquid-liquid extractor 30 through line 96, the movement of the aqueous phase being caused by the pumping action of the pump 4. Flow meter 4a is connected in the line 93.

The system also includes a regeneration container 13 which is connected to the exit of the column 50 through conduit 92, valves 13a and 5d being connected in the conduit in addition to the pump 13b.

A water container 14 is connected to the conduit 92 by line 91 which latter is valve-controlled by valve 14a. The top of the water container 14 is connected to a solvent recovery unit 16 through line 89. Valve 16b is connected in line 89. The solvent recovery unit 16, in turn, is linked to the drier 80 through line 88 and to the solvent storage container 70 through line 87. Valve 16a is connected in line 87.

The drier 80 is interiorly provided with a spraying means indicated by reference numeral 17, the spraying means being fed by the aqueous phase through pipe 85 which connects the spraying means 17 to conduit 96 of the aqueous circuit. Valve 4b and flow meter 4c are connected in the pipe 85.

The system, moreover, includes a regenerate collecting vessel 12, the bottom of which is communicating with

a regenerate processing unit 15 through line 82, valve 12a being connected in the line 82. The top of the regenerate collector 12 is connected to the top of the ion exchange column through line 79, valve 5b and pump 5c being connected in this line.

The continuously operating solid-liquid extractor 20 may be arranged horizontally or vertically. The liquid-liquid extractor 30 may be constructed in different embodiments. For example, it may be a centrifugal extractor, or it may be a column filled with filling bodies. In respect to the ion exchange column 50, any of the conventional constructions providing for continuous regeneration may be employed. However, it is also feasible to use two or several ion exchange units which are alternately charged and automatically regenerated. In respect to the solvent recovery unit 16, this may comprise a cooler, a wash tower with distillation arrangements, an absorption device or a combination of such units. The specific construction will depend on the individual circumstances. The same applies to the other devices such as the drier 80 which may be a drum drier, a turbulent drier or a belt drier.

Under certain circumstances, it may be advisable to operate in the solid-liquid extractor 20, as well as in the drier 80, under slight negative pressure conditions. This is recommended in order to prevent loss of solvent. It may also be advantageous for this purpose to arrange both units, to wit, the solid-liquid extraction device 20 and the drier 80 in a common closed casing which is maintained under slight vacuum.

The casing should only be open at those areas where the tobacco is introduced and discharged. The vapors which are sucked off from the casing may then be supplied to the solvent recovery system 16. It will be appreciated that the showing of the plant is diagrammatical only and that, of course, other conventional means may be provided such as filters, fans and the like. The conveying means 2a which transports the moist tobacco from the extractor 20 to the drier 80 should preferably be in the form of a screw conveyor.

When the plant of FIG. 2 is to be operated, the tobacco is first conveyed from the storage container 60 into the extractor 20. This may be accomplished, for example, by a suitable shaking device or by pneumatic transportation. The transporting means for this purpose has been indicated by reference numeral 6a. The solvent which is stored in the container 70 flows by gravity through line 99 into the extractor 20. If necessary, a pump may, of course, be employed for this purpose. The solvent which thus is charged with nicotine flows then through line 98 into the liquid-liquid extractor 30. At the same time, the aqueous solution existing through line 93 from the ion exchange column 50 is pumped by pump 4 and through line 96 into the liquid-liquid extractor 30. The liquid phase thus extracts the nicotine from the organic phase in the extractor 30. After phase separation, the pump 111 again forces the organic phase through line 98 back into the solid-liquid extractor 20 for extraction of further amounts of nicotine from the tobacco. The cycle for the organic phase is thus complete. When the tobacco in the extractor 20 has been sufficiently extracted, it is conveyed by the conveying means 2a, to wit, for example a screw conveyor, into the drier 80. The drier 80 is heated and as previously set forth may be operated under vacuum condition. The solvent therefore evaporates and the solvent vapors flow through line 88 into the solvent recovery unit 16. The solvent may be recovered in the unit 16, for example, by cooling, compression, washing or absorption. Recovered solvent is returned to the solvent storage vessel 70 through valve-controlled line 87.

If desired, the tobacco may be conditioned within the drier 80 by spraying a portion of the aqueous phase onto the tobacco. For this purpose, the spraying device 17 is arranged which is fed through line 85 with the aqueous phase, to wit, the effluent discharged from the ion ex-

change column 50. The aqueous phase used for this purpose may be a component of a customary tobacco sauce.

Any water which evaporates together with the solvent is separated in the solvent recovery unit 16, and after phase separation from the solvent, is collected in the water container 14. For this purpose line 89 is provided which connects the water container 14 to the solvent recovery unit 16. The water collecting vessel 14, in turn, is connected through lines 91 and 92 with the ion exchange column 50 and supplemental water may thus be supplied from the water collecting vessel 14 to the ion exchange unit 50 in order to supply additional water to the aqueous phase, particularly if an amount of the aqueous phase has been bled off to the spraying unit 17. The additional water emanating from the water storage container 14 is also used for flushing back the filling in the ion exchanger. In order to prepare the regenerant for the ion exchangers, the water from the container 14 may also be conveyed into the regenerating container 13 as indicated in FIG. 2.

The nicotine-containing regenerate obtained in the regeneration of the ion exchange column 50 is first conveyed into the regenerate collector 12. This is accomplished through line 79 and pump 5c. The processing of the regenerate takes place in the regenerate processing unit 15 which is connected with the regenerate collector 12 through line 82. Water or, after addition of alkalis, water and nicotine are evaporated in the regenerate processing unit 15.

The tobacco which is discharged from the drier 80 may be conveyed through conveying means 8a into the conditioning unit 90 where the tobacco is brought to the desired moisture content and may also be admixed with suitable additives. The tobacco finally reaches, through conveying means 9a, the tobacco storage unit 100 which may consist of barrels, silos, or any other suitable storage device.

The invention will now be described by 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 in the process conditions without in any way affecting the scope and spirit of the invention as recited in the appended claims.

In the examples, the expressions "water" and "aqueous phase" are used. With a view to avoiding misunderstandings, the following should be observed: Both the organic solvent and the water as referred to in the examples are not pure substances but the terms rather refer to phases which have already repeatedly been used for the same purpose and therefore, pursuant to the invention, have become enriched or even essentially saturated with soluble tobacco constituents or have reached a certain solubility equilibrium in respect to these constituents as previously referred to. Generally, it has been observed that if the solvent has been used about ten times for extracting a quantity of tobacco, the desired equilibrium condition is achieved.

Example 1

The test was performed with 7.5 kilograms of Rhodesian tobacco having a nicotine content of 2.9% and a moisture content of about 13%. This tobacco quantity was treated in a solid-liquid extractor with circulating 1,2-dichloroethane. 1000 liters of organic solvent were circulated per hour. The nicotine-containing solvent was conveyed to a liquid-liquid extractor where the organic phase was continuously brought into counter current contact with water. The amount of water forced through the liquid-liquid extractor amounted to 100 liter per hour. The water removed nicotine from the organic phase and was circulated through an ion exchange column. The ion exchange column contained a cation exchanger in pearl form (particle size 0.2 to 1.0 millimeter) of the sulfonated polystyrene type. The specific load on the ion exchanger

was 10 liters per hour water per one liter of ion exchanger.

After a treatment time of 60 minutes at about 25° C., the tobacco was removed from the extractor and was dried together with 14 kilogram of 1,2 dichloroethane which adhered to the tobacco. The heat drying was effected in a closed unit, whereby the evaporating organic solvent was recovered by suitable cooling. 7.0 kilograms of tobacco having a nicotine content of 0.63% were obtained in this manner. The exterior of the tobacco appeared unchanged and the burning characteristics and the smoke aroma of the tobacco were the same as before the treatment.

The ion exchanger was exhausted after the performance of several tests in accordance with the above description. The effluent was tested in order to ascertain when the ion exchanger was spent. It was ascertained that the ion exchanger was capable of absorbing about 250 grams of nicotine per 1 liter of ion exchanger. Regeneration of the ion exchanger was effected with sulphuric acid of 5% concentration and the collected eluates were subsequently concentrated until the nicotine concentration amounted to 40%. The regenerated ion exchanger was then again used for further experiments.

Example II

This example was carried out with 10.0 kilograms of Burley tobacco having a nicotine content of 4.1% and exhibiting a moisture content of 9%. The extraction with the organic phase was carried out with 3000 liters per hour of circulating methylenechloride at room temperature. The organic solvent was brought into extracting contact in a liquid-liquid extractor with 500 liter per hour of water. The water, enriched with nicotine, was circulated through an ion exchange resin column which contained an acid cation exchanger. After a total extraction time of 2 hours, the tobacco was dried in a closed drier together with 29 kilograms of adhering methylenechloride. The drying was effected so that the temperature of the tobacco did not rise above 55° C. Towards the end of the drying procedure, to wit, at a time when about 1 kilogram of organic solvent was still present within the tobacco mass, the tobacco was sprayed with 1.0 liter of the aqueous phase. The aqueous solution was bled from the aqueous circuit of the process directly behind the ion exchange column. The tobacco, after the spraying, was further dried to evaporate the water and residual solvent.

The methylenechloride which exited from the drier in vapor form was condensed by cooling and uncondensable vapors were absorbed on active carbon.

Upon termination of the drying procedure, the tobacco was again moistened by further spraying of a small amount of the aqueous phase to impart the tobacco with its original moisture content of 9%. The yield of tobacco amounted to 9.5 kilogram and the nicotine content of the treated tobacco was 0.45%.

Example III

This experiment was carried out with Burley tobacco which was extracted with 1,1,1 trichloroethane. The extraction was effected in a continuous solid-liquid extractor operating on the screw conveyor principle and 100 kilograms of the Burley were fed through the screw conveyor per hour with 25,000 liter per hour of the 1,1,1 trichloroethane flowing in counter current to the tobacco. The 1,1,1 trichloroethane exiting from the extractor was contacted with 3000 liter per hour of water in a liquid-liquid extractor and was thereafter again recycled to the tobacco. The nicotine-containing water was circulated through an ion exchange column. In this particular instance, a cation exchanger of the sulfonated organic coal or carbon type was used having a particle size of 20 to 50 mesh and a capacity of about 80 grams of nicotine per liter. The temperatures of the organic and the aqueous phases were between 20 and 25° C. and the total extraction time amounted to 90 minutes. The regeneration of the ion ex-

changer was effected after ten tests of the indicated kind with sulphuric acid of 8% concentration. Upon concentration of the combined eluate, a nicotine sulphate solution of 40% concentration, calculated on the nicotine base, was recovered.

Upon termination of the extraction, drying of the tobacco and remoistening of the tobacco to its original moisture content, 97.0 kilograms of tobacco per hour and having a nicotine content of 0.6% were continuously obtained on the average.

Example IV

1.0 kilogram of dried hips are extracted with cyclohexanol at room temperature in a solid-liquid extractor. The organic solvent exiting from the extractor is conveyed into a Raschig column where the organic phase is continuously brought in contact with water flowing in counter current. The organic phase is then again recycled to the hip-containing solid-liquid extractor for further extraction purposes. The aqueous phase is conveyed from the Raschig column to an ion exchange column containing 1 liter of anion exchanger of the aliphatic amine-phenol-formaldehyde condensate-type where ascorbic acid contained in the aqueous phase is removed. The effluent is recycled to the Raschig column. The procedure is briefly interrupted after a treatment time of 5 hours in order to exchange the extracted hips for a fresh amount, whereafter the process is continued. After 15 batches of this kind, the anion exchanger is regenerated with sodium hydroxide solution of 2% concentration and 12.3 grams of ascorbic acid are recovered from the discharged regenerate according to known methods.

As will be appreciated from the Example IV, the inventive procedure is not restricted to the selective extraction of basic constituents from plant material, such as of nicotine from tobacco, but by suitably adapting the procedure it may equally successfully be used for the selective extraction of acidic plant constituents such as ascorbic acid from fruits and leaves. All that is required for extracting acid constituents is the use of an anion exchanger and, if necessary, the aqueous phase can be adjusted to a pH above 7 by the addition of suitable bases.

Concerning the extraction of basic constituents, again the process is not limited to the removal of nicotine from tobacco but other alkaloids may, of course, be obtained in the inventive manner. For example, the process is suitable for extracting quinine from China bark, morphine from opium or atropine from dried leaves of atropine belladonna.

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 process of selectively removing a particular water- and organic solvent-soluble substance of acid and/or basic reaction from solid plant particles containing a plurality of water- and organic solvent-soluble substances which comprises:

- (a) extracting the plant particles with an organic solvent which is essentially insoluble in water, whereby a solution of said substances in said solvent is obtained;
- (b) extracting said particular substance from said solution with water which is essentially saturated with said substances except for said particular substance, whereby an aqueous solution of said particular substance is obtained; and
- (c) removing said particular substance from the aqueous solution by ion exchange.

2. A process as claimed in claim 1, wherein said organic solvent is recycled for further extraction of the plant particles after said particular substance has been extracted from the organic solvent by said water.

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3. A process as claimed in claim 1, wherein said water is recycled for extracting a further amount of said particular substance from said organic solvent after said particular substance has been removed from the water by said ion exchange.

4. A process as claimed in claim 1, wherein said plant particles are tobacco particles and said particular substance is nicotine.

5. A process as claimed in claim 1, wherein the removal of said particular substance from the aqueous solution by ion exchange is effected by passing the aqueous solution through an ion exchange column.

6. A process as claimed in claim 5, wherein said particular substance is of basic reaction and said ion exchange column contains a cation exchanger.

7. A process as claimed in claim 5, wherein said particular substance is of acidic reaction and said ion exchange column contains an anion exchanger.

8. A process as claimed in claim 1, wherein the extraction of said particular substance from said solution with water is effected by contacting said solution and said water in counter current.

9. A process as claimed in claim 1, wherein said plant particles, after the extraction with said organic solvent, are dried with organic solvent adhering thereto whereby said organic solvent evaporates and said water- and organic solvent-soluble substances, except for said particular substance, are restored to the plant particles.

10. A process as claimed in claim 1, wherein a portion of said water, after removal of said particular substance therefrom by ion exchange, is recycled to said plant particles, after said plant particles have been essentially liberated from said organic solvent, and the portion of recycled water is replenished by fresh water.

11. A cyclic process of removing nicotine from tobacco which comprises:

(a) extracting tobacco with an organic solvent essentially insoluble in water and capable of dissolving nicotine and other soluble tobacco constituents, whereby a solution of nicotine and said other constituents in said organic solvent is obtained;

(b) extracting said solution with water essentially devoid of nicotine but being in substantial solubility

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equilibrium with said organic solvent in respect to said other tobacco constituents, whereby an aqueous nicotine solution and said organic solvent enriched with said other constituents are obtained;

(c) recycling said organic solvent to the tobacco for extraction of further amounts of nicotine;

(d) passing said aqueous nicotine solution through a cation exchanger to exchange the nicotine;

(e) recycling the effluent from the cation exchanger to said nicotine-containing organic solvent solution to extract more nicotine therefrom; and

(f) regenerating said cation exchanger for removal and recovery of nicotine therefrom.

12. A process as claimed in claim 11, wherein the tobacco, after a major portion of its nicotine has been extracted by said organic solvent, is dried in the presence of a portion of said organic solvent, whereby the organic solvent evaporates and said other constituents contained in said organic solvent are restored to the tobacco.

13. A process as claimed in claim 12, wherein the amount of said other tobacco constituents in the organic solvent present in the tobacco before drying is equivalent to the amount of said other tobacco constituents removed from said tobacco by the dissolving action of said organic solvent.

14. A process as claimed in claim 11, wherein the tobacco is taken out of the extraction with an amount of adhering solvent by weight at least twice the weight of the tobacco to be extracted.

15. A process as claimed in claim 11, wherein a portion of the effluent of (e) is bled off and recycled to the tobacco, said portion being replenished by adding fresh water to said effluent.

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SAMUEL KOREN, *Primary Examiner*.

D. J. DONOHUE, *Assistant Examiner*.