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(54) Title: A METHOD FOR TREATING GREEN TOBACCO AND THE PRODUCT OBTAINED THEREBY

(57) Abstract

The invention relates to the manufacture of a nicotine-containing crude tobacco material from green tobacco by means of a process in which the tobacco plant material is converted to a pulp which is divided into a solid, coarse fraction and a liquid fraction. Suspended or emulsified fine material is then separated from the liquid fraction, resulting in a leaf nutrient concentrate containing pigment. This concentrate is divided into several pigment fractions with the aid of different extraction processes and these fractions are suitably treated and then optionally returned to the first, solid coarse fraction to enhance the flavour and appearance of the products. Proteins present in the liquid fraction left over at separation of leaf nutrient concentrate are totally removed, whereafter the deproteinized liquid fraction is processed while retaining the nicotine, so as to obtain a concentrated liquid phase that contains nicotine, flavourants and colourants and is then returned to the solid, coarse fraction, optionally together with other additives, so as to obtain the desired crude tobacco material. The invention also relates to the crude tobacco material obtained, intended for smokable and smokeless nicotine-containing products which in use have considerably reduced mutagenicity according to the Ames test and a low TSNA content.

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A Method for Treating Green Tobacco and the Product Obtained Thereby

The present invention relates to a processing method for treating green tobacco plants or parts thereof for obtaining a crude tobacco product. The invention also relates to the product obtained by the method.

Green tobacco plants, i.e. immature tobacco plants, or parts of green tobacco plants are normally not used in the manufacture of crude tobacco material. Irrespective of whether the whole of the tobacco plant is used or whether only the leaves of the tobacco plant are used, these are normally not harvested until yellowing of the leaves or the whole of the plant has begun, i.e. the plants have begun to ripen whereafter the harvested plant material is dried either with air at ambient temperature or with heated air.

The work of cultivating and drying or curing tobacco is highly labour-intensive. The fact that the plants or parts thereof used must have begun to turn yellow before being harvested itself implies a limitation with regard to suitable cultivation climates. If green tobacco could be used in the manufacture of tobacco products, this would shorten the growing time. This would also enable tobacco to be grown on latitudes where the growing season is normally not long enough for the tobacco to yellow or ripen, either completely or partially. A favourable climate would also allow more than one crop to be harvested each season.

In the manufacture of crude tobacco an aspect of ever increasing importance is that in addition to obtaining a crude product in which the flavour is retained or

improved, there is also obtained a product from which some of the allegedly deleterious substances which are included in tobacco or formed in the burning of tobacco are essentially eliminated or reduced significantly. These substances include plant proteins and their derivatives which can form undesirable substances when heated. Smoking products which have low "tar contents" are considered desirable.

The majority of earlier techniques for reducing the concentrations of these substances in smoking products has resulted in a change in the construction of the product, for instance cigarettes have been provided with filters and/or have been wrapped in a porous paper casing. Porous cigarette paper enables air to be drawn into the cigarette through the pores in the paper, so as to dilute the gases that are generated when the tobacco is smoked. Filters, however, are not efficient enough and the use of porous cigarette paper reduces the flavour of the product. As an alternative to a change in the construction of the smoking product, attempts have been made to develop a crude tobacco material in which the concentration of these substances is reduced but the flavour of the tobacco is retained. One method for obtaining such modified tobacco products, i.e. deproteinized tobaccos, is described in US-A-4,289,147. The process described in that U.S. specification, however, results in a nicotine-free product, which is considered a drawback by most consumers, since nicotine-free smoking products have never been successful commercially.

Accordingly, the present invention relates to the manufacture of a crude tobacco material for smokable or smokeless nicotine-containing products of changed composition but with taste and aroma of tobacco. The invention also relates to the product obtained by the pro-

cess.

One object of the invention is to provide a different and improved product containing reduced amounts of those components which may form undesirable substances when burning.

Another object of the invention is to enable whole tobacco plants to be used, since this simplifies the process and improves the yield. Another object of the invention is to enable green, i.e. immature, tobacco, to be used, so as to shorten the tobacco plant cultivation time, thereby enabling tobacco cultivation zones to be extended and several harvests to be obtained during one and the same season.

Still another object of the invention is to enable the separation of such by-products as those which can be either used directly or processed to form valuable products.

A further object of the invention is to enable the manufacture of a crude tobacco material of reduced mutagenicity as defined according to Ames test, for instance, and having a low TSNA (Tobacco Specific Nitrosamines) content.

Other objects of the invention and advantages afforded thereby will be apparent from the following description.

According to the present invention, the aforesaid objects are achieved with a process for treating green tobacco that comprises conversion of the tobacco plant material to a pulp and dividing the pulp into a solid, coarse fraction and a liquid fraction. Suspended or emulsified fine materials in the liquid fraction are

removed therefrom, therewith obtaining a pigment-containing leaf nutrient concentrate which, with the aid of various extraction processes, is divided into a plurality of pigment fractions which subsequent to being appropriately processed are wholly or partly returned to the first coarse solid fraction. The proteins present in the liquid fraction that remains subsequent to separating the leaf nutrition concentrate therefrom are removed either totally or partially, whereafter the deproteinized liquid fraction is processed while retaining the nicotine, so as to obtain a concentrated liquid phase, so-called brown juice, which contains nicotine, flavourants and colourants. This concentrated liquid phase is then returned to the solid, coarse fraction, optionally together with other additives, so as to obtain the desired crude tobacco material.

US-A-4,343,317 describes a process for treating whole green tobacco leaves, by expressing protoplasmic juice from the leaves and artificially curing the pulp obtained. Suitable artificial curing methods are said to be 1) thermal browning by drying at ambient temperature and subsequent heating to a temperature of 190°C for 15 minutes; 2) photobleaching, optionally after pretreating the pulp with steam, alcohol, etc., followed by thermal browning; 3) exposure of the tobacco pulp to SO_2-gas, so as to remove green pigment and flavourants; and 4) soaking pressed tobacco in an acidic medium (pH 1.5-3.5) and incubating the soaked pulp at elevated temperature, preferably at a temperature of about 50°C, until the green colour has disappeared. The expressed juice can be processed to remove different components therefrom, e.g. proteins, and/or to develop the flavourants, and is then returned to the artificially cured pulp. According to this patent specification, one advantage is that whole green tobacco leaves can be used in the process without

disintegrating the leaves. As whole green tobacco leaves are used, only a limited removal of protoplasmic juice from the tobacco leaves is obtained and the final product of the present invention cannot therefore be obtained. A further disadvantage is the difficulty in the processing operations.

The earlier mentioned US-A-4,289,147 describes a process for treating tobacco, including green tobacco, in order to obtain a crude tobacco material from which protein, nicotine and green pigment have been removed. According to this process, the tobacco is converted into a pulp which is divided into a liquid fraction and a solid coarse fraction. The liquid fraction, from which finely particulate material has first been removed and which contains water-soluble plant material, is treated for the removal of proteins and nicotine. Pigment is removed from the finely particulate material and divided into green and non-green pigments. The coarse fibre fraction, which has been decoloured by extraction of green and non-green pigments, is then combined with non-green pigments obtained in earlier stages and the liquid fraction, from which nicotine and protein have been removed.

Even though these two patent specifications describe processes for treating green tobacco, the processes taught thereby have certain drawbacks and do not result in a satisfactory crude tobacco material. Furthermore, the process taught by US-A-4,343,317 is primarily intended for the treatment of whole tobacco leaves and the aforesaid artificial curing methods do not result in a high grade product. As far as is known, the process taught by this patent has not yet been utilized commercially and is probably very difficult to apply on a commercial scale with respect to tobacco products. According to US-A-4,289,147, a product which is essentially

free of nicotine is obtained. The demand for tobacco products which are essentially free of nicotine is, however, almost non-existent. Furthermore, the tobacco material obtained is not of high quality and the method is difficult to carry out on a commercial scale.

On the other hand, according to the present invention the method described in the introduction for treating green tobacco yields a high-grade crude tobacco product which when smoked has strikingly lowered mutagenicity, for instance according to Ames test. The process also gives a low TSNA content in the tobacco product. It is also well suited for commercial use.

The inventive process will now be described in more detail with reference to the accompanying drawing, the single Figure of which is a flow sheet illustrating a suitable embodiment of the inventive process.

The present invention thus relates to a process for treating tobacco plant material so as to obtain crude tobacco material intended for smokable and smoke-free nicotine-containing products which have considerably reduced mutagenicity and a low TSNA content when used, said process comprising

(a) disintegrating green tobacco in the form of whole plants or parts thereof to form a pulp;

(b) separating the pulp into a solid fraction (I) which includes coarse solid material and a liquid fraction (II) that includes water-soluble tobacco plant material and suspended or emulsified fine material;

(c) separating the liquid fraction (II) into a liquid fraction (III) and a fraction (IV) which contains the fine material; and

(d) separating from the liquid fraction (III) proteins in the form of at least one fraction (V), to obtain a

liquid fraction (VI); said process comprising the further steps of

(e) adjusting the liquid fraction (VI) to an appropriate pH-value, and then concentrating said fraction, optionally subsequent to oxidation thereof, to form a fraction (VII) which contains nicotine, colourants and flavourants;

(f) suspending the fraction IV obtained from step (c) in alcohol or some other water-miscible solvent and hydrolyzing the suspension, suitably while adding a base, and then evaporating the solvent partially and extracting the residue with non-polar solvent, whereafter a fraction (VIII) containing yellow-orange pigment is separated from a basic fraction (IX) containing green pigment;

(g) extracting the fraction (IX) with polar, water-immiscible solvent and then separating an extraction solvent fraction (X) containing polar pigment;

(h) precipitating from the aqueous phase (XI) obtained in step (g) modified green pigment (XII) which, after suitable treatment, forms chlorophyll-derived colourants and flavourants (XIII); and

(i) combining the fraction (I) obtained in step (b), optionally after heating and drying said fraction, with the fractions VII and VIII and optionally also with the fraction X and/or the fraction XIII, thereby obtaining a crude tobacco for the smoking product or the smoke-less product.

The various process steps and the numbering of the fractions obtained are illustrated in the accompanying flow sheet.

Green tobacco is used as the starting material when practicing the inventive process. Whole tobacco plants or parts thereof, for instance the leaves, can be used.

Preferably, whole tobacco plants are used, especially when the tobacco has been harvested at an early growth stage. The plant material is disintegrated in step (a) to form a pulp, optionally with the addition of water. Disintegration is effected in a conventional manner, for instance by crushing or grinding the plant material. This disintegration process releases the liquid phase containing water-soluble growth material from the coarse, solid material. The disintegration process can be carried out in the presence of suitable additives, such as antioxidants, for instance reducing agents, to prevent oxidation of the polyphenols present.

The division in step (b) is effected in a conventional manner. The pulp is preferably pressed, so as to separate a press juice or green juice (fraction (II)) from the press cake (fraction (I)).

Fraction (I) is kept for use in the final process step, and is preferably dried with the purpose of preserving the fraction so as to avoid its quality being impaired due to bacterial and/or mould growth during the storage period, for instance.

The green juice (fraction (II)) containing water-soluble plant material and suspended or emulsified fine material may be divided directly in accordance with step (c) into the liquid fraction (III) and the fraction (IV) containing the fine material. The fraction (IV) is normally referred to as leaf nutrient concentrate (LNC).

According to one preferred embodiment of the invention, the green juice (fraction (II)), however, is heated to a temperature of at most 55°C and preferably from 40-45°C prior to step (c). Heating of the juice results in partial coagulation of fine, green-pigment material,

thereby facilitating separation of this material together with other substances constituting LNC in the following separation step (c).

The separation effected in step (c) is carried out with conventional techniques, for instance centrifugation or filtration. In case the aforesaid heat treatment process is carried out, in general moderate centrifugal forces are sufficient to achieve separation.

Proteins are then removed from the liquid fraction (III). It is known that proteins give rise to an impaired flavour (Schmuck index) when smoking, for instance, a cigarette. Mutagenic substances may also form when burning proteins. It is therefore desirable to remove proteins completely or at least partially from the crude tobacco material. This can be achieved by removing proteins in the form of one or more protein fractions. When the proteins are separated in the form of a single fraction (V), this is preferably achieved by heating the liquid fraction (III) to a temperature at which all proteins will coagulate, i.e. at a lowest temperature of 52°C and preferably at a temperature of from 75-85°C. The fraction (V) can then be separated, for instance by centrifugation. Alternatively, the proteins can be precipitated by acidification, suitably to a pH-value of 4.5 or less.

It is preferred that essentially all proteins are precipitated in one single step and are subsequently removed. Processes for carrying this out have been well-known for a long time.

The pH-value of the liquid fraction (VI), which thus contains no proteins or has a reduced protein content,

is then adjusted in step (e) to a suitable value, whereafter certain components present in this fraction are optionally oxidized. If the oxidation process is carried out in an acidic environment, a suitable pH-range is from 3 to 4, and particularly a pH-value of 3, which can be obtained by adding phosphoric acid. Alternatively, other acids can be used, such as citric acid, succinic acid and other organic acids.

Oxidation is effected by adding an oxidant at room temperature or preferably at a higher temperature, e.g. a temperature in the region of 30-100°C, preferably 70-90°C. A suitable oxidant is H_2O_2. Other oxidants that can be used are organic peracids.

The oxidation process results in at least partial oxidation of aromatics to obtain a quinoid-type structure. This corresponds to the oxidation that occurs during conventional curing of tobacco by air-drying the tobacco.

Concentration of the possibly oxidized fraction in a conventional manner, e.g. by evaporation, produces a fraction (VII), normally designated brown juice, which contains nicotine, colourants and flavourants. This brown juice is returned to the fraction (I) in the final process step.

The leaf nutrient concentrate (LNC), i.e. fraction (IV), is processed in accordance with the invention in the steps (f)-(h) to recover valuable products, which can be returned to the fraction (I) and/or used in some other way. As distinct from earlier known techniques, when processed the green pigment also produces a valuable

product (fraction (XIII)).

The fraction (IV) is suspended in step (f) in alcohol, e.g. methanol, ethanol or isopropanol, or in some other water-miscible solvent. This solubilizes the leaf pigments and decolourizes LNC, which becomes suitable as animal feed rich in protein and starch. Since green pigment is insoluble in water, the dissolved pigments are hydrolyzed, e.g. at ambient temperature, by adding a base, for instance an alkali hydroxide, such as potassium hydroxide. The alcohol used is suitably ethanol, e.g. in an amount which gives a solution containing 60-85 % ethanol by volume. Subsequent to partial evaporation of the solvent, e.g. to a 20% ethanol solution, the residue is extracted with a non-polar solvent, e.g. heptane. The isolated fraction (VIII) thus contains yellow-orange pigment dissolved in the non-polar solvent. Extraction is suitably effected at ambient temperature. Preferably, the hydrolysis is also carried out at ambient temperature, even though an elevated temperature may be used, for instance to accelerate the process of hydrolysis.

The aqueous solvent fraction (IX) containing dissolved green pigment is then extracted with a polar, water-immiscible solvent, to separate a polar pigment (fraction (X)). A suitable solvent is ethyl acetate.

By "water-immiscible" solvent, it is meant that the solvent is essentially immiscible with water. Solvents which are included in this term may thus be miscible with water to a certain extent, namely up to about 10%.

The polar pigment is comprised of xanthophyll.

This can be returned to the fraction (I) or used for

other purposes, such as animals feed additives and food colourants.

A modified green pigment is then precipitated from the remaining aqueous fraction (XII) comprising green pigment, with the aid of some suitable precipitating agent, such as iron compounds including ferric nitrate, ferric citrate, ferric acetate, ferric formate, ferric oxide and corresponding ferrous compounds under acidic conditions.

This precipitation may cause the green colour to convert to another colour, e.g. black when the precipitation process is effected with an iron compound. This modified "green pigment" is then further processed, whereby chlorophyll-derived colourants and flavourants (fraction (XIII)) are obtained. This processing of the modified "green pigment" involves oxidation and esterification. Initially the black precipitate is solubilized in acetic acid to which H_2O_2 is added, and the mixture is refluxed at 108-110°C for 30 minutes, and is then cooled. To the same mixture, propanol is added for esterification in the presence of a small amount of sulfuric acid. The resulting solution contains chlorophyll derivatives with a reddish brown colour and is neutralized to a pH of 6 to 7 with a base, such as potassium hydroxide.

The final step of the inventive method involves combining the fraction (I) with the brown juice (fraction (VII)) and optionally also with other fractions obtained in previous method steps. For example, it can be achieved by spraying the brown juice and/or other liquid fractions over the fraction (I) while mixing said fraction and said juice and/or other liquid fractions together, e.g. by rotation. An elevated temperature of 80-100°C is preferably used. Non-liquid fractions which are

to be returned to the fraction (I) are dissolved in an appropriate solvent, for instance an alcohol, such as ethanol, and then sprayed over the fraction (I), as described above.

The following Examples illustrate a number of suitable embodiments of the invention. It will be understood, however, that the invention is not limited to these particular embodiments.

Example 1

5500 kg of young, green tobacco consisting of whole plants of which less than 25% had flowered were harvested. The plants were chopped into pieces of about 30 mm in length, whereafter they were disintegrated in a mill while adding water in an amount corresponding to 0.5 weight equivalents and containing 1% by weight sodium metabisulphite. The resultant suspension was pressed in a screw press so as to divide the suspension into a solid fraction (fraction I) and a liquid phase, i.e. green juice, (fraction II).

The solid phase (fraction I) was washed with water, and diluted with three to four times as much water while stirring, and then was again passed through a screw press so as to obtain a solid phase and a liquid phase, this latter phase being discarded. The solid phase was fluffed, and optionally disintegrated, in a refiner, prior to being dried to 90% dry solids with heat, in a conventional manner.

The resultant solid material, i.e. the treated fraction (I), can be used as it is. In the present case, however, the fraction (I) was divided into two further fractions, namely a fibrous, long fraction, called Fibre A in the

table below, (120 kg), and a small tobacco fraction (110 kg). The former fraction was used as a reference material for evaluating smoking properties. The results from these evaluations are set forth in Table I below.

Green juice (fraction II) from the first pressing process was adjusted to a pH of 5.15 with phosphoric acid and heated to 45°C. This temperature was maintained for about ten minutes, whereafter the precipitated fine material, the leaf nutrient concentrate (fraction IV) (240 kg, 28% dry solids), was separated by decanting from the aqueous phase (III), also designated yellow juice.

Carbon dioxide (g) was added to the resultant aqueous phase until saturation was reached, the F1-proteins precipitating or crystallizing and being separated from the juice by centrifugation or filtration.

The remaining, soluble proteins were coagulated, by adjusting the pH of the juice to 3.5 with phosphoric acid (the juice was allowed to stand for about one hour and the F2-proteins were then separated from the liquid phase (fraction VI), i.e. the brown juice, by centrifugation or filtration.

The pH-value of the brown juice was adjusted to 3 by adding phosphoric acid, and 30-percent hydrogen peroxide was added (200:1, based on volume), whereafter the brown juice was heated at 80°C over night. The brown juice was concentrated to a dry solids content of 50% (fraction VII), and could then be stored for later use. This concentrated brown juice (fraction VII) is designated BJ A in the following.

Subsequent to diluting the concentrated brown juice

BJ A with water, the juice was added to the long, fibrous material, Fibre A, obtained above, in a proportion of 4 kg dry solids brown juice to 10 kg dry solids of solid material, whereafter the material obtained was dried to 14-18% dry solids and filterless cigarettes were manufactured in a conventional cigarette machine. Cigarettes were also manufactured from the fibrous, long material, Fibre A, without adding BJ A.

The results obtained with tests carried out on the cigarettes and also results of comparison tests carried out on a retailed conventional, filterless cigarette of "American blend" type are set forth in Table I below.

Example 2

2900 kg of green, flowering tobacco were harvested, chopped, disintegrated and pressed in the manner described in Example 1. The solid material (fraction I) was washed, pressed, fluffed, dried and sieved thereafter in the same manner as that described in Example 1, resulting in 100 kg of the fibrous, long fraction, designated Fibre B, and 90 kg of a small tobacco fraction. The green juice (fraction II) from the first pressing process was adjusted to a pH of 3.5, by adding phosphoric acid, and was then heated to a temperature of 75°C, this temperature being maintained for about fifteen minutes, whereafter the precipitated fine material (175 kg, 28% dry solids) (fraction (IV) + (V)) was separated by decanting said material from the aqueous phase (the brown juice) (fraction (VI)). The brown juice (fraction (VI)) was adjusted to a pH-value of 3 with phosphoric acid, and 30-percent hydrogen peroxide was added (200:1, based on volume), whereafter the brown juice was heated to a temperature of 80°C, this temperature being maintained over night. The brown juice was

then concentrated to 80% dry solids and could then be stored for later use. This concentrated brown juice (fraction (VII)) is designated BJ B in the following. Subsequent to being diluted with water, the concentrated brown juice, BJ B, was added to the long fibrous material, Fibre A, obtained from Example 1, in a proportion of 4 kg dry solids brown juice to 10 kg dry solids of solid material, whereafter the material obtained was dried to 14-18% dry solids and filterless cigarettes were manufactured in a conventional cigarette machine. Cigarettes were also manufactured from the fibrous, long material obtained above from the fully grown plants (Fibre B).

The results obtained with tests carried out on the cigarettes are also set forth in Table 1.

Table I

AMES TEST ON SMOKE CONDENSATES FROM MACHINE-SMOKED CIGARETTES

Cigarette	Tar (mg/ct)	Ames (TA98+S9, rev/mg tar)
Fibre A	18.2	553
Fibre B	16.6	422
Fibre A+BJ A	18.7	524
Fibre A+BJ B	19.4	629
"American blend" type	22.0	1698

It is evident from the above Table that the products manufactured in accordance with the invention have a reduced tar content and a considerably lower mutagenicity than a conventional cigarette ("American blend") when used.

Claims

1. A process for treating tobacco plant material so as to obtain crude tobacco material intended for smokable and smokeless nicotine-containing products which have considerably reduced mutagenicity according to the Ames test and a low TSNA content when used, said process comprising

(a) disintegrating green tobacco in the form of whole plants or parts thereof to form a pulp;

(b) separating the pulp into a solid fraction (I) which includes coarse solid material and a liquid fraction (II) that includes water-soluble tobacco plant material and suspended or emulsified fine material;

(c) separating the liquid fraction (II) into a liquid fraction (III) and a fraction (IV) which contains the fine material; and

(d) separating from the liquid fraction (III) proteins in the form of at least one fraction (V), to obtain a liquid fraction (VI); c h a r a c t e r i z e d by the further steps of:

(e) adjusting the liquid fraction (VI) to an appropriate pH-value, and then concentrating said fraction, optionally subsequent to oxidation thereof, to form a fraction (VII) which contains nicotine, colourants and flavourants;

(f) suspending the fraction IV obtained from step (c) in alcohol or some other water-miscible solvent and hydrolyzing the extract, suitably using a base, and then evaporating the solvent partially and extracting the concentrate with a non-polar solvent, whereafter a fraction (VIII) containing yellow-orange pigment is separated from a basic fraction (IX) containing green pigment;

(g) extracting the fraction (IX) with polar, water-immiscible solvent and then separating an extraction

solvent fraction (X) containing polar pigment;
(h) precipitating from the aqueous phase (XI) obtained in step (g) modified green pigment (XII) which, after suitable treatment, forms chlorophyll-derived colourants and flavourants (XIII); and
(i) combining the fraction (I) obtained in step (b), optionally after heating and drying said fraction, with the fractions VII and VIII and optionally also with the fraction X and/or the fraction XIII, thereby obtaining a crude tobacco for the smoking product or the smoke-free product.

2. A process according to Claim 1, c h a r a c - t e r i z e d by heating the liquid fraction (II) to a temperature of at most 47°C prior to (c).

3. A process according to Claim 1 or 2, c h a r a c - t e r i z e d by carrying out the oxidation process in step (e) at a temperature of 20-100°C using the oxidant H_2O_2.

4. A process according to Claim 1, 2 or 3, c h a r - a c t e r i z e d by using ethanol as the alcohol in step (f).

5. A process according to any one of the preceding Claims, c h a r a c t e r i z e d by using potassium hydroxide as the base in step (f).

6. A process according to any one of the preceding Claims, c h a r a c t e r i z e d by using heptane as the non-polar solvent in step (f).

7. A process according to any one of the preceding Claims, c h a r a c t e r i z e d by extracting with ethyl acetate or propyl acetate in step (g) or ethers,

such as diethyl, dipropyl and diisopropyl ethers.

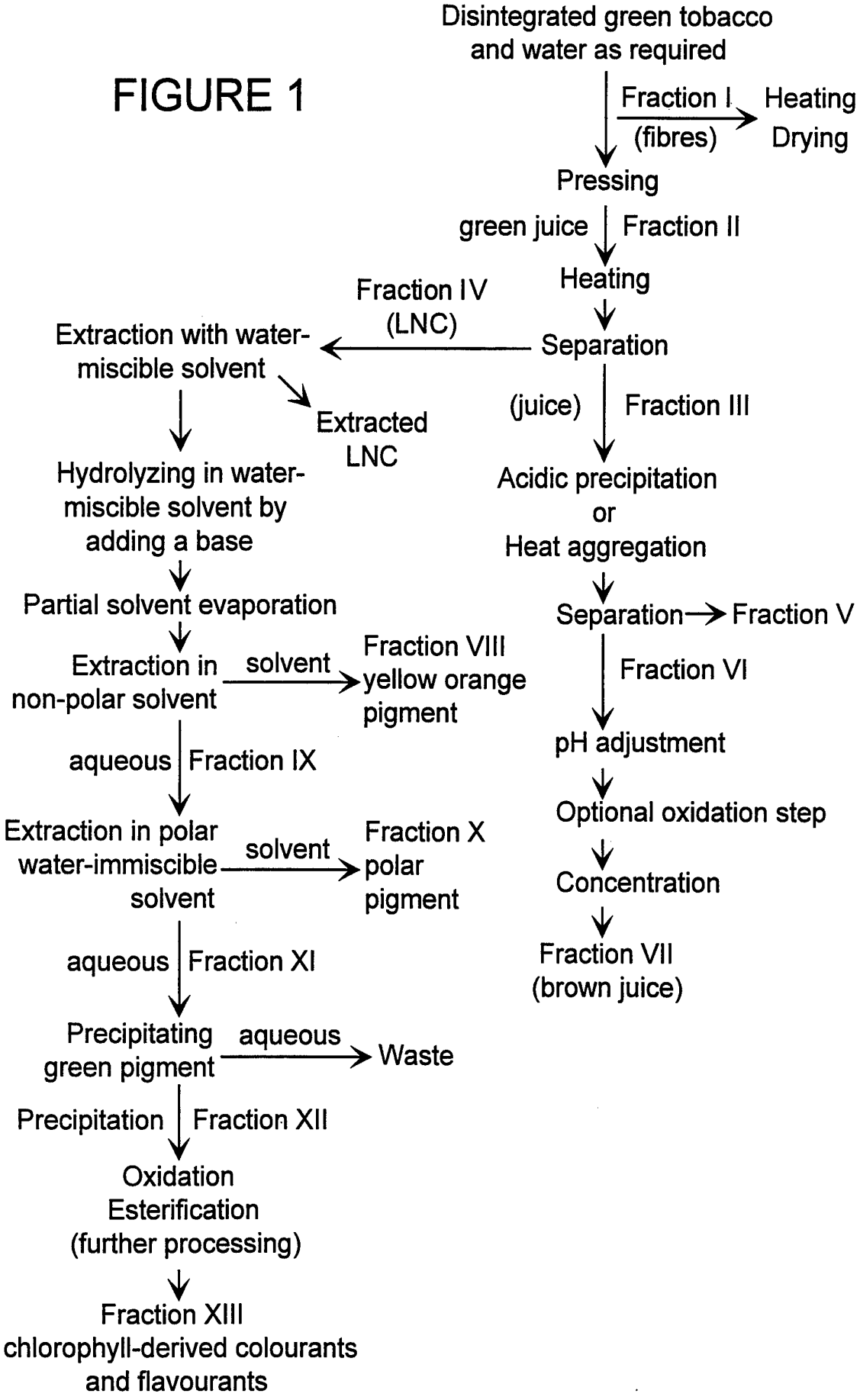
8. A process according to any one of the preceding claims, c h a r a c t e r i z e d by precipitating modified green pigment with iron at pH 6 or lower in step (h), the iron being in the form of ferric or ferrous compounds.

9. A process according to any one of the preceding claims, c h a r a c t e r i z e d by oxidizing the iron-green pigment precipitate in acetic acid in step (h) at a temperature of 108-110°C, using H_2O_2 as the oxidant.

10. A process according to any one of the preceding claims, c h a r a c t e r i z e d by esterifying the oxidized iron-green pigment in propanol in step (h) at a temperature of 80-110°C.

11. A product, c h a r a c t e r i z e d in that said product is comprised of a crude tobacco material for smokable and smokeless, nicotine-containing products which in use have considerably reduced mutagenicity and are produced by the process according to Claim 1.

FIGURE 1


Disintegrated green tobacco and water as required
Fraction I (fibres)
Heating Drying
Pressing
green juice
Fraction II
Heating
Fraction IV (LNC)
Extraction with water-miscible solvent
Separation
Extracted LNC
(juice)
Fraction III
Hydrolyzing in water-miscible solvent by adding a base
Acidic precipitation or Heat aggregation
Partial solvent evaporation
Separation
Fraction V
Extraction in non-polar solvent
solvent
Fraction VIII yellow orange pigment
Fraction VI
aqueous
Fraction IX
pH adjustment
Optional oxidation step
Extraction in polar water-immiscible solvent
solvent
Fraction X polar pigment
Concentration
aqueous
Fraction XI
Fraction VII (brown juice)
Precipitating green pigment
aqueous
Waste
Precipitation
Fraction XII
Oxidation Esterification (further processing)
Fraction XIII chlorophyll-derived colourants and flavourants

INTERNATIONAL SEARCH REPORT

International application No.
PCT/SE 93/00764

A. CLASSIFICATION OF SUBJECT MATTER

IPC5: A24B 15/24
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC5: A24B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

CA, PTS PROMT, TRADE AND INDUSTRY INDEX, WPI, CLAIMS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US, A, 4289147 (WILDMAN ET AL), 15 Sept 1981 (15.09.81) --	1-11
A	US, A, 4343317 (BOKELMAN), 10 August 1982 (10.08.82) -- --------	1-11

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:
"A" document defining the general state of the art which is not considered to be of particular relevance
"E" erlier document but published on or after the international filing date
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)
"O" document referring to an oral disclosure, use, exhibition or other means
"P" document published prior to the international filing date but later than the priority date claimed
"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
30 December 1993	11 -01- 1994
Name and mailing address of the ISA/ Swedish Patent Office Box 5055, S-102 42 STOCKHOLM Facsimile No. +46 8 666 02 86	Authorized officer Carolina Gomez Lagerlöf Telephone No. +46 8 782 25 00

INTERNATIONAL SEARCH REPORT

Information on patent family members

27/11/93

International application No.
PCT/SE 93/00764

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US-A- 4289147	15/09/81	NONE	
US-A- 4343317	10/08/82	NONE	