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- (71) **Applicants (for all designated States except US):** **BRITISH AMERICAN TOBACCO (INVESTMENTS) LIMITED** [GB/GB]; Globe House, 1 Water Street, London WC2R 3LA (GB). **IMPERIAL TOBACCO CANADA LIMITED** [CA/CA]; 3711 Saint-Antoine Street, Montréal, Québec H4C 3P6 (CA).
- (72) **Inventors; and**
- (75) **Inventors/Applicants (for US only):** **HU, Jin** [CN/GB]; British American Tobacco (Investments) Limited, Globe House, 1 Water Street, London WC2R 3LA (GB). **DE GRANDPRÉ, Yves** [CA/CA]; Imperial Tobacco Canada

Limited, 3711 Saint-Antoine Street, Montréal, Québec H4C 3P6 (CA). **JOHN, Edward Dennis** [GB/GB]; British American Tobacco (Investments) Limited, Globe House, 1 Water Street, London WC2R 3LA (GB). **HARRIS, Alan** [GB/GB]; British American Tobacco (Investments) Limited, Globe House, 1 Water Street, London WC2R 3LA (GB). **THORN, Garrick** [NZ/GB]; British American Tobacco (Investments) Limited, Globe House, 1 Water Street, London WC2R 3LA (GB).

- (74) **Agents:** **HEARE, Tanya** et al.; 200 Aldersgate, London EC1A 4HD (GB).

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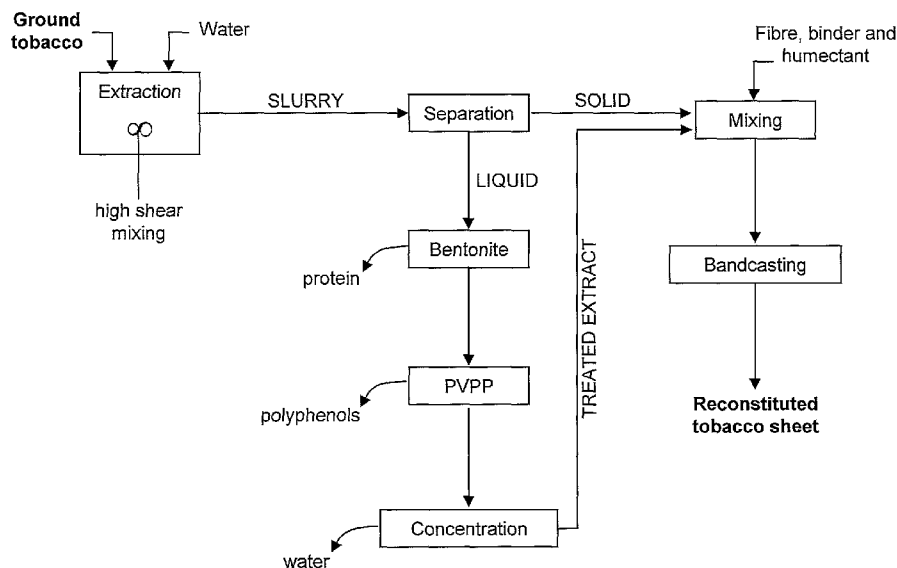


Figure 1

- (57) **Abstract:** This invention provides a method for removing certain constituents from tobacco which includes a step in which ground tobacco is contacted with a solvent and subjected to an intensive mechanical action to generate a tobacco extract and an insoluble tobacco portion. The tobacco extract and insoluble tobacco portion may be separated, further treated to remove selected constituents and recombined. The product may be processed into a reconstituted tobacco sheet.



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Tobacco Treatment

Field of the Invention

The present invention relates to the treatment of tobacco and, in particular, to the
5 extraction of selected constituents from tobacco, for example, in order to reduce certain smoke components.

Description

Tobacco material may be treated and processed to produce a modified blend which, when combusted, gives rise to smoke in which specific constituents of the smoke
10 are selectively reduced or removed compared to untreated tobacco. The sugars and nicotine content of the tobacco, however, preferably remains substantially unaffected by this process, so that the taste and smoking properties of the tobacco are maintained.

15 Methods for removing or at least reducing selected constituents from tobacco are known in the art. Such methods may comprise an extraction step in which tobacco material is extracted with an aqueous or organic solvent, a separation step in which the extracted solution is separated from the insoluble tobacco residue by filtration or the like and a treatment step in which the extracted solution is treated to remove
20 specific constituents. The treated tobacco extract is then recombined with the extracted tobacco to ensure that certain constituents of tobacco are retained in the regenerated tobacco material.

Various extraction and treatment steps have been described in the prior art,
25 depending upon the particular constituent(s) to be removed.

For example, proteins have traditionally been removed by extracting tobacco with an aqueous solvent, optionally comprising a surfactant and/or a proteolytic enzyme. The aqueous extract (comprising solubilised tobacco components) is subsequently
30 separated from the insoluble tobacco residue and treated to remove polypeptides using, for example, an insoluble coagulant such as bentonite. Other nitrogenous compounds, such as nitrates and nitrosamines, have typically been removed from

aqueous tobacco extracts using similar methods. The use of foam fractionation to remove polypeptides from tobacco extracts has also been described.

5 Methods for removing phenolic compounds from tobacco have been described in the art. For example, it is known to extract tobacco with an aqueous or organic solvent, separate the extract from the insoluble tobacco residue and treat the extract with a phenol oxidising enzyme or an adsorbent such as polyvinylpolypyrrolidone (PVPP, which selectively removes polyphenols).

10 However, although processes such as those noted above have been successful in reducing selected constituents from tobacco, they are not without their problems.

For example, it has been found to be relatively easy to remove proteins from uncured tobacco leaf, but the curing of those proteins actually contributes to the
15 flavour of the smoke when the tobacco is combusted. It would consequently be beneficial for such proteins to be removed only after curing; this, however, has proven difficult in the past. Indeed, curing makes proteins more difficult to extract than those proteins present in uncured tobacco.

20 Many known processes also employ enzymes to break down protein; however, enzymes are expensive, their activity depends upon specific reaction conditions and it can prove challenging to remove the produced amino acids, and indeed the enzyme, from the treated tobacco once the reaction has ended.

25 Other reagents used in the treatment of tobacco, such as alkaline solutions, can also have adverse effects on the physical structure of the tobacco, which renders the produced tobacco less amenable to cigarette manufacture and visually unappealing to the end user.

30 The present invention seeks to provide a method that overcomes or alleviates problems such as these. It is also clearly desirable to continue to provide new, alternative methods for modifying tobacco content which results in a tobacco material having suitable physical characteristics for processing into smoking articles.

It would also be advantageous to improve upon the efficiency of known extraction processes.

Statements of the Invention

5 According to a first aspect of the present invention, a process for the treatment of tobacco is provided, comprising a step in which ground tobacco is contacted with a solvent and subjected to an intensive mechanical action to generate a tobacco extract and an insoluble tobacco portion.

10 The process may further comprise, as a preceding step, preparation of ground tobacco in a form of tobacco particles having a mean diameter of less than 500 μm , preferably less than 250 μm , and more preferably less than 50 μm . The ground tobacco may be prepared from cured tobacco.

15 In an embodiment, the solvent is an aqueous solvent. The aqueous solvent may be water.

In an embodiment, the intensive mechanical action is homogenisation, high shear mixing or blending.

20

The process may further comprise separating the tobacco extract from the insoluble tobacco portion. In an embodiment, the tobacco extract is separated from the insoluble tobacco portion using a vibrating sieve filtration system.

25 The tobacco extract may be treated to remove protein therefrom, for example, by treating the tobacco extract with bentonite. The tobacco extract may be treated to remove polyphenols therefrom, for example, by treating the tobacco extract with PVPP. The tobacco extract may be concentrated.

30 In an embodiment, the tobacco extract and the insoluble tobacco portion are recombined.

In an embodiment, the insoluble tobacco portion is processed into a reconstituted tobacco sheet. The processing of the insoluble tobacco portion into a reconstituted tobacco sheet may comprise drying and milling the insoluble tobacco portion to a particle size suitable for making a reconstituted tobacco sheet, optionally adding
5 fibre, a binding agent and/or a humectant to the insoluble tobacco portion, and/or bandcasting the insoluble tobacco portion into a reconstituted tobacco sheet. In any of these embodiments, the process for the treatment of tobacco may further comprise shredding the reconstituted tobacco sheet to produce cut strands of a width suitable for use in a smoking article.

10

According to a second aspect of the present invention, tobacco or reconstituted tobacco obtainable by a process according to the first aspect is provided.

According to a third aspect of the present invention, a smoking article comprising
15 tobacco or reconstituted tobacco according to the second aspect is provided.

Drawings

Embodiments of the invention are described, by way of example only, with reference to the accompanying drawing in which:

20

Figure 1 illustrates a suitable operational sequence for the extraction of tobacco and processing of the extracted tobacco into a reconstituted tobacco sheet.

Figure 2 illustrates the % reduction in nitrogen as a function of time for finely
25 ground tobacco extracted using a homogeniser and a variety of solvents.

Figure 3 illustrates the % nitrogen of extracted cut tobacco as a function of the concentration of solvent (sodium dodecylsulphate (SDS)) used for the extraction.

30 Figure 4 illustrates a process flow diagram of the experimental setup for trials investigating the use of a vibrating sieve to separate extracted tobacco solids from a tobacco extract.

Figure 5 illustrates the results of the vibrating sieve trials. The graph shows the concentration of both total nitrogen and protein nitrogen for ground tobacco feedstock (dried dust) and of the ground dust following extraction of the ground tobacco and separation of the tobacco extract from the tobacco solids.

5

Figure 6 further illustrates the results of the vibrating sieve trials. The graph shows the percent reduction in nitrogen in extracted and separated ground tobacco samples compared to ground tobacco feedstock.

10 Detailed Description of the Invention

The invention provides a process for preparing a tobacco product, which process comprises the step of extracting tobacco with a solvent to remove selected constituents, and particularly protein, therefrom.

15 The tobacco starting material is ground tobacco. It is believed that the use of ground tobacco facilitates protein removal during extraction, so avoiding the need for any proteolytic enzymes. Indeed, the high surface area of the tobacco particles is expected to yield improved extraction efficiencies compared to non-ground tobacco. The ground tobacco may be prepared from any part(s) of the tobacco
20 plant, such as stems, veins, whole leaf and part thereof (such as the lamina portion of the leaf). Preferably it is prepared from raw leaf. The source of the tobacco starting material may be any tobacco species from which it is desired to make a tobacco product. Of particular interest is tobacco from the species *Nicotiana tabacum*. In a preferred embodiment, the ground tobacco is prepared from cured
25 tobacco (such as flue-cured tobacco). Any tobacco or blend of tobaccos can be used.

The ground tobacco is preferably prepared by grinding, milling or the like.

Processing should be carried out with sufficient force to produce small tobacco
30 particles, for example, having a mean diameter of less than about 2 mm, preferably less than about 1 mm, 500 μm , 400 μm , 300 μm , 250 μm , 200 μm , 150 μm or 100 μm , and most preferably less than about 75 or 50 μm . In a preferred embodiment, 80% of the particles are smaller than about 60 μm , preferably smaller than about 55

µm and most preferably smaller than about 53 µm. It is hypothesised that the high surface area to mass ratio of such small tobacco particles yields high extraction efficiencies. Suitable equipment for reducing particle size will be apparent to those skilled in the art.

5

The ground tobacco may be mixed with a solvent for extraction to form slurry. The solvent may be added to the tobacco in a ratio of between 10:1 and 50:1, preferably between 10:1 and 30:1 and most preferably between 15:1 and 20:1 by weight. In a particularly preferred embodiment, the solvent is added to the tobacco in a ratio of
10 18:1 by weight.

15

The solvent may be an aqueous, organic solution, including but not limited to alcohols such as ethanol and inorganic solvents including liquid or supercritical carbon dioxide. Preferably, however, it is an aqueous solution and, most preferably,
15 it is water without any additives, such as de-ionised water.

20

At the very start of the extraction process, the solvent is usually water, but it can also contain alcohols such as ethanol or methanol, metal salts such as potassium hydroxide, sodium chloride or magnesium chloride or it can contain a surfactant,
20 such as SDS. Suitable concentrations of such additives range from 0-20% (v/v), preferably 0-15% (v/v), and more preferably 0-10% (v/v). Exemplary concentrations for the additives identified above are 1% or 10% (v/v) aqueous potassium hydroxide, 0.5% (v/v) aqueous sodium chloride, 0.5% (v/v) aqueous magnesium chloride or 1%, 1.5% or 2% aqueous SDS.

25

The extraction may be a two-step process, featuring a pre-extraction step (with organic solvent, for example) followed by extraction with one of the solvents identified above (such as aqueous potassium hydroxide). Other solvents could be used, depending on the particular constituents to be extracted from the tobacco.

30

The tobacco and solvent mixture, or slurry, may first be formed in a tobacco mix tank before being pumped into a second tank, such as a plug flow reactor or a continuous stirred tank reactor, for extraction to be performed.

In the methods described herein, the mixture or slurry is subjected to an intensive mechanical action, such as blending, homogenising or high shear mixing, during extraction.

5 “Intensive mechanical action”, as used herein, can mean the use of a mechanical force sufficient to apply a velocity or force to the particles of ground tobacco to improve mass transfer and/or to physically alter the particles of ground tobacco. Mass transfer is dependent on system hydrodynamic. Through turbulent action, the laminar layer that surrounds particles is reduced, thus improving the dissolution of
10 constituents. The action can lead to a reduction in nicotine content in the solid of 50-90%. For example, a homogeniser may be used in the intensive mechanical action step, which combines mechanical shearing with cavitation and, to a small degree, sonic energy, so ensuring fast breakdown of material. The destructive action of the homogeniser is based on two inter-related forces, namely, direct
15 mechanical action and cavitation. The latter is the formation of partial vacuums in a liquid by a swiftly moving solid body and the resulting breakdown of substances in that liquid when those vacuums cease to exist. In another example, a high shear mixer may be used to create flow with different velocities in adjacent areas, exerting shear forces on the fluid.

20

The selection of appropriate conditions and equipment to provide the required degree of force will be within the ability of the skilled person.

It is hypothesised that vigorous extraction procedures are advantageous in order to
25 extract the proteins present from the cured tobacco. The continual agitation of the tobacco and solvent mixture, or slurry, during extraction also ensures that the tobacco remains in suspension, which assists the extraction process.

The aqueous extraction may be performed at 15-85 °C, but is preferably performed
30 at 50-70 °C and most preferably at about 60 °C.

Extraction should be performed for between 10 minutes and two hours. In a preferred embodiment, extraction is performed for approximately 40 minutes.

During extraction, extractable tobacco components are removed from the tobacco material and enter solution. These include nicotine, sugars, some proteins, amino acids, pectins, polyphenols, salts and flavours. Up to about 55% of the initial tobacco weight may become solubilised. The use of intensive mechanical action
5 during extraction is believed to improve protein removal from the ground tobacco.

Following extraction, the mixture or slurry may be fed from the extraction tank into equipment suitable for separating the bulk of the tobacco solids from the tobacco extract. As used herein, "tobacco solids" can mean the solid fraction or insoluble
10 tobacco portion that remains after the extraction step, i.e. that which can be physically separated from the tobacco extract, which is in liquid form. Without being bound by theory, extracted protein may be in a suspended or a solubilised format. Preferably, protein particles or molecules in the solvent are also separated from the tobacco solids in this step. Separation is an important step in being able
15 to utilise the extracted material. It is also believed that the efficiency of protein removal may depend upon the nature of the separation step, as preferably the protein fraction needs to remain in the extract in order that it can be removed.

Separation may be achieved by filtration, the tobacco solids being retained by the
20 filter medium and the soluble tobacco extract and much finer particles (such as suspended protein particles) passing there through. Suitable filters will be known to those skilled in the art. A common problem, however, is the filter becoming blocked with solids during separation. Thus, in a preferred embodiment, a vibrating sieve is used to separate the tobacco mixture or slurry. Suitable equipment will be
25 known to those skilled in the art. If a vibrating sieve is used, it should be fitted with mesh of a size suitable for separating the aqueous solution or liquid fraction, containing suspended fine particles of tobacco, from the larger tobacco dust particles. For example, the mesh size may be from about 10 μm to about 50 μm and preferably from about 20 μm to about 40 μm . The mesh size may be about 15,
30 20, 25, 30, 35, 40 or 45 μm . Most preferably, it is about 40 μm . Alternatively, other types of sieving, liquid extraction or foam fractionation may be used for the separation step.

The separated tobacco extract (for example, the filtrate) should be collected and treated further, if desired. Suitable treatment steps are discussed further below.

Meanwhile, the bulk tobacco solids may be further extracted by washing, preferably
5 with a fresh batch of the same solvent as used in the extraction step (such as water),
so that as many soluble constituents as possible are removed from the tobacco.
Although the majority of tobacco proteins are not soluble, they are much smaller
than the extracted tobacco particles and will be removed from the latter as fine
particulates during the washing process. If any additives were used in the extraction
10 process, or a solvent pre-wash was used, these must also be removed from the
tobacco solids after the extraction process with water.

The collection and upstream reapplication of washings to incoming tobacco for
extraction may be repeated a number of times, preferably three, four or even five
15 times. The extraction process may thus be a continual process in which fresh
tobacco is extracted using recycled washings. In this embodiment, only at start-up
of the extraction process is tobacco extracted with fresh solvent.

As the extraction process continues, the extract thus becomes more concentrated in
20 soluble tobacco constituents and protein particles. These constituents include those
that entered solution during primary extraction in the extraction tank (forming the
tobacco extract), as well as those that entered solution during secondary extraction
during washing (forming the washings).

25 The final extract thus comprises both the original tobacco extract and any washings.
The tobacco residue that results after washing is hence devoid of those constituents
that are soluble in the solvent used for extraction or are fine enough to be removed
as fine particulates during the extraction and/or washing process.

30 Depending on the downstream process, the washed tobacco solids can be
dewatered. This increases the capacity of the tobacco solids to reabsorb the
tobacco extract, if and when it is reapplied to the solids. For example, the extracted
tobacco may be squeezed at the end of washing, so as to remove any excess liquid

from it. The extracted tobacco emanating from the washing step is thus typically in the form of a dewatered mat.

Liquid removed in the dewatering process may be recycled and used as the solvent
5 in tobacco extraction, as described above for the washings, to maximise yield of the selected constituents to be removed from the tobacco (such as sugars, nicotine and polyphenols). This would particularly be the case if a washing step was not performed.

10 The tobacco, having been extracted in a solvent, such as an aqueous solution or water, as discussed above, may be further extracted to remove one or more further constituents before being recombined with the concentrated tobacco extract. Any suitable methods, including the use of enzymes, could be employed in addition to the foregoing, to remove further protein from the residual tobacco particulate
15 phase. Suitable methods are disclosed in European Patent Nos. 1094724, 0619708 and 0862865.

It is preferred, however, that the process does not make use of any enzymes. Rather, protein and/or other constituents should be removed from ground tobacco
20 without use of an enzyme. The removal of protein from tobacco without enzyme treatment would be advantageous for the smoking industry, for the reasons already given.

It is believed that a process as described herein, in which ground tobacco is
25 subjected to intensive mechanical action under the described extraction conditions, enables sufficient protein to be removed from the tobacco without the need for any enzymes.

As should be clear from the foregoing, the described extraction and treatment
30 processes may comprise a series of separate steps. They may form a continuous process in which fresh tobacco is continually being fed into the extraction process and processed tobacco is continually being produced downstream.

The treated tobacco may then be further processed rendering it suitable for later recombination with the tobacco extract.

Meanwhile, the final liquid fraction, containing the original tobacco extract, any
5 liquid removed in a dewatering process, hereinafter referred to as 'the tobacco
extract', may be subsequently processed to remove those constituents not desired in
the final tobacco product. Undesirable constituents may include proteins,
polypeptides, amino acids, polyphenols, nitrates, amines, nitrosamines, wax and
pigment compounds. The levels of certain constituents such as sugar and nicotine
10 may, however, remain unaffected so that the flavour and smoking properties of the
extracted tobacco are comparable to those of the original material.

Thus, in a preferred embodiment, the proteins, polypeptides and/or amino acids are
removed from the extracted components in the tobacco extract. Other extracted
15 tobacco components can hence be recuperated in order to add them back to the
extracted tobacco residue in a downstream processing step. This reduces weight
losses from the tobacco, while ensuring that components like sugar, flavours and
nicotine are retained.

20 In other embodiments, proteins, polypeptides and/or amino acids are removed
from the extracted components in the tobacco extract in addition to other selected
constituents, such as polyphenols.

Up to 90% of the extracted proteins may be removed using an insoluble coagulant
25 such as bentonite. This process can be performed as a batch or continuous process.

The tobacco extract is preferably treated with bentonite, to facilitate removal of
polypeptides therefrom. Bentonite may be added to the extract in an amount of 1-
4% of the weight of tobacco initially extracted.

30

Following bentonite treatment, the tobacco extract may be purified from the slurry
by centrifugation and/or filtration.

If an additive was included as an aid to extraction, it should also be removed from the tobacco extract. Suitable methods include precipitation and membrane filtration. For example, SDS may be removed from the tobacco extract by reducing the temperature of the extract below the Krafft point (20 °C for SDS in water) or by
5 adding calcium chloride, both of which cause the SDS to precipitate out of solution. The additive, such as SDS, may be removed from the tobacco extract prior to removal of any protein. For example, the sequential addition of calcium chloride and hydroxyapatite to the tobacco extract is an efficient way to reduce the protein level.

10

The tobacco extract may also, or alternatively, be treated to remove polyphenols therefrom. PVPP is an insoluble adsorbent for polyphenols, traditionally used in the brewing industry to remove polyphenols from beer. PVPP in an amount of 25-75%, preferably 45-55%, of the weight of tobacco initially extracted may be added
15 to the extract. This amount of PVPP is capable of removing between 50 and 90% of the polyphenols in solution. If enhanced polyphenol removal is required, the PVPP treatment step can be carried out twice or carried out on partially concentrated extract.

20 The optimum pH for removal of polyphenols from the tobacco extract by PVPP is believed to be below 5. The efficiency of adsorption by PVPP may therefore be increased by reducing the pH of the extract via the addition of a suitable acid.

As an alternative to using PVPP to adsorb the polyphenols, one or more enzymes
25 may be added to the tobacco extract to degrade the polyphenols therein. A suitable enzyme is laccase (urishiol oxidase).

Electrophoresis could alternatively be used to remove proteins and/or polyphenols.

30 The invention is not, however, limited to methods for removing only proteins and/or polyphenols from tobacco. Alternative or additional enzymes, agents or adsorbents may be used to remove other tobacco constituents from the tobacco

extract. Examples of further tobacco constituents that could be removed from the extract include nitrates, amines and nitrosamines.

If a plurality of constituents are to be removed from the tobacco extract, a number
5 of process unit operations may be set up in series, each one utilising a different
enzyme, agent or adsorbent, in order for a chosen complement of constituents to be
removed. Alternatively, a single unit operation may contain a plurality of enzymes,
agents or adsorbents so that the constituents may be removed in a single step. For
example, a bentonite or PVPP holding tank could comprise one or more additional
10 enzymes, agents or adsorbents so as to remove not only protein or phenols from
the tobacco, but one or more further constituents also.

Following treatment of the tobacco extract to remove the selected constituent(s),
the extract is preferably concentrated to a solids concentration of between 20 and
15 50% by weight. Concentrations of up to 10% solids are most efficiently achieved
using reverse osmosis. A further concentration to approximately 40-60% solids may
be achieved by means of a falling or rising film evaporator. Preferably,
concentration is achieved using evaporation under vacuum. Sufficiently volatile
compounds could first be vacuum distilled off; this would be suitable where those
20 compounds affect the flavour and/or character of the tobacco, as the compounds
could be recovered without chemical damage due to subsequent treatment. Other
methods of concentration can be used and will be known to a person skilled in the
art.

25 The concentrated tobacco extract may be subsequently recombined with the
extracted tobacco. This may depend on whether and how the tobacco needs to be
stored prior to further processing, for example, into a reconstituted tobacco sheet.
Adding the treated extract back to the extracted tobacco ensures retention of
soluble flavour components of tobacco and nicotine in the final product.
30 Recombination therefore results in a tobacco product that can be reconstituted into
a form suitable for use in smoking articles, but with reduced levels of protein,
polyphenols or other constituent(s) of choice.

Recombination may be achieved by spraying the tobacco extract onto the tobacco. The amount of the original extract being recombined with the processed tobacco depends upon the amount that was lost during treatment of the extract to remove selected constituents, and will vary from one type of tobacco to the next.

5

Alternatively, and in a preferred embodiment, the separate streams of extracted tobacco and tobacco extract are fed into a mixing tank, where they may be mixed with additional ingredients, such as fibre, humectants and/or binder. Diluents, catalysts and/or filtration substances may also be mixed with the extracted tobacco
10 and tobacco extract, in order to modify the properties of the tobacco. The mixture is used to make a reconstituted tobacco sheet.

A conventional drying process may be used to dry the tobacco, either before, during or after recombination with the treated tobacco extract. The starting moisture
15 content of the tobacco is typically approximately 70-85%. In a preferred embodiment, the moisture content after drying should be approximately 14%.

A heated dryer, such as an apron dryer, may be used to reduce the moisture content in the tobacco or mixture to approximately 14%. Alternatively, the apron dryer may
20 be used to reduce the tobacco product moisture to 20-30%. A second heated dryer, such as a flash dryer, may then be used to further reduce the moisture content to approximately 14%.

If the tobacco has been dried, whether recombined with the tobacco extract or not,
25 it may form larger particles than the original grind or mill size, due to agglomeration. The dried tobacco may thus be remilled to at most 2 mm, preferably less than about 1 mm, 500 μm , 400 μm , 300 μm , 250 μm , 200 μm , 150 μm or 100 μm , and most preferably less than about 75 or 50 μm in mean diameter, to ensure that the particle size is suitable for any downstream processes, such as processing
30 into a reconstituted tobacco material, such as a reconstituted tobacco sheet.

The final dried product may subsequently be processed into a finished form, such as a sheet, which, when shredded, can form all or part of a cigarette filler.

In a particularly preferred embodiment, a method of making a reconstituted tobacco material is provided. The tobacco particles are contacted with water and subjected to intensive mechanical action, such as homogenisation, to generate a liquid tobacco
5 extract and a solid tobacco portion. A reconstituted tobacco sheet is then manufactured from the solid tobacco portion.

The reconstituted tobacco sheet recipe may include a portion of fibre for sheet strength. Fibre for inclusion in the sheet may be refined to the appropriate fibre
10 size of 0.2-3 mm, preferably 1 mm, using, for example, a mixed tank for dispersion of the fibre in water, a pump and a refiner. As above, the fibre may be simply mixed with the extracted tobacco and tobacco extract, which extracted tobacco and tobacco extract are fed into the mixing tank as separate streams. The fibre may be refined to a size suitable for making a reconstituted tobacco sheet either prior to, or
15 after, mixing with the extracted tobacco and tobacco extract.

Binder may be included in the reconstituted tobacco sheet recipe. A binder dosing and mixing system can be used to incorporate the binder into a water stream, for hydration. Suitable binders include pectins, guar gum, acacia gum, alginates and
20 xanthan gum; guar gum is preferred.

A mixing system may be used to bind the ingredients of the reconstituted tobacco sheet together. If the tobacco extract has not been recombined with the tobacco solids in an earlier stage of the treatment process, it may be incorporated into the
25 mixture at this stage. Flavouring agents may be added. Water, such as deionised or softened water, may be added; however, the calculation of water content should take into account any aqueous components added during the mixing step, such as flavouring agents, the tobacco extract and also any water required to hydrate the required amounts of fibre and binder; all of these may have significant water
30 content. A suitable method is as follows. The wood fibre is dispersed in the softened water with a high shear mixer for 15 minutes. The binder is added to the mixture to hydrate the binder. Mixing is achieved with a combination of the high shear mixer and recirculation through an inline high shear mixer for a period of 15

minutes or until the mixture is homogeneous. Subsequently, half of the tobacco solids, the tobacco extract and glycerol are added to the mixture and mixed with the same mixing system for a further 10 minutes.

- 5 A conventional bandcasting process can be used to cast the material into a reconstituted sheet. Alternatively, other tobacco reconstitution processes may be used, such as those based on paper making processes.

10 In a particularly preferred embodiment, the process for the treatment of tobacco comprises the steps of: (a) generating tobacco particles, having a mean diameter of less than 500, preferably less than 250, and most preferably less than 50 μm , from cured tobacco; (b) contacting the tobacco particles with water; (c) using a homogeniser to generate a tobacco extract and an insoluble tobacco portion; and (d) processing the insoluble tobacco portion into a reconstituted tobacco sheet.

15 In another particularly preferred embodiment, the process for the treatment of tobacco comprises the steps of: (a) contacting ground tobacco with water and subjecting it to high shear mixing to generate a tobacco extract and an insoluble tobacco portion; (b) separating the tobacco extract from the insoluble tobacco
20 portion; (c) treating the tobacco extract with bentonite to remove protein therefrom; (d) treating the tobacco extract with PVPP to remove polyphenols therefrom; (e) concentrating the tobacco extract; (f) mixing the tobacco extract and the insoluble tobacco portion, optionally with fibre, a binding agent and/or a humectant to form a mixture; and (g) bandcasting the mixture into a reconstituted
25 tobacco sheet.

The reconstituted tobacco sheet preferably contains about 80% tobacco and about 5% fibre, 5% binder and 5% glycerol. This means that the amount of protein in the reconstituted sheet is already reduced in comparison to untreated tobacco;
30 treatment of the tobacco using a process as described herein, however, reduces the amount of protein in the final product even further.

As there is a high content of tobacco in the reconstituted sheet, the sheet re-ordering at the end of the band needs to be available to ensure good release of the sheet from the band. The finished sheet should be collected and stored; for example, by slitting the sheet into manageable widths and reeling.

5

To aid understanding of the invention, a scheme of a suitable operational sequence for the extraction of tobacco, separation of the extracted tobacco from the tobacco extract and downstream processing of both components to produce a reconstituted tobacco sheet is shown in Figure 1.

10

In the illustrated embodiment, ground tobacco is contacted with water and subjected to high shear mixing. The resulting slurry is separated into a liquid tobacco extract and a solid tobacco portion. The tobacco extract is treated to remove protein and polyphenols therefrom (using bentonite and PVPP, respectively), and then concentrated. The treated extract and tobacco solids are mixed with fibre, binder and humectant and the resulting mixture bandcast into a reconstituted tobacco sheet. All steps are performed as herein described.

15

A reconstituted tobacco sheet, produced in accordance with any of the processes described herein, may be subsequently reduced in size, for example, by using a shredder that is capable of producing a cut tobacco-like strand width suitable for cigarette making, for example, 20-45 cuts-per-inch (c.p.i.).

20

The shredded tobacco may be included in a final blend, before incorporation into a smoking article, such as a cigarette.

25

Alternatively, the reconstituted tobacco sheet may be used to wrap around tobacco in the formation of a smoking article.

30

All of the features described herein (including any accompanying claims, abstract and drawings), and/or all of the steps of any method so disclosed, may be combined with any of the above aspects in any combination, except combinations where at least some of such features and/or steps are mutually exclusive.

The invention also includes further modifications and variations falling within the scope of the claims.

- 5 The following examples are provided in order to further illustrate the invention, but should not be construed as limiting the scope thereof.

Examples

10 **Example 1: Extraction of Protein from Ground Tobacco**

Finely ground tobacco (80% of the particles were smaller than 53 µm in particle diameter) was extracted using the following groups of solvents:

1. deionised water;
2. salt and water;
- 15 3. potassium hydroxide and water;
4. methanol pre-extraction, followed by potassium hydroxide and water.

Extraction was performed using a Waring blender, Brinkmann homogeniser and/or Soxhlet extractor. Details of the extraction procedure in each case are given in the
20 first column of Tables 1-5 below.

Each extraction procedure is recorded in Tables 1-5 as “solvent; method of extraction (total time of extraction in minutes); sequence time/tobacco:solvent ratio/number of sequences”. Thus, for example, Table 1 includes an extraction
25 procedure recorded as “H₂O - Blender (15); 5 min/1:10/3X”. This means that the tobacco was extracted in water using a Waring blender, the total time of extraction being 15 min. Extraction was performed in three sequences (“3X”), each sequence lasting five minutes. The tobacco:solvent ratio was 1:10.

30 Where more than one sequence of extraction was performed, the tobacco slurry was filtered after the first sequence and the tobacco residue was redispersed in a batch of fresh solvent for the second extraction, and so on. All the solvent fractions were combined when the extraction was completed.

The Kjeldahl method was used to measure the total nitrogen content of untreated and extracted tobacco residue. Protein content was calculated from the nitrogen determination using a conversion factor of 6.25, which corresponds to an average protein nitrogen content of 16% (w/w) per protein, as is standard in the art (G. Bokelman, W.S. Ryan Jr and E.T. Oakley, "Fractionation of Bright Tobacco", *J. Agric. Food Chem.* 31, 897-901 (1983)). In order to remove the contribution of non-protein nitrogen from certain tobacco constituents, the tobacco samples were extracted with a hot acetic solution (0.5% (v/v)) prior to analysis using the Kjeldahl method.

The results from the water extraction are presented in Table 1 and Figure 2.

TABLE 1				
% yield and % reduction in nitrogen of tobacco extracted with water				
Extraction Sequence	Tobacco Residue (dry weight basis)			
	% nitrogen	% yield	% weight loss	% weight reduction in nitrogen
Untreated	1.80	--	--	--
H ₂ O - magnetic stirrer 18 h/1:10/1X	1.59	48.4	51.6	57.2
H ₂ O - blender (15) 5 min/1:10/3X	1.42	34.2	65.8	73.0
H ₂ O - homogeniser (3) 3 min/1:10/1X	1.44	52.9	47.1	57.7
H ₂ O - homogeniser (6) 3 min/1:10/2X	1.50	41.8	58.2	65.1
H ₂ O - homogeniser (9) 3 min/1:10/3X	1.22	33.6	66.4	77.2
H ₂ O - homogeniser (12) 3 min/1:10/4X	1.15	32.3	67.7	79.3

The results indicated that vigorous extraction procedures were necessary to reduce the nitrogen level in cured tobacco extracted with water. The highest reduction in nitrogen content (79.3%) was obtained with a 12 minute extraction (four sequences of three minutes each) using a homogeniser. The powerful action of the homogeniser caused the tobacco tissue to literally explode. This demolishing action

solubilised many of the tobacco components, including proteins, and also explained the large weight losses observed (up to 68% weight loss).

The results obtained for tobacco samples extracted with water and various salts are given in Table 2 and shown in Figure 2.

TABLE 2 % yield and % reduction in nitrogen of tobacco extracted with water and various salts				
Extraction Sequence	Tobacco Residue (dry weight basis)			
	% nitrogen	% yield	% weight loss	% weight reduction in nitrogen
0.5% (w/v) NaCl-H ₂ O Homogeniser (3) 3 min/1:10/1X	1.58	52.1	47.9	54.3
0.5% (w/v) NaCl-H ₂ O Homogeniser (6) 3 min/1:10/2X	1.47	46.5	53.5	61.8
0.5% (w/v) NaCl-H ₂ O Homogeniser (9) 3 min/1:10/3X	1.27	37.3	62.7	73.7
0.5% (w/v) MgCl ₂ -H ₂ O Homogeniser (3) 3 min/1:10/1X	1.58	60.1	39.9	47.2
1.0% (w/v) SDS-H ₂ O Blender 5 min/1:10/1X	0.88	26.4	73.6	87.2

The presence of sodium chloride (NaCl) or magnesium chloride (MgCl₂) in solution did not markedly increase the efficiency of the solvent, in terms of nitrogen reduction, compared to pure water extraction (47.2-73.7% reduction and 57.2-79.3% reduction, respectively). There were also higher yields (i.e. less weight loss) measured for this series of extractions compared to those obtained with the pure water extractions.

However, the extraction performed with sodium dodecyl sulphate (SDS), using a Waring blender to homogenise the tobacco slurry, produced a very high reduction in tobacco nitrogen content (87.2%). It is thought that the micelles formed in water

by this surfactant were efficient at breaking tobacco membranes in order to dissolve tobacco components. The efficiency with which SDS solubilised tobacco components was also indicated by the short processing time (five minutes) necessary to achieve the high reductions in nitrogen content. However, it did incur
 5 large weight losses (73.6%).

The results obtained for tobacco samples extracted with aqueous potassium hydroxide (KOH) are given in Table 3 and shown in Figure 2.

TABLE 3 % yield and % reduction in nitrogen of tobacco extracted with water and potassium hydroxide				
Extraction Sequence	Tobacco Residue (dry weight basis)			
	% nitrogen	% yield	% weight loss	% weight reduction in nitrogen
0.8% (w/v) KOH-H ₂ O 3 h/150:4000/1X	1.07	19.5	80.5	88.4
1% (w/v) KOH-H ₂ O Homogeniser (3) 3 min/1:10/1X	1.42	54.5	45.5	57.0
1% (w/v) KOH-H ₂ O Homogeniser (6) 3 min/1:10/2X	1.42	43.5	56.5	65.7
10% KOH-H ₂ O Homogeniser (3) 3 min/1:10/1X	0.53	67.0	33.0	80.3
10% (w/v) KOH-H ₂ O Homogeniser (6) 3 min/1:10/2X	0.47	46.9	53.1	87.8

10

A high reduction in nitrogen was achieved using either a low KOH concentration (0.8% (w/v)) and a long processing time (three hours at 100 °C) or a high KOH concentration (10% (w/v)) and a short processing time (three to six minutes at room temperature). A long processing time resulted in very large weight losses
 15 (>80% weight loss), while a high KOH concentration affected the integrity of valuable tobacco components like sugars and flavours. Extraction using low KOH concentration (1% (w/v)) and a short processing time (three minutes) gave similar results to a pure water extraction (57.0%-57.7% nitrogen reductions).

In the final series of extractions, tobacco was first extracted with methanol using a Soxhlet extractor or a Waring blender. The extracted tobacco residue was left to dry overnight, and was then re-extracted under the various conditions summarised in Table 4. The pre-extraction and extraction conditions are shown in the two
 5 columns under the heading "Extraction Sequence". The results of these sequential extractions are given in Table 4.

TABLE 4 % yield and % reduction in nitrogen of tobacco extracted with methanol followed by water-potassium hydroxide extraction					
Extraction Sequence		Tobacco Residue (dry weight basis)			
		% nitrogen	% yield	% weight loss	% weight reduction in nitrogen
MeOH – Soxhlet - 18 h/1:10/1X		1.93	83.7	16.3	10.2
MeOH Soxhlet 18h/1:10/1X	H ₂ O Homog. (3) 3 min/1:10/1X	1.55	42.2	57.8	63.6
MeOH Soxhlet 18h/1:10/1X	1% (w/v) KOH-H ₂ O Homog. (3) 3 min/1:10/1X	1.43	46.7	53.3	62.9
MeOH Soxhlet 18h/1:10/1X	10% (w/v) KOH-H ₂ O Homog. (3) 3 min/1:10/1X	0.42	47.0	53.0	89.0
MeOH – Blender - 15min/1:12.5/1X		1.72	71.8	28.2	31.4
MeOH Soxhlet 15min/1:12.5/1X	10% (w/v) KOH-H ₂ O Homog. (6) 3 min/1:10/2X	0.16	24.5	75.5	97.7

The low extraction efficiency of the methanol-Soxhlet extraction, compared to the
 10 methanol-Waring blender extraction (16.3% weight loss compared to 28.2%) was due to the high packing density of the tobacco sample in the Soxhlet thimble.

The reductions in nitrogen of the methanol pre-extracted tobaccos were slightly higher than the reductions obtained, for the same treatment, using a non-pre-
 15 extracted tobacco. For example, a reduction of 57.7% in nitrogen was achieved when unextracted tobacco was treated with water and the homogeniser (Table 1, H₂O-homegeniser (3)), while a 63.6% reduction was obtained with a methanol pre-

extracted tobacco. A comparison of the extraction efficiencies for both types of treatment is presented in Table 5.

TABLE 5		
Comparison of nitrogen reduction for unextracted and methanol pre-extracted tobacco		
% weight decrease of nitrogen on tobacco residue		
Treatment	Unextracted %	Pre-extracted %
H ₂ O Homogeniser (3)	57.7	63.6
1% (w/v) KOH-H ₂ O Homogeniser (3)	57.7	62.9
10% (w/v) KOH-H ₂ O Homogeniser (3)	80.3	89.0
10% (w/v) KOH-H ₂ O Homogeniser (6)	87.8	97.7

- 5 The higher nitrogen reductions obtained with methanol pre-extracted tobacco can be explained by the lower amount of soluble material present in methanol pre-extracted tobacco. When the second extraction step was performed, the solvating power of the second solvent used was not reduced by the large amount of soluble material that was present in unextracted tobacco.

10

In summary, these results demonstrate that protein can be extracted from ground flue-cured tobacco using a non-enzyme-involved laboratory scale procedure. The greatest solubilisation of total nitrogen was obtained with either 10% (w/v) aqueous KOH or 1% (w/v) aqueous SDS (Tables 2 and 3 and Figure 2). Solubilisation of
 15 total nitrogen in water using the homogeniser increased with time of extraction, regardless of the solvent used (Figure 2). The solubilisation of total nitrogen from methanol pre-extracted tobacco was slightly better than from unextracted tobacco samples (Table 5).

- 20 Although the use of KOH and SDS increased the efficiency of extraction (by achieving a higher reduction in protein content), such chemicals can remain on the tobacco residue. Indeed, it is very difficult to rinse these chemicals from tobacco residue and a large volume of water is required for this purpose. The presence of

such chemicals in the final product is undesirable and can affect the taste of the tobacco. Moreover, noxious smoke compounds could be generated from these chemicals if present in the final product when smoked. Thus, deionised water remains the preferred solvent for these types of extractions.

5

Example 2: Extraction of Cut Tobacco

For comparative purposes, cut tobacco was extracted in an aqueous SDS solution; various concentrations of solvent were used. The cut lamina used for this series of extractions was a grade from the upper mid-stalk positions (CLX-85) cut at 35 cuts-
 10 per-inch (c.p.i.). The tobacco samples were soaked for 18 hours at 60-70 °C in aqueous solutions of SDS at the following concentrations: 0, 1, 1.5 and 2 % (w/v). The tobacco:solvent ratio was 1:20. The different slurries were then filtered and the extracted tobacco residues were thoroughly rinsed with warm water to remove all
 15 SDS left on the tobacco strands. The extracted tobacco residues were dried and analysed for total nitrogen. The results are presented in Table 6 and shown in Figure 3.

TABLE 6		
Cut tobacco (lamina only) extracted with various SDS concentrations		
Extraction	% nitrogen	% nitrogen reduction (relative to untreated)
Untreated	1.82	--
H ₂ O	2.02	-11.0
1% (w/v) SDS-H ₂ O	1.80	1.1
1.5% (w/v) SDS-H ₂ O	1.80	1.1
2% (w/v) SDS-H ₂ O	0.94	48.0

As the results show, 2% (w/v) SDS solution was the most efficient in reducing total
 20 nitrogen on cut tobacco lamina. The proportion of nitrogen in this sample (0.94%) was similar to the proportion obtained in ground tobacco using a 1.0% (w/v) SDS solution and a Waring blender (0.88%, see Table 2).

These results thus show that the extraction of ground tobacco using an intensive
 25 mechanical action is a much more effective method for reducing nitrogen content of tobacco than a simple extraction (i.e. without agitation) of cut tobacco. Indeed, comparing the results obtained with a 1% (w/v) SDS solution in each case, 0.88%

nitrogen was obtained in the sample extracted according to the former (Table 2), but only 1.80% nitrogen was obtained in the sample extracted according to the latter (Table 6).

5 **Example 3 – Vibrating Sieve Trial**

This study investigated the use of a vibrating sieve to separate extracted tobacco solids from a tobacco extract. Figure 4 shows a process flow diagram of the experimental setup for the trial.

- 10 Three experiments were conducted. In each, 11.25 kg of ground tobacco was slurried in 200 litres of deionised water at 60 °C, and kept in a tank for 40 minutes while being mixed with a high shear mixer. The high shear action encouraged the removal of protein from the structure of the tobacco fibre.
- 15 The resulting slurry was then pumped to a 400 mm diameter vibrating sieve, which had been fitted with a 40 µm mesh (in the first two experiments) or 20 µm mesh (in the third experiment). The aqueous solution, with the suspended fine particles, passed through the sieve mesh, and the retained tobacco dust particles travelled over to the solids outlet across the mesh propelled by the vibratory action, which
- 20 also served to prevent the sieve mesh from blocking with fine particles. The incoming slurry was transferred to the vibrating sieve in one hour, giving a flow rate of 211 kg/h. The feed to the sieve was stopped on a few occasions to allow the solids build-up on the sieve to reduce.
- 25 In the second experiment only, the process was repeated with the wet solids retained by the sieve being re-slurried in 180 litres of deionised water at 60 °C and the process repeated. Table 7 summarises the three experiments.

TABLE 7		
Summary of the protocol for the three vibrating sieve experiments		
	Number of Washes	Mesh Size
Experiment 1	1	40 µm
Experiment 2	2	40 µm
Experiment 3	1	20 µm

The solid and liquid portions resulting from each experiment were weighed, and the yields measured. Samples of the solids were analysed for protein, total nitrogen and moisture content. The liquid was analysed for solids content via evaporation and a gravimetric technique.

5

The measures of protein nitrogen and total nitrogen were corrected for the mass lost during the extraction by measuring the total solids in the liquid extract. The results are therefore presented as a percentage of the original pre-extracted weight.

10 Table 8 summarises results for the various experiments.

TABLE 8			
Summary of the results from the three vibrating sieve experiments			
	40µm Mesh, Single Wash	40µm Mesh, Double Wash	20µm Mesh, Single Wash
Mass initial tobacco (kg)	11.25	11.25	11.25
Initial % protein nitrogen (dry weight basis; dwb)	0.85	0.85	0.85
Initial % total nitrogen (dwb)	2.5	2.5	2.5
Mass water (kg)	200	380	200
Mass extract collected (kg)	144	321*	117
% dissolved and suspended matter in extract	3.46	1.88	3.44
Mass solids collected (kg) (wet weight)	63	55	83
% protein nitrogen in extracted solids (dwb)	0.65	0.66	0.77
% protein in extracted solids (dwb)**	4 %	4.1%	4.8%
% Total Nitrogen in extracted Solids (dwb)	1.18%	0.81%	1.36%

* Including mass of extract from 1st extraction

** Assuming nitrogen is 16% of the protein

15 The vibrating sieve was a simple and effective way of separating the tobacco fibre from the extract. The vibratory action prevented the sieve from blinding as has been noted with other filter arrangements.

Figure 5 shows the concentration of both total nitrogen and protein nitrogen for the various sieve trials described above. These data represent the nitrogen levels in the solids collected after the sieving (following drying). The results show a removal of both protein nitrogen and total nitrogen during the extraction and separation of the ground tobacco.

Figure 6 shows the same data, but presented as a percentage change in both total nitrogen and protein nitrogen, compared to the feedstock. The results show a reduction in protein nitrogen and total nitrogen after extraction.

10

The experiment using a smaller sieve mesh resulted in a higher concentration of both protein and total nitrogen in the residual solids compared with the larger mesh size, i.e. it resulted in lower nitrogen removals. This demonstrates that the fibre separation step was instrumental to the effectiveness of the protein removal of protein in this process.

15

Example 4 – Production of Reconstituted Material

A tobacco extraction scheme using ground tobacco and an intensive mechanical action, followed by separation of the extract solution from the unextracted portion and purification of the extract solution, was exemplified in Examples 1 and 3. The main goal of that work was to produce an acceptable smoking product having low protein content. Treated reconstituted tobacco may be produced and used to make cigarettes, as described below.

A conventional reconstituted tobacco (“conventional recon”) and a reconstituted tobacco produced in accordance with the present invention (“treated recon”) were produced according to the recipes described in Table 9. Methods of production of the two materials are described in A) for the treated recon and B) for the conventional recon.

30

TABLE 9		
Ingredients of treated reconstituted tobacco and conventional reconstituted tobacco		
	Treated Recon	Conventional Recon
Ground tobacco (dry weight basis; dwb)	-	85%
Extracted ground tobacco (dwb)	46.75%	-
Treated tobacco extract(dwb)	38.25%	-
Glycerol(dwb)	5%	5%
Guar gum (dwb)	5%	5%
Wood fibre (dwb)	5%	5%

A) Production of Treated Recon

11.25 kg ground tobacco was slurried in 200 litres of deionised water at 60 °C, and kept in a tank for 40 minutes while being mixed with a high shear mixer. The high shear action encouraged the removal of protein from the structure of the tobacco fibre.

The resulting slurry was then pumped to a 400 mm diameter vibrating sieve which had been fitted with a 40 µm mesh. The aqueous extract, with the suspended fine particles, passed through the sieve mesh, and the retained tobacco dust particles travelled over to the solids outlet across the mesh propelled by the vibratory action, which also served to prevent the sieve mesh from blocking with fine particles. The incoming slurry was transferred to the vibrating sieve at a flow rate of approximately 200 kg/h.

The aqueous extract was first treated to remove suspended protein particles. These were coagulated using bentonite at 1 g/l extract. The separation was achieved using a disc bowl centrifuge with a trap filter inline after. This treatment was followed by the removal of polyphenols from solution in the extract. In this process, polyvinylpolypyrrolidone (PVPP) was used to adsorb the polyphenols in a mixing tank at 3% of the extract. The PVPP adsorbed polyphenols as the extract passed through it. The loaded PVPP was filtered out of the liquid using a filter press. The PVPP was regenerated using caustic soda solution for re-use. The extract was

concentrated to approximately 50% dissolved matter, using a spinning cone evaporator (under vacuum of -90 kPa). The extract was frozen.

5 A mixing system was used to mix the ingredients (Table 9) for the reconstituted tobacco sheet with water to a final solids concentration of 15%. Firstly, the wood fibre was dispersed in softened water with a high shear mixer for 15 minutes. The binder was added to the mixture to hydrate the binder. Mixing was achieved with a combination of the saw mixer and recirculation through an inline high shear mixer for a period of 15 minutes or until the mixture was smooth. Subsequently half of
10 the tobacco solids, and the tobacco extract (as described above) and glycerol were added to the mixture, and mixed with a paddle mixer and high shear for 5 minutes. The remaining tobacco solids were added and mixed using the same mixing system, for a further 10 minutes.

15 The mixture was laid at a thickness of preferably 1.0 – 1.5 mm on a stainless steel band through an extrusion manifold at a flow rate of 65 kg/hr. The material was conveyed into a three zone apron drier which had a total retention time of 15 minutes. Each of the three zones (Z1, Z2 and Z3) had an electric heating unit controlled at 110 °C and exhaust extraction for removing humid air at a flow of 23
20 m³/min. The majority of the drying was driven by saturated steam boxes (two for each zone) underneath the band on the opposing side of the stainless steel band. The temperatures of each of the three zones Z1, Z2 and Z3 were approximately 85 °C, 95 °C and 100 °C respectively.

25 Once the material had left the oven, moisture was added through a re-ordering zone which condensed steam from a preheated box and humidifying lance (feed from reduced steam at 2.5 bar and a control valve open at 21%). The resulting sheet was further dried in a tray drier at ambient conditions for approximately 30 minutes. The resulting sheet was reconditioned at 22 °C and 60% relative humidity at least
30 overnight.

The sheet material was shredded at 28 cuts-per-inch (c.p.i.) and blended with 20% (by mass) of expanded tobacco and 80% recon. King size cigarettes were manufactured.

5 B) Production of Conventional Recon

A mixing system was used to mix the ingredients (Table 9) for the reconstituted tobacco sheet with water to a final solids concentration of 15%. Firstly, the wood fibre was dispersed in softened water using a high shear mixer for 15 minutes. The binder was added to the mixture and hydrated using a high shear mixer for a period
10 of 15 minutes or until the mixture was smooth. Subsequently, half of the ground tobacco and the glycerol were added to the mixture and mixed with a paddle mixer and high shear for 5 minutes. The remaining tobacco was then added and mixed using the same mixing system for a further 10 minutes.

15 The mixture was laid at a thickness of preferably 1.0 – 1.5 mm on a stainless steel band through an extrusion manifold at a flow rate 65 kg/hr. The material was conveyed into a three zone apron drier which had a total retention time of 15 minutes. Each of the three zones (Z1, Z2 and Z3) had an electric heating unit controlled at 110 °C and exhaust extraction for removing humid air at a flow of 23
20 m³/min. The majority of the drying was driven by saturated steam boxes (two for each zone) underneath the band on the opposing side of the stainless steel band. The temperatures of each of the 3 zones, Z1, Z2 and Z3, were approximately 85 °C, 95 °C and 100 °C respectively.

25 Once the material had left the oven, moisture was added through a re-ordering zone, which condensed steam from a preheated box and humidifying lance (feed from reduced steam at 2.5 bar and a control valve open at 21%). The resulting sheet was further dried in a tray drier at ambient conditions for approximately 30 minutes. The resulting sheet was reconditioned at 22 °C and 60% relative humidity
30 at least overnight.

The sheet material was shredded at 28 cuts-per-inch (c.p.i.) and blended with 20% (by mass) of expanded tobacco and 80% recon. King size cigarettes were manufactured.

- 5 Table 10 shows the total nitrogen and the protein nitrogen of the cigarette blends for both the treated reconstituted material and the conventional reconstituted material.

TABLE 10		
Total and protein nitrogen content of treated recon blend and conventional recon blend as taken from king size cigarettes		
	Total Nitrogen	Protein Nitrogen
Treated recon blend	2.36%	0.82%
Conventional recon blend	2.42%	0.93%

- 10 As the results show, a reduction of 10% in protein nitrogen was achieved in the treated recon compared to the conventional recon.

Claims

1. A process for the treatment of tobacco comprising a step in which ground tobacco is contacted with a solvent and subjected to an intensive mechanical action
5 to generate a tobacco extract and an insoluble tobacco portion.
2. A process as claimed in claim 1, further comprising, as a preceding step, preparation of ground tobacco in a form of tobacco particles having a mean diameter of less than 500 μm .
10
3. A process as claimed in claim 2, wherein the tobacco particles have a mean diameter of less than 250 μm .
4. A process as claimed in claim 3, wherein the tobacco particles have a mean
15 diameter of less than 50 μm .
5. A process as claimed in any one of claims 1-4, wherein the ground tobacco is prepared from cured tobacco.
- 20 6. A process as claimed in any one of claims 1-5, wherein the solvent is an aqueous solvent.
7. A process as claimed in claim 6, wherein the aqueous solvent is water.
- 25 8. A process as claimed in any one of claims 1-7, wherein the intensive mechanical action is homogenisation, high shear mixing or blending.
9. A process as claimed in any one of claims 1-8, further comprising separating the tobacco extract from the insoluble tobacco portion.
- 30 10. A process as claimed in claim 9, wherein the tobacco extract is separated from the insoluble tobacco portion using a vibrating sieve filtration system.

11. A process as claimed in claim 9 or claim 10, further comprising treating the tobacco extract to remove protein therefrom.
12. A process as claimed in claim 11, wherein the tobacco extract is treated with
5 bentonite.
13. A process as claimed in any one of claims 9-12, further comprising treating the tobacco extract to remove polyphenols therefrom.
- 10 14. A process as claimed in claim 13, wherein the tobacco extract is treated with polyvinylpyrrolidone (PVPP).
15. A process as claimed in any one of claims 9-14, further comprising concentrating the tobacco extract.
- 15 16. A process as claimed in any one of claims 9-15, further comprising recombining the tobacco extract and the insoluble tobacco portion.
17. A process as claimed in any one of claims 1-16, further comprising
20 processing the insoluble tobacco portion into a reconstituted tobacco sheet.
18. A process as claimed in claim 17, wherein the processing of the insoluble tobacco portion into a reconstituted tobacco sheet comprises drying and milling the insoluble tobacco portion to a particle size suitable for making a reconstituted
25 tobacco sheet.
19. A process as claimed in claim 17 or claim 18, wherein the processing of the insoluble tobacco portion into a reconstituted tobacco sheet comprises adding:
- 30 a) fibre;
b) a binding agent; and/or
c) a humectant;
to the insoluble tobacco portion.

20. A process as claimed in any one of claims 17-19, wherein the processing of the insoluble tobacco portion into a reconstituted tobacco sheet comprises bandcasting the insoluble tobacco portion into a reconstituted tobacco sheet.
- 5 21. A process as claimed in any one of claims 17-20, further comprising shredding the reconstituted tobacco sheet to produce cut strands of a width suitable for use in a smoking article.
- 10 22. Tobacco or reconstituted tobacco obtainable by a process as claimed in any one of claims 1-21.
23. A smoking article comprising tobacco or reconstituted tobacco as claimed in claim 22.

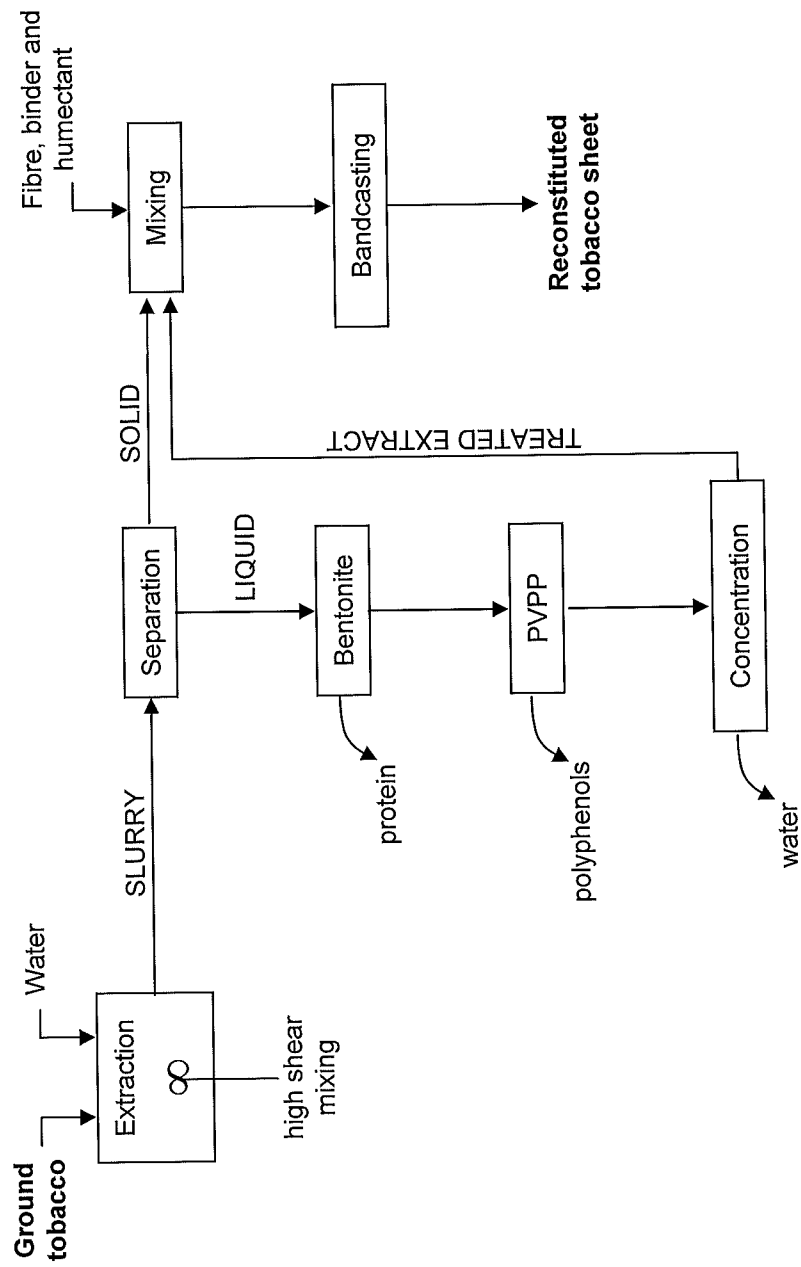


Figure 1

2/6

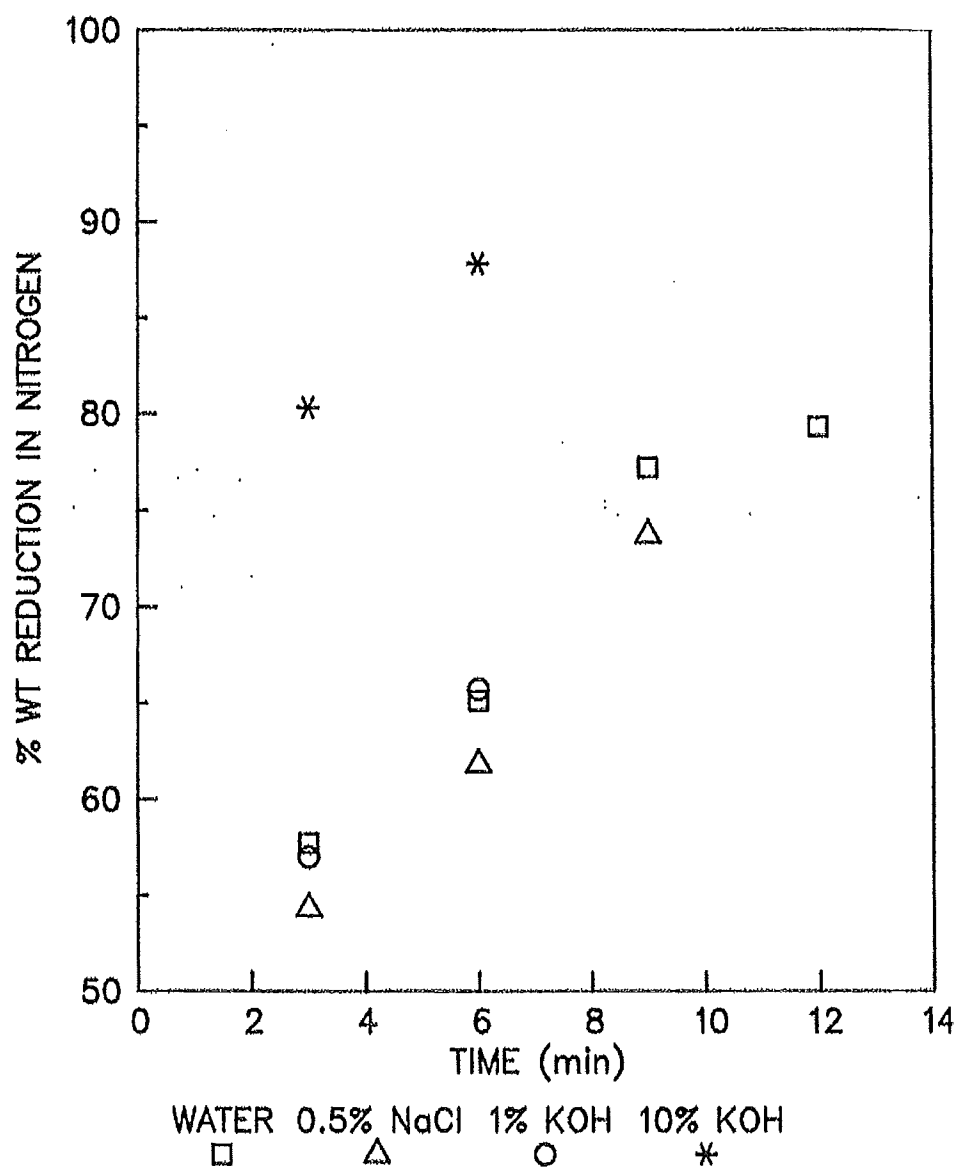


Figure 2

3/6

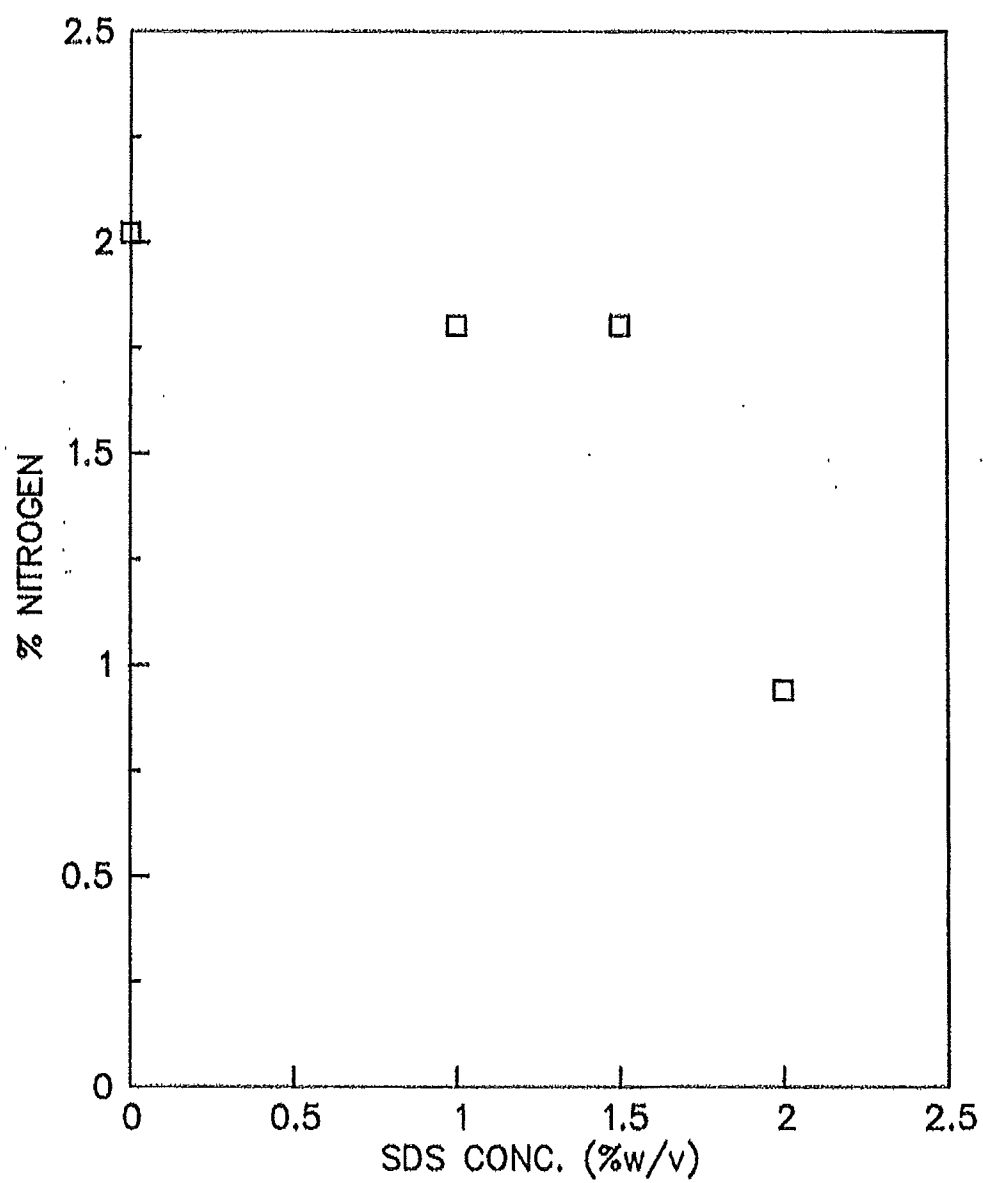


Figure 3

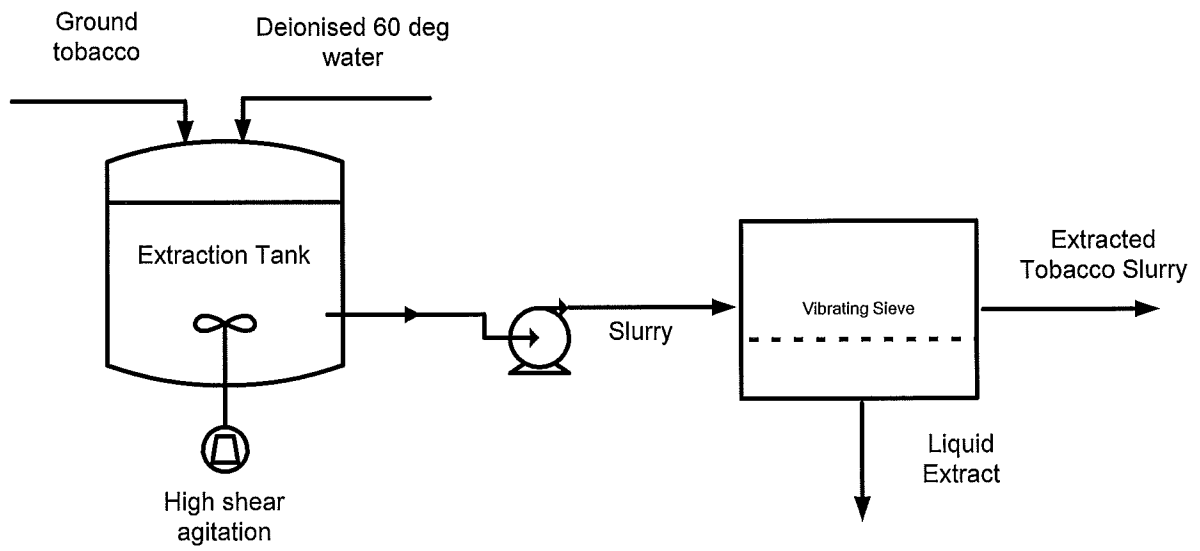


Figure 4

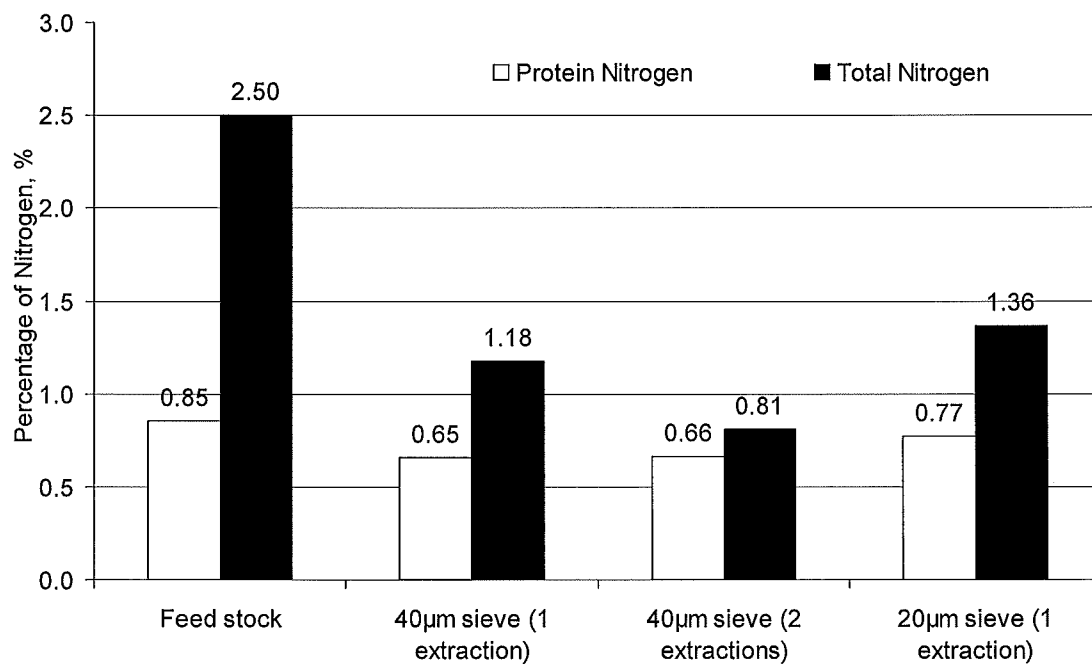


Figure 5

- 6/6 -

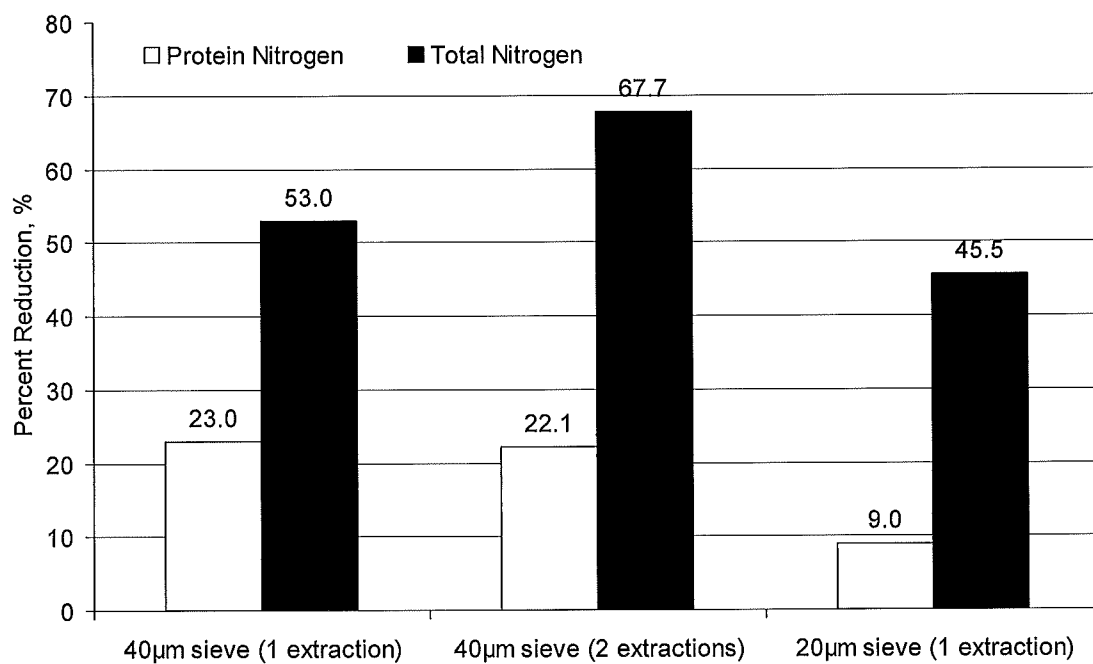


Figure 6

INTERNATIONAL SEARCH REPORT

International application No
PCT/GB2012/051152

A. CLASSIFICATION OF SUBJECT MATTER
INV. A24B3/14 A24B15/24
ADD.

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)
A24B

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

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

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 343 879 A (TEAGUE RICHARD A [US]) 6 September 1994 (1994-09-06) Claims 1, 6, 9, 22, 23; column 1, line 44 - line 62 Claim 15; column 1, line 67 - column 2, line 4 Claim 5; column 2, line 38 - line 46 Claims 1, 2; column 2, line 54 - line 64 Claim 7; column 3, line 2 - line 4 Claim 1; column 4, line 9 - line 19 Claims 9, 10; column 4, line 34 - line 36 Claim 15; column 4, line 58 - line 63 -/--	1-7,9, 10,15, 22,23

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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Name and mailing address of the ISA/

European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040,
Fax: (+31-70) 340-3016

Authorized officer

Dimoula, Kerasina

INTERNATIONAL SEARCH REPORT

International application No

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	Claim 3; column 5, line 60 - line 61 ----- US 2005/241657 A1 (MUA JOHN-PAUL [US] ET AL) 3 November 2005 (2005-11-03) Claims 1, 6, 7, 9, 10, 16, 17; paragraph [0006] Claims 15, 20; paragraph [0007] Claim 13; paragraph [0015] Claim 11; paragraphs [0008], [0011] -----	1,6,7, 9-11,13, 15-17, 20,22
X	WO 02/03817 A2 (BRITISH AMERICAN TOBACCO CO [GB]) 17 January 2002 (2002-01-17) Claims 1, 5, 6, 9, 15, 16, 17, 22, 23; page 3, paragraph 1 Claims 1, 6, 7, 8, 11, 12; page 5, paragraph 3 Claim 21; page 8, paragraph 3 -----	1,5-12, 15-17, 21-23
X	WO 2004/021809 A1 (BRITISH AMERICAN TOBACCO CO [GB]; MUA JOHN-PAUL [US]; HAYES BRAD L [US]) 18 March 2004 (2004-03-18) Claims 1, 5, 6, 9, 22, 23; page 2, line 15 - line 19; examples 4A, 4B, 5A, 5B Claims 7, 9, 11, 12, 13, 14; page 4, line 5 - line 22 -----	1,5-7,9, 11-13, 21-23
X	US 2006/037620 A1 (THOMPSON BRUCE T [US]) 23 February 2006 (2006-02-23) Claims 1, 22, 23; paragraph [0009]; claim 3 Claims 17, 20; paragraph [0012] Claims 6, 7, 9; paragraph [0025] Claim 11; paragraph [0027] Claim 5; paragraph [0032] -----	1,5-7,9, 11,17, 20,22,23

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INTERNATIONAL SEARCH REPORT

International application No
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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 2005/099493 A2 (PHILIP MORRIS PROD [CH]) 27 October 2005 (2005-10-27) Claims 1, 5, 6, 9, 13, 14, 15, 16; page 2, paragraph 5 Claims 5, 22, 23; page 4, paragraph 14 Claim 1; page 5, paragraph 15 - paragraph 16 Claims 15, 16; page 5, paragraph 18 Claim 7; page 5, paragraph 19 -----	1,5-7,9, 13-16, 22,23
X	US 2006/021626 A1 (MUA JOHN-PAUL [US]) 2 February 2006 (2006-02-02) Claims 1, 22, 23; page 1, paragraph 3 Claims 1, 6, 7, 22, 23; page 1, paragraph 16 Claims 9, 15, 16, 19, 20, 21; page 2, paragraph 17 - paragraph 19 -----	1,5-7,9, 15-23

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2012/051152

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 5343879	A	06-09-1994	NONE
US 2005241657	A1	03-11-2005	AR 049891 A1 13-09-2006
			AR 061809 A2 24-09-2008
			AT 385703 T 15-03-2008
			AT 444000 T 15-10-2009
			BR PI0510353 A 06-11-2007
			CA 2563483 A1 01-12-2005
			CA 2711641 A1 01-12-2005
			DE 602005004773 T2 10-06-2009
			EP 1742546 A2 17-01-2007
			EP 1847184 A2 24-10-2007
			ES 2302227 T3 01-07-2008
			ES 2334269 T3 08-03-2010
			HN 2005000186 A 14-04-2009
			MY 138392 A 29-05-2009
			SG 135182 A1 28-09-2007
			US 2005241657 A1 03-11-2005
			WO 2005112669 A2 01-12-2005
			ZA 200608736 A 25-06-2008
WO 0203817	A2	17-01-2002	AU 7428901 A 21-01-2002
			AU 2001274289 B2 05-08-2004
			CN 1440242 A 03-09-2003
			EP 1301095 A2 16-04-2003
			JP 3761519 B2 29-03-2006
			JP 2004502440 A 29-01-2004
			NZ 523318 A 30-07-2004
			UA 73196 C2 15-07-2003
			US 6508254 B1 21-01-2003
			WO 0203817 A2 17-01-2002
			ZA 200300034 A 07-11-2003
WO 2004021809	A1	18-03-2004	AR 041167 A1 04-05-2005
			AU 2003270035 A1 29-03-2004
			BR 0314153 A 12-07-2005
			CA 2502674 A1 18-03-2004
			CN 1694628 A 09-11-2005
			EP 1545249 A1 29-06-2005
			ES 2326413 T3 09-10-2009
			NZ 538851 A 22-12-2006
			RU 2306836 C2 27-09-2007
			US 2004045565 A1 11-03-2004
			US 2006130859 A1 22-06-2006
			WO 2004021809 A1 18-03-2004
US 2006037620	A1	23-02-2006	AR 050461 A1 25-10-2006
			HN 2005000447 A 19-08-2010
			US 2006037620 A1 23-02-2006
			WO 2006023330 A1 02-03-2006
WO 2005099493	A2	27-10-2005	US 2005279374 A1 22-12-2005
			WO 2005099493 A2 27-10-2005
US 2006021626	A1	02-02-2006	AR 050441 A1 25-10-2006
			AR 067829 A2 21-10-2009
			AT 401010 T 15-08-2008
			AU 2005269288 A1 09-02-2006

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/GB2012/051152

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
		BR PI0513980 A	20-05-2008
		CA 2574858 A1	09-02-2006
		CN 101102684 A	09-01-2008
		EP 1771096 A1	11-04-2007
		ES 2310842 T3	16-01-2009
		JP 4467617 B2	26-05-2010
		JP 2008507978 A	21-03-2008
		KR 20070039601 A	12-04-2007
		MY 139598 A	30-10-2009
		RU 2358621 C2	20-06-2009
		US 2006021626 A1	02-02-2006
		WO 2006015070 A1	09-02-2006
		ZA 200700722 A	25-09-2008
